UDC 004.383.4+621.375
DOI: 10.15587/1729-4061.2025.339913

IMPROVING THE CONDUCTIVE PROPERTIES OF PRINTED ELECTRONICS LAYERS BY TREATING PAPER SUBSTRATES WITH CORONA DISCHARGE BEFORE SCREEN PRINTING

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This study's object is the conductive layers of printed electronics based on graphene plastisol ink, applied by screen printing on glossy and matte paper substrates, pre-modified by corona discharge. The problem addressed is low adhesion and instability of conductive layers on paper substrates because of their roughness, porosity, and hydrophilicity.

It was found that corona discharge treatment reduces the specific resistance of conductive tracks on matte paper by 25–30% compared to untreated samples; on glossy paper – by 8–12%. The best results were obtained at a power of 3000 W: for matte paper, the resistance of 1 mm wide tracks decreased from 1447.1 Ohm to 1035.6 Ohm, and for 5 mm tracks – from 184.0 Ohm to 161.1 Ohm. After testing, the increase in resistance in the M3000 samples averaged 2–5%, while in untreated counterparts it was up to 46%.

Interpretation of the results revealed that the increase in surface energy and micro-roughness after corona treatment contributes to better wetting and fixation of graphene ink, the formation of a denser conductive layer, and a reduction in contact defects. A distinctive feature is the confirmed stability of electrical characteristics after thermal cycles and a decrease in the proportion of complete failures in pre-treated glossy paper samples. Additionally, a reduction in measurement scatter and improved print reproducibility for narrow tracks (1–2 mm) on matte paper after 3000 W corona treatment was noted.

The practical significance of the results is the possibility of using corona discharge treatment in the production of flexible printed electronics on paper media, especially for miniature elements with high requirements for conductivity and wear resistance. The method is effective in mass roll-to-roll (R2R) production, compatible with thin substrates, and does not require complex integration into the technological process

Keywords: printed electronics, flexible electronics on paper, graphene conductive layers, wear resistance

Received 27.06.2025
Received in revised form 27.08.2025
Accepted 05.09.2025
Published 30.10.2025

How to Cite: *Kyrychok, T., Klymenko, T., Bardovskyi, B., Avdiakov, Y. (2025). Improving the conductive properties of printed electronics layers by treating paper substrates with corona discharge before screen printing. Eastern-European Journal of Enterprise Technologies, 5 (1 (137)), 19–30. https://doi.org/10.15587/1729-4061.2025.339913*

1. Introduction

Printed electronics is one of the most dynamic and promising areas of modern science and technology, focused on designing flexible, lightweight, and cost-effective electronic devices. Unlike conventional silicon technologies, it makes it possible to apply functional materials directly to various substrates using printing methods, in particular screen printing, inkjet printing. or offset printing [1]. This approach opens up wide opportunities for industry – from the production of flexible displays and RFID tags to biosensors and energy system elements, which makes the topic of research in this area relevant for the high-tech market.

One of the key challenges in the development of printed electronics is the selection and preparation of the substrate for applying functional layers. Paper is considered a promising material due to its low cost, biodegradability, and availability. However, its natural roughness, porosity, and hydrophilicity make it difficult to form uniform conductive layers and reduce the stability of electrical characteristics [2]. This necessitates the development and improvement of methods for modifying the surface of paper substrates, which enable proper adhesion of functional materials and increase the durability of electronic components.

Among the most effective methods of surface preparation for flexible electronics, plasma treatment [3] and solvent cleaning [4] are distinguished. Corona discharge treatment occupies a special place due to its easy integration into roll production lines [5], non-contact effect, and compatibility with thin substrates [6].

At the same time, screen printing technology is key for the manufacture of elements where the conductivity and stability of the layer are critical. The combination of a properly prepared substrate and an optimized printing process makes it possible to reduce coating defects, reduce contact resistance, and increase the stability of electrical characteristics in flexible electronic devices.

Therefore, it is a relevant task to carry out studies on improving the suitability of paper substrates for printed electronics by treating them with corona discharge before screen printing of conductive layers, both from the point of view of fundamental science and for practical implementation in the production of flexible electronics.

2. Literature review and problem statement

Review of current research demonstrates increased interest in improving the printing technologies of electronics on flexible substrates, in particular with the use of cheaper materials such as paper. Notwithstanding, the systematization of methods reported in [7] shows that most approaches are focused more on the classification and listing of printing capabilities than on finding ways to overcome technological barriers. In particular, although the authors emphasize the influence of porosity and roughness of paper substrates on the formation of conductive layers, they almost do not analyze the extent to which these parameters allow for reproducibly and stably produce electrical characteristics under real conditions of use. Thus, the question of effective adaptation of technological processes, especially methods of preliminary surface preparation, remains open.

In [8], the prospects for integrating printing technologies with scalable methods, in particular roll-to-roll (R2R) production, which is positioned as the key to designing functional sensors and circuits, are outlined. However, the emphasis is mainly on the potential of the technology, rather than the actual results of its application. The question of the practical implementation of R2R processes specifically for paper substrates remains open as the review is limited to the general characteristics of the materials and only states their heterogeneity and adhesion problems. It is not analyzed to what extent these factors affect the long-term stability of electronic structures or the reproducibility of production cycles on an industrial scale. Thus, although the promising nature of the area is recognized, the literature lacks critical studies that would combine the scalability of production with a realistic assessment of the reliability of paper substrates, which forms a significant gap for further work.

Study [9] demonstrates the high potential of graphene and other carbon nanomaterials as conductive components and emphasizes the importance of post-printing processing methods, such as thermal or UV irradiation, to increase the conductivity of structures. However, the results mostly demonstrate the effectiveness of these approaches on polymer or specially prepared substrates while the issue of their compatibility with paper substrates is practically not analyzed. The temperature instability and high hygroscopicity of paper significantly limit the possibility of using standard sintering or photonic processing methods but the authors limit themselves to general statements without a specific analysis of how these factors affect the stability and durability of conductive layers. As a result, the promise of nanomaterials is declared but the lack of research on optimizing post-processing modes on paper substrates leaves open the question of their practical implementation in scalable printing technologies.

All this indicates the presence of a research niche related to studying the effect of corona discharge pretreatment on the electrical conductivity and stability of conductive layers deposited by screen printing on paper substrates, in order to improve their suitability for use in flexible electronics.

Study [10] confirmed that corona discharge pretreatment of paper substrates can reduce the electrical resistance of deposited conductive tracks and increase their adhesion and stability, especially in the case of matte paper with a chromium sublayer. However, the results were obtained for magnetron sputtering, which is characterized by controlled film formation conditions and does not reflect the specific challenges of printing deposition methods. In particular, the study does not analyze how the effectiveness of corona treatment would be preserved when using screen printing or other types of printing, where ink permeability, layer heterogeneity, and adhesion variability on large-scale planes are critical. Thus, although corona treatment does show potential in reducing the limitations of paper substrates, the lack of its testing in direct printing technologies leaves open the question of the real effectiveness of the method for the practical production of printed electronics.

In work [11], it is shown that the parameters of corona treatment directly affect the surface morphology and the quality of reproduction of prints: both the type of discharge and the power level determine the suitability of the paper for layering. The authors note that excessive activation leads to increased roughness and causes uneven distribution of ink, which reduces the quality of the formed structures. However, the study mainly focuses on color reproduction in digital offset printing, and not on functional conductive layers, which are critical for printed electronics. Accordingly, the work confirms the importance of optimizing corona treatment modes but does not provide an answer to the extent to which these parameters affect the electrical characteristics of conductive tracks or long-term stability under real operating conditions. This indicates a gap between the visual quality of prints and their functional properties, which requires further research.

According to theoretical provisions [12], corona treatment does indeed increase the surface energy and wettability of the material through the formation of polar functional groups, which promotes adhesion. However, such statements remain mostly general and do not take into account the complexity of real printing processes. In particular, the authors note the possibility of changing the surface microrelief during bombardment with charged particles, but do not analyze how controlled this modification is and whether it does not lead to excessive roughness, which complicates uniform ink spreading. In addition, although the importance of power density (watt density) as a key parameter is indicated, there are no specific data on its optimal values for paper substrates, where the combination of increased hygroscopicity and structural heterogeneity can significantly change the effect of the treatment. This indicates the need for applied research that goes beyond general theory and focuses on the relationship between surface morphological changes and the quality of conductive structures in printed electronics.

Thus, although corona treatment has already proven its effectiveness in combination with vacuum deposition methods, there are no comprehensive experimental works regarding its adaptation to screen printing with carbon nanomaterials. This determines the feasibility of research aimed at optimizing the processing parameters to ensure stable electrical characteristics when printing on paper substrates.

Precise and stable formation of conductive layers is rightly defined as a key stage in the development of printed electronics. In study [13], it was demonstrated that the use of a silicon stencil in the process of screen printing with graphene inks makes it possible to achieve high resolution of prints, increased electrical conductivity, and mechanical flexibility of structures. The authors also emphasize the critical role of controlling the rheological properties of the inks and process parameters; however, their results are based mainly on polymer and silicon substrates. This limits the applicability of the findings to paper substrates, where significantly higher roughness, heterogeneity, and hygroscopicity can significantly affect the quality of ink transfer and long-term stability of the layers. The lack of data on the adaptation of the silicon stencil to cheaper and less stable substrates such as paper indicates that, despite the technological breakthrough, the work rather outlines the potential of the method than answers the question of its practical effectiveness for the mass production of printed electronics.

In [14], it was proven that the coordination of the rheological properties of the ink, the stencil configuration, and the adhesive characteristics of the substrate is a necessary condition for achieving high conductivity and structural integrity of printed circuits. The authors showed that the use of gel-like graphene pastes with high concentration makes it possible to form lines up to 40 μm wide with a resistivity of about 30 Ω after a short drying at 100°C. At the same time, the work focuses mainly on demonstrating technical feasibility and does not contain an in-depth analysis of the long-term stability of such structures, especially on paper substrates, where heterogeneity and hygroscopicity can significantly affect the repeatability of results in large-scale production. Thus, despite the technological breakthrough in reducing the drying temperature and adapting to industrially relevant materials, the issues of controlling the stability and reliability of printed layers under actual operating conditions remain unresolved.

In study [15], it was demonstrated that the use of specially gelled graphene pastes makes it possible to form conductive layers up to 25 μm thick with a surface resistivity of about 30 Ohms, and the drying process occurs at a temperature of 100°C. This opens up the possibility of using such inks on paper substrates that cannot withstand high-temperature annealing. At the same time, the results focus primarily on laboratory conditions and do not contain a detailed analysis of the long-term stability of the layers under cyclic deformations or under conditions of high humidity, which are typical for paper substrates. In addition, the cited work practically does not consider the problem of paper surface heterogeneity, which can significantly affect the reproducibility of resistance and mechanical reliability in large-scale production. Thus, despite the promising potential of low-temperature drying for the integration of printed electronics on cellulose substrates, the question of the practical implementation of such approaches beyond model experiments remains open.

A separate area considered in the literature is the search for environmentally-friendly alternatives to polymer bases. Thus, in [16], the use of transparent wood films in combination with lignin-containing inks was proposed. The combination of flexibility, transparency, and mechanical strength was demonstrated; however, the work focuses mainly on unique ecological properties and does not analyze the behavior of such materials in scaled printing, where wettability and adhesion are key.

In [17], the application of inkjet printing of nanomaterials for RFID tags and sensors, in particular using paper substrates, was systematized. However, the authors emphasize the functional capabilities of IoT systems and almost do not consider the influence of moisture or mechanical loads on the conductivity of printed tracks.

In [18] the approaches to the design of paper electrochemical sensors based on inkjet printing were summarized. The authors note the low cost, the possibility of forming complex microfluidic structures, as well as the high selectivity of electrochemical measurements, which opens the way to mass production of disposable biosensors. However, the emphasis of the work is on demonstration examples of sensors for environmental diagnostics and monitoring, while the issue of long-term conductivity of conductive tracks on cellulose substrates in scaled production processes remains practically unaddressed.

Our review of the literature [4, 8, 9, 11, 13] confirms that the formation of stable conductive structures in printed electronics is determined by the complex interaction among a number of factors – surface energy, substrate microrelief, ink rheological properties, and printing modes. Despite significant progress, most works highlight only individual aspects – optimization of paste composition, improvement of adhesion or improvement of equipment – without their comprehensive combination. The influence of surface pre-treatment methods, in particular corona discharge treatment, has been studied in detail for polymer and specialized substrates but its effectiveness in combination with paper substrates has not been thoroughly studied. In turn, studies on screen printing with graphene pastes confirm the promising nature of the method but ignore the specific problems of paper associated with heterogeneity and hygroscopicity. Available reports demonstrate progress in reducing the drying temperature and increasing conductivity but almost do not analyze the long-term stability of structures under real operating conditions.

Therefore, a general unsolved task is to determine optimal corona discharge treatment regimes capable of enabling stable adhesion and conductivity of high-viscosity graphene pastes screen-printed on paper substrates. Solving this task is a key prerequisite for the reproducible and industrially relevant use of cellulose substrates in flexible printed electronics.

3. The aim and objectives of the study

The aim of our work is to determine the effect of corona discharge treatment as a method for preparing the surface of paper substrates on the quality of screen printing of conductive structures based on graphene. This will make it possible to define paper processing modes in order to significantly increase its adhesive properties and obtain high-quality conductive layers for the production of flexible printed electronics by screen printing.

To achieve this aim, the following objectives were accomplished:

– to investigate changes in the surface energy and morphology of paper substrates after corona treatment at different power levels;

– to compare the electrical characteristics and mechanical stability of printed structures on treated and untreated paper;

– to investigate changes in the properties of conductive tracks under the influence of simulated external factors;

– to conduct a comprehensive assessment of substrate processing options taking into account the influence of loads.

4. The study materials and methods

4. 1. The object and hypothesis of the study

The object of our study is the conductive layers of printed electronics based on graphene plastisol ink, applied by screen printing on glossy and matte paper substrates, pre-modified by corona discharge.

The principal hypothesis of the study assumes that changing the corona discharge treatment parameters (in particular, power) affects the morphology and surface energy of paper substrates, which in turn improves the wettability and adhesion of the ink, reduces the resistivity of conductive tracks, and increases their stability under the influence of mechanical, thermal, and chemical loads.

Assumptions adopted: the structure and composition of graphene ink remain constant throughout the experiment; the influence of other technological parameters of screen printing (printing speed, ink blade pressure, drying temperature) on conductivity and wear resistance is insignificant compared to the influence of corona treatment parameters.

Simplifications accepted: the influence of humidity and ambient temperature during printing and testing was not taken into account; analysis focuses only on two types of paper substrates (glossy and matte paper) and two corona treatment power levels (1000 W and 3000 W), without considering other treatment options or additional surface modifications.

Table 2

Characteristics of conductive ink for screen printing [20]

Parameter	Unit of measurement	Value	Test method
Type of ink	–	Conductive, plastisol screen printing ink	–
Conductive component	–	Graphene	–
Solvent	–	UG-002H (standard drying)	–
Ink/solvent	%	100/5–15	–
Drying conditions	°C/min	50–60/10	–
Visual appearance	–	Free from defects such as foreign matter, craters, orange peel, etc.	–
Resistivity	Ω	100–3000	IEC 60093:1980
Recommended film thickness	μm	> 5	ISO 2808:2019
Mesh size	tpi	60–90	–
Adhesion (100 × 2 mm^2)	–	Nichiban tape tightening at 45°C. No peeling	ASTM D3359-22
Hardness (should not peel off from a class F pencil)	–	Passed	ISO 15184:2012
Alcohol resistance (80% ethanol) 1 kgf/m^2 × 10 cycles	–	Passed	ISO 2812-1:2007
Abrasion resistance (rubber method) 1.64 kgf × 35 cycles at film thickness > 5 μm	–	Passed	ISO 7784-2:2006
Lubricity resistance (Vaseline – 40°C × 95% RH × 24 hours)	–	Passed	ISO 2812-2:2007
Substrate	–	Plastic, paper, ceramics, metal and fabric	–

4. 2. Materials

To conduct experiments aimed at improving the adhesion properties of different types of paper surfaces, samples were selected that are characterized by the type: glossy and matte. The corresponding samples and their parameters are given in Table 1.

Table 1

Paper substrate properties

Characteristics	Unit of measurement	Value		Test method
		Type 1	Type 2	
Type	–	Glossy	Matte	–
Weight	1 m^2, g	115	TAPPI. T. 410 om23, ISO 536:2019	
Brightness	%	89%	ISO 2470-1:2016	
Roughness	nm	1.4	2.7	ISO 4287:1997
Opacity	%	95%	ISO 2471	
Gloss	%	65	32	TAPPI T 480 om-20

The substrates were grouped according to the type of paper: Group 1 included glossy paper, Group 2 included matte paper.

A special plastisol graphene ink [19] was used. Preparation of the ink for printing: 4–5% of an oil-based solvent for plastisol inks was used per 100 g of ink. The characteristics are given in Table 2.

The given ink is a common option for applying simple conductive elements.

4. 3. Surface treatment with corona discharge

Corona treatment was performed using a corona generator in an Ahlbrandt flexographic machine [6].

The substrates were divided into groups according to the paper and mode – power 1000 and 3000 W, processing speed 50 m/min.

Designation of the sample group:
– M0 – matte paper without treatment;
– M1000 – matte paper under a processing mode of 1000 W;
– M3000 – matte paper under a processing mode of 3000 W;
– G0 – glossy paper without treatment;
– G1000 – glossy paper under a processing mode of 1000 W;
– G3000 – glossy paper under a processing mode of 3000 W.

4. 4. Application of conductive layers by screen printing

The application of conductive layers was carried out using a standard screen printing process, in accordance with the requirements of the conductive ink supplier [19], the main ones of which are:
– mesh resolution 150 tpi;
– drying mode 45–60°C in a drying cabinet for 10–15 min.

Adobe Illustrator software was used to design; CtF technology – to shape.

The topology of the mask is shown in Fig. 1.

Thus, we aim to compare different track configurations.

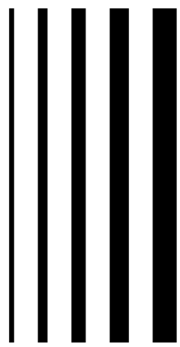


Fig. 1. An image exposed on a photosensitive layer of a stencil for applying conductive tracks

4. 5. Methods for studying the properties of the printed surface

4. 5. 1. Contact profilometry

In the study, contact profilometry is used to analyze the surface profile of paper substrates and determine parameters R_a, R_z, $R_{\max}$ (according to ISO 4287:1997).

The roughness of the samples was measured using a MarSurf PS1 profilometer [21]. A probe with a needle radius of 2 µm was used; the measuring force was 0.7 mN; and the tracing length was 4 mm.

Surface analysis method: the paper surface is analyzed using a diamond needle. This needle builds an enlarged image of the surface profile, providing a detailed idea of the surface topography.

Measurement conditions: measurements are performed at a sensor movement speed of 0.15 mm/s. This speed is selected to balance the accuracy and efficiency of data acquisition [21].

This contact profilometry procedure is necessary to determine microscopic changes in the paper surface after processing. The detailed surface profile data obtained will be valuable for correlating surface texture with ink adhesion.

Parameters measured:

– R_a – average roughness value – arithmetic mean of absolute values of the ordinates of the roughness profile (DIN EN ISO 4287);

– R_z – height of profile irregularities – average height of the five largest profile irregularities on the base length segment. It is defined as the sum of the height of the five largest protrusions and the depths of the five largest depressions, divided by five (DIN EN ISO 4287);

– $R_{\max}$ – maximum profile height – the largest vertical distance between the highest peak and the deepest depression of the roughness profile on the base length (DIN EN ISO 4287).

Profilometry of six groups of paper samples was carried out. Each sample in the group has a certain marking and codes. Number of samples – 6 groups of 3 samples. Number of measurements – 10 measurements along the direction and 10 across the direction for each of the samples.

4. 5. 2. Optical microscopy

Optical studies of the samples were carried out using a Sigeta Biogenic Lite 40× microscope [22].

Visualization and calibration were carried out in the ToupView software [20].

The surface structure of the paper was investigated in groups of treated and untreated samples.

After applying conductive tracks to the paper, the surface of the applied layer was also examined using a microscope, which was carried out in three areas along each studied applied conductive track, to obtain generalized information along the entire track.

The size of the samples for measurement was 80 × 80 mm. Magnification was 40×.

4. 6. Study of the resistance of conductive layers

The resistance of the applied tracks was measured using a laboratory multimeter Dnipro-M SM600 [23]. To avoid the negative effect of damage to the track surface due to the soft surface profile, it is proposed to use copper terminals, which ensure the absence of distortion of values during measurements. The measurement is carried out at a distance of 0.5 cm from the edges of the conductive tracks.

4. 7. Methods of wear imitation

The study of resistance to thermal effects was carried out by simulating thermal wear in a thermal installation, Tabai Espec MC-71 Mini Subzero [24], designed for testing in a wide range of temperatures and humidity. Such treatment is typical for annealing thin films.

The main parameters of the installation:

– temperature range: from – 70°C to + 100°C;

– temperature control accuracy: ± 0.3°C;

– cooling method: mechanical cooling;

– heating method: electric heater.

The studies used thermal cycles:

– heating – 10 cycles, + 60 degrees Celsius, cycle duration 30 min;

– cooling – 10 cycles, – 40 degrees Celsius, cycle duration 30 min.

This methodology involves measuring electrical properties before conducting thermal cycles.

After the thermal cycles are completed, a control measurement of the electrical properties is performed, as well as a microscopic examination of the surface of the applied tracks.

To simulate the wear of the samples, a methodology was used that was previously applied to study the wear resistance of banknote products [25]: mechanical impact is provided by contact with a wear agent (glass beads) inside a drum on a rotary mechanism. The duration of wear is 2 cycles of 10 minutes each.

The test of the impact of substances that imitate human sweat was also carried out according to the methodology from [25] with the addition of a pollutant. The duration of wear is 2 cycles of 10 minutes each. The use of this type of wear is justified by one of the target uses of printed, flexible electronics products – the medical field, where contact of products with the body, and, as a result, body fluids, is expected.

5. Results of investigating the effect of corona treatment on the conductive layers of electronics

5. 1. Surface morphology and chemical changes

Fig. 2–4 show the average values of parameters R_a and R_z for different paper samples, measured along and across the machine direction. After corona treatment, an increase in the average surface roughness is observed.

Fig. 4 demonstrates that the average value of $R_{\max}$ in the group of samples M1000 is the largest compared to all other investigated variants.

The results of our studies conducted using optical microscopy are shown in Fig. 5.

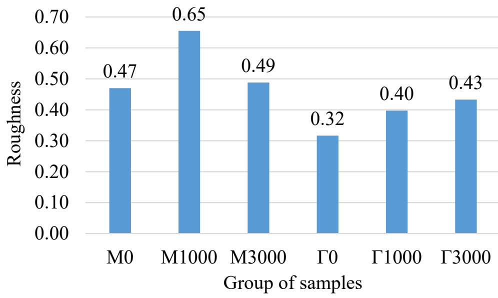


Fig. 2. Average values of indicators R_a at a 95% confidence interval

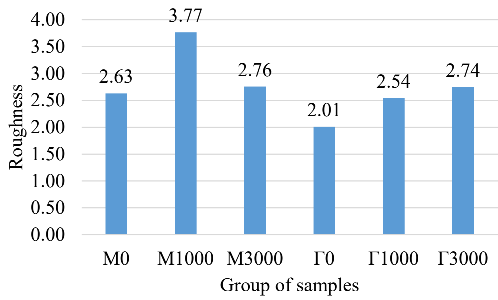


Fig. 3. Average values of indicators R_z at a 95% confidence interval

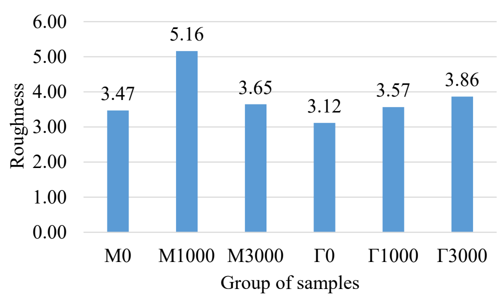
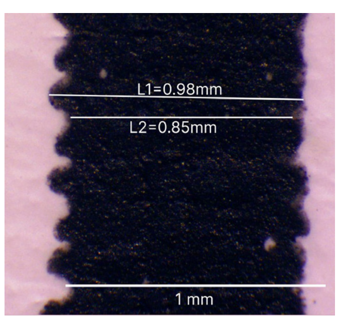
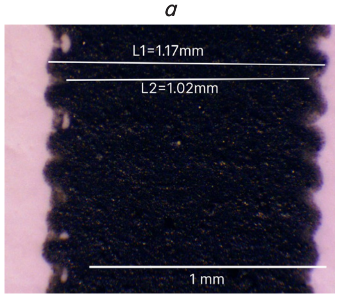


Fig. 4. Average values of indicators R_{max} at a 95% confidence interval



a



b

Fig. 5. Typical surface of the applied conductive material on the samples, magnification 40×: *a* – a sample from group M0; *b* – a sample from group M1000

Due to the viscosity and natural spreading of the paste during the drying stage, the coating adapts to the irregularities of the substrate, which enables the integration of the layer with the substrate. The surface looks homogeneous, with minimal local irregularities.

5. 2. Electrical characteristics of printed tracks

To assess the stability of conductive properties and detect outliers, all resistance measurement results were subjected to statistical processing with the calculation of the average value ($\bar{x}$) and standard deviation (s). Values that fell outside the $(\bar{x}) \pm 2s$ interval were considered outliers and were excluded from the calculation of the adjusted average values.

The processing showed that in 29 out of 30 combinations (6 groups × 5 widths) the resistance values are within the permissible statistical spread. The exception is only one outlier in the G0 group for a width of 2 mm (Table 3). This confirms the stability of the coating process and the absence of systematic technological errors.

Table 3

Results of statistical analysis

Group	Track width, mm	$\bar{x}$, Ohm	s, Ohm	Interval $(\bar{x}) \pm 2s$, Ohm	Outlier value
G0	2	542.33	136.71	[268.91; 815.75]	826

After eliminating singlets, all average resistance values were recalculated. A decrease in average resistance is observed as a result of corona discharge treatment. The results of our study of screen-printed conductive tracks are given in Table 4.

Table 4

Results of track resistance measurements based on width

Group of samples	5 mm	4 mm	3 mm	2 mm	1 mm
G0	169.2	219.2	319.5	516.5	1353.8
G1000	176.3	220.3	316.3	523.1	1280.6
G3000	161.1	218.3	310.1	507.5	1275.8
M0	184.0	248.6	346.7	589.8	1447.1
M1000	181.9	234.8	319.8	520.3	1232.4
M3000	161.1	201.1	267.5	425.1	1035.6

The lowest resistance values were obtained for samples M3000, i.e., for matte paper treated at 3000 W. Compared with untreated analogs (M0), the decrease in resistance reached more than 25% on wide tracks (5 mm) and more than 30% on narrow ones (1 mm).

5. 3. Change in the properties of conductive tracks under the influence of simulated external factors

The results of the change in resistivity for different groups of samples (G0, G1000, G3000, M0, M1000, M3000) are shown in Fig. 6–10. Analysis of the change in resistance allows us to assess the influence of the type of substrate and corona discharge treatment mode on the resistance of conductive tracks to mechanical loads.

Analysis of the acquired data reveals that samples on matte paper treated at a power of 3000 W (M3000) demonstrated the smallest increase in resistance after mechanical loading. Samples on glossy paper without treatment (G0) showed the largest increase in resistance. Corona discharge treatment for both matte and glossy paper allowed us to reduce the rate of degradation of electrical properties. The diagrams in Fig. 11–15 show the change in resistance of conductive tracks 1–5 mm wide for six groups of paper samples (glossy and matte, with different treatment modes), after passing through two temperature cycles (heating to + 60°C and cooling to – 40°C).

The most pronounced effect is observed in samples that did not undergo corona discharge pretreatment (M0, G0). In samples with pretreatment (especially M3000 and G3000), the changes were smaller.

After the second cycle, the resistance in some cases increases slightly. However, in most cases, after the first cycle, the resistance remained stably lower than the initial level.

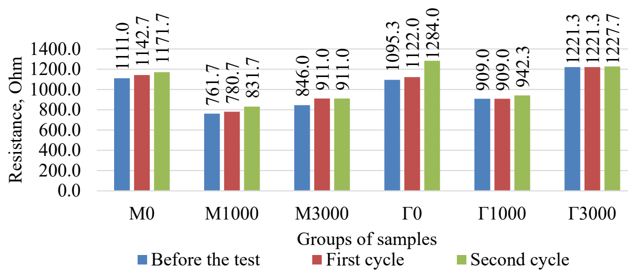


Fig. 6. Resistance change after mechanical tests for all groups of samples (for track width 1 mm)

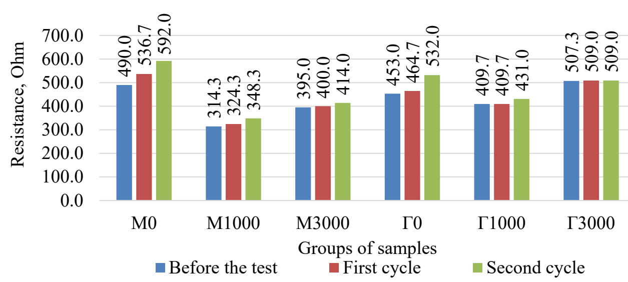


Fig. 7. Resistance change after mechanical tests for all groups of samples (for track width 2 mm)

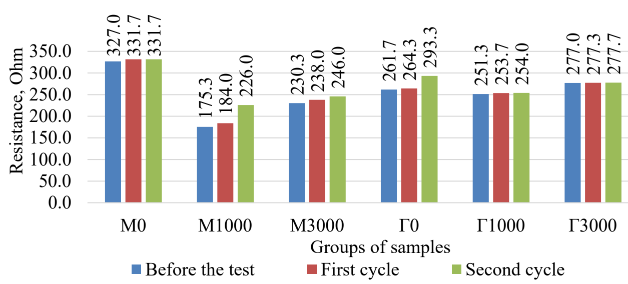


Fig. 8. Resistance change after mechanical tests for all groups of samples (for track width 3 mm)

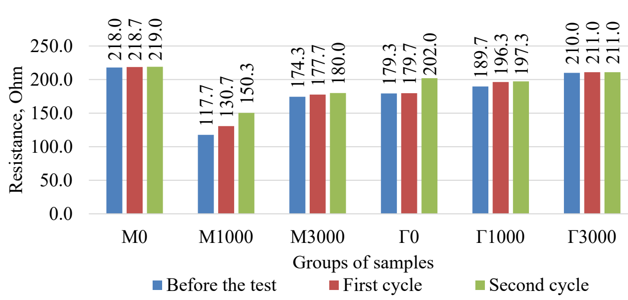


Fig. 9. Resistance change after mechanical tests for all groups of samples (for track width 4 mm)

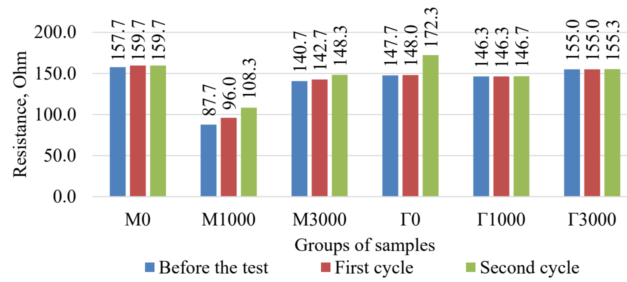


Fig. 10. Resistance change after mechanical tests for all groups of samples (for track width 5 mm)

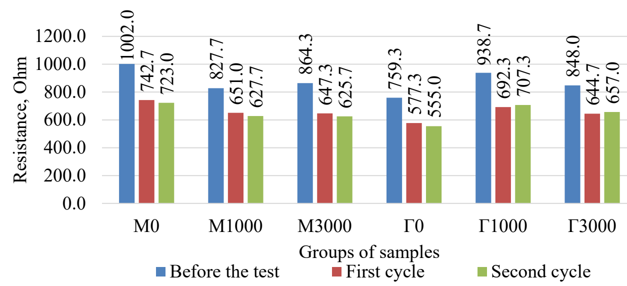


Fig. 11. Resistance change after thermal testing for all groups of samples (for track width 1 mm)

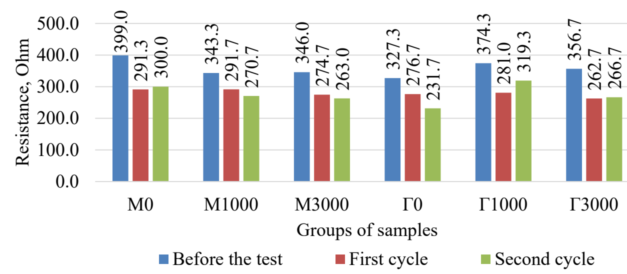


Fig. 12. Resistance change after thermal testing for all groups of samples (for track width 2 mm)

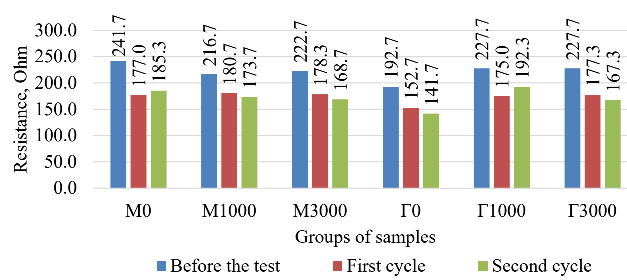


Fig. 13. Resistance change after thermal testing for all groups of samples (for track width 4 mm)

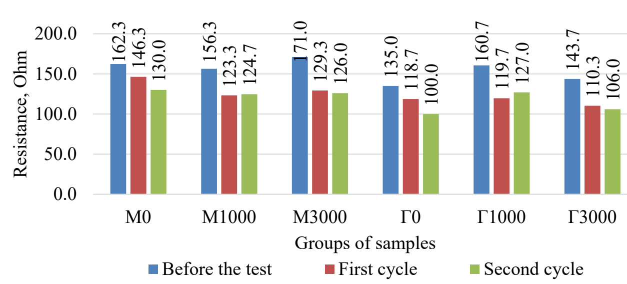


Fig. 14. Resistance change after thermal testing for all groups of samples (for track width 4 mm)

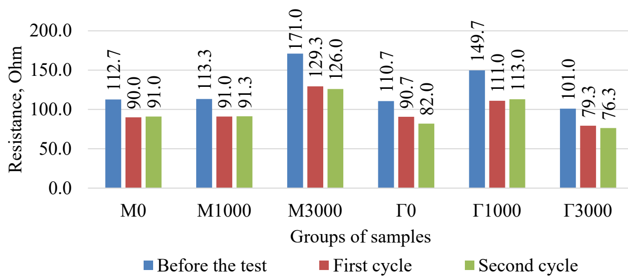


Fig. 15. Resistance change after thermal testing for all groups of samples (for track width 5 mm)

To assess the impact of simulated wear by contact with liquids, the proportion of failures in each group of samples after the first and second wear cycles was analyzed. A complete violation of conductivity was considered to be an excess of the resistance value above the measurement limit of the device (2000 Ohm). The results are shown in Fig. 16. In each group, the proportion of tracks that completely lost conductivity was taken into account, regardless of the width.

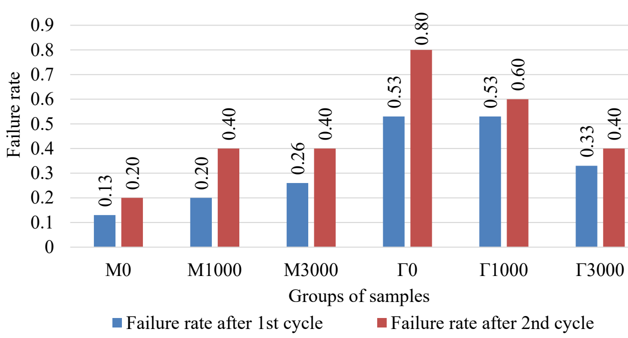


Fig. 16. Charts of failure rates after chemical tests

Analysis of the failure rate indicates a different reaction of the samples of the G and M series to external chemical influences.

For the G series, a pattern is clearly observed: an increase in the pre-treatment power (from 0 to 3000 W) leads to a decrease in the number of complete failures both after the first and after the second wear cycles.

In contrast, a similar dependence is not observed in the M series. On the contrary, the M0 samples demonstrate a lower failure rate than the M1000 and M3000.

After mechanical and thermal tests, no visual signs of degradation of the surface of the samples were detected. All the tested tracks retained their original structure, without the appearance of cracks, scratches, or discoloration. However, after the action of chemical reagents, a significant degradation of the appearance of the samples was observed. Most of the samples acquired a pronounced grayish hue due to the spreading of the conductive ink, scratches and local pits appeared on the surface, which violated the geometry of the applied tracks. Only a small part of the samples retained the integral shape of the tracks but, even in these cases, a decrease in the uniformity of the layer was noted.

5. 4. Comprehensive assessment of substrate processing options taking into account the influence of loads

Evaluation of wear resistance of a certain variant of manufacturing such tracks is considered as a multicriterion problem of selecting, as proposed in [26]. This is a case of making a decision on a set of permissible alternatives $X=\{x_i \mid i=\overline{1,m}\}$ – types of paper, corona discharge treatment modes, etc. In this case, the set of properties is taken into account, in particular the conductivity indicators before and after wear, which are characterized by a vector of objective functions $\overline{f}_i=\{f_j(x_i) \mid j=\overline{1,n}; x\in X\}$, where $f_j(x_i)$ corresponds to the j-th property by which the alternative $x_i \in X$ is evaluated. Hereafter, for convenience, we shall denote the partial objective function $f_j(x_i)=x_{ij}$.

Thus, it is proposed to evaluate the operational characteristics of the finished product by the set of properties measured before and after the tests. These include absolute resistance values (AVs); change in resistance (RC) due to imitation of mechanical (Mech) and thermal (Therm) effects, determined after the first (1c) and second (2c) cycles; failure rate (FR) of samples after the wear simulation cycles using a contaminated mixture. The values of these properties are given in Tables 5–9. Note that all properties are destimulators.

The decision-making process on a set of alternatives with several partial criteria is proposed to be carried out by reducing them to one generalized criterion – an integrated indicator of the operational properties of printed electronics elements. It is formed by convolution of many criteria and determination of the utility function. Formation of an integrated indicator of the operational properties of printed electronics elements taking into account the maximum possible number of indicators established before wear and at different stages and types of wear will allow for an objective selection of technological parameters.

Table 5

Resistivity values and resistance changes of printed tracks of different widths after the first cycle of mechanical wear (treated and untreated paper substrates)

Matrix x_{ij}		Property									
		Resistance of the track 5 mm, Ohm	Resistance of the track 4 mm, Ohm	Resistance of the track 3 mm, Ohm	Resistance of the track 2 mm, Ohm	Resistance of the track 1 mm, Ohm	RC, Mech, 1c, 5 mm	RC, Mech, 1c, 4 mm	RC, Mech, 1c, 3 mm	RC, Mech, 1c, 2 mm	RC, Mech, 1c, 1 mm
		x_{i1}	x_{i2}	x_{i3}	x_{i4}	x_{i5}	x_{i6}	x_{i7}	x_{i8}	x_{i9}	x_{i10}
G0	x_{1j}	169.2	219.2	319.5	319.5	319.5	0.3	0.3	2.7	11.7	26.7
G1000	x_{2j}	176.3	220.3	316.3	316.3	316.3	0.0	6.7	2.3	0.0	0.0
G3000	x_{3j}	161.1	218.3	310.1	310.1	310.1	0.0	1.0	0.3	–14.7	0.0
M0	x_{4j}	184	248.6	346.7	346.7	346.7	2.0	0.7	4.7	46.7	31.7
M1000	x_{5j}	181.9	234.8	319.8	319.8	319.8	8.3	13.0	8.7	10.0	19.0
MM3000	X_{6j}	161.1	201.1	267.5	267.5	267.5	2.0	3.3	7.7	5.0	65.0

Table 6

Changes in the electrical resistance of printed tracks after the second cycle of mechanical wear and after the first cycle of thermal cycling (treated and untreated samples)

Matrix x_{ij}		Property									
		RC, Mech, 2c, 5 mm	RC, Mech, 2c, 4 mm	RC, Mech, 2c, 3 mm	RC, Mech, 2c, 2 mm	RC, Mech, 2c, 1 mm	RC, Therm, 1c, 5 mm	RC, Therm, 1c, 4 mm	RC, Therm, 1c, 3 mm	RC, Therm, 1c, 2 mm	RC, Therm, 1c, 1 mm
		x_{i1}	x_{i2}	x_{i3}	x_{i4}	x_{i5}	x_{i6}	x_{i7}	x_{i8}	x_{i9}	x_{i10}
G0	x_{1j}	24.3	22.3	29.0	67.3	162.0	20.0	16.3	40.0	50.7	182.0
G1000	x_{2j}	0.3	1.0	0.3	21.3	33.3	38.7	41.0	52.7	93.3	246.3
G3000	x_{3j}	0.3	0.0	0.3	8.3	6.3	21.7	33.3	50.3	94.0	203.3
M0	x_{4j}	0.0	0.3	0.0	55.3	29.0	22.7	16.0	64.7	107.7	259.3
M1000	x_{5j}	2.8	1.3	4.0	34.7	14.5	22.3	33.0	36.0	51.7	176.7
M3000	X_{6j}	5.7	2.3	8.0	14.0	0.0	33.3	41.7	44.3	71.3	217.0

Table 7

Effect of the second thermal cycling cycle on the resistance of printed tracks and failure rate (treated and untreated paper substrates)

Matrix x_{ij}		Property						
		RC, Therm, 2c, 5 mm	RC, Therm, 2c, 4 mm	RC, Therm, 2c, 3 mm	RC, Therm, 2c, 2 mm	RC, Therm, 2c, 1 mm	FR, 1c	FR, 2c
		x_{i1}	x_{i2}	x_{i3}	x_{i4}	x_{i5}	x_{i6}	x_{i7}
G0	x_{1j}	8.7	18.7	11.0	45.0	22.3	0.5	0.6
G1000	x_{2j}	–2.0	–7.3	–17.3	–38.3	–15.0	0.5	0.1
G3000	x_{3j}	3.0	4.3	10.0	–4.0	–12.3	0.3	0.1
M0	x_{4j}	–1.0	16.3	–8.3	–8.7	19.7	0.1	0.1
M1000	x_{5j}	–0.3	–1.3	7.0	21.0	23.3	0.2	0.3
M3000	X_{6j}	–3.3	3.3	9.7	11.7	21.7	0.3	0.2

Table 8

Absolute values of electrical resistance of printed tracks after the first and second cycles of mechanical wear (treated and untreated samples)

Matrix x_{ij}		Property									
		AV, Mech, 1c, 5 mm	AV, Mech, 1c, 4 mm	AV, Mech, 1c, 3 mm	AV, Mech, 1c, 2 mm	AV, Mech, 1c, 1 mm	AV, Mech, 2c, 5 mm	AV, Mech, 2c, 4 mm	AV, Mech, 2c, 3 mm	AV, Mech, 2c, 2 mm	AV, Mech, 2c, 1 mm
		x_{i1}	x_{i2}	x_{i3}	x_{i4}	x_{i5}	x_{i6}	x_{i7}	x_{i8}	x_{i9}	x_{i10}
G0	x_{1j}	148.0	179.7	264.3	464.7	1122.0	172.3	202.0	293.3	532.0	1284.0
G1000	x_{2j}	146.3	196.3	253.7	409.7	909.0	146.7	197.3	254.0	431.0	942.3
G3000	x_{3j}	155.0	211.0	277.3	492.7	1221.3	155.3	211.0	277.7	501.0	1227.7
M0	x_{4j}	159.7	218.7	331.7	536.7	1142.7	159.7	219.0	331.7	592.0	1171.7
M1000	x_{5j}	96.0	130.7	184.0	324.3	780.7	108.3	150.3	226.0	348.3	831.7
M3000	X_{6j}	142.7	177.7	238.0	400.0	911.0	148.3	180.0	246.0	414.0	911.0

Table 9

Absolute values of electrical resistance of printed tracks after the first and second thermal test cycles (treated and untreated paper substrates)

Matrix x_{ij}		Property									
		AV, Therm, 1c, 5 mm	AV, Therm, 1c, 4 mm	AV, Therm, 1c, 3 mm	AV, Therm, 1c, 2 mm	AV, Therm, 1c, 1 mm	AV, Therm, 2c, 5 mm	AV, Therm, 2c, 4 mm	AV, Therm, 2c, 3 mm	AV, Therm, 2c, 2 mm	AV, Therm, 2c, 1 mm
		x_{i1}	x_{i2}	x_{i3}	x_{i4}	x_{i5}	x_{i6}	x_{i7}	x_{i8}	x_{i9}	x_{i10}
G0	x_{1j}	90.7	118.7	152.7	276.7	577.3	82.0	100.0	141.7	231.7	555.0
G1000	x_{2j}	111.0	119.7	175.0	281.0	692.3	113.0	127.0	192.3	319.3	707.3
G3000	x_{3j}	79.3	110.3	177.3	262.7	644.7	76.3	106.0	167.3	266.7	657.0
M0	x_{4j}	90.0	146.3	177.0	291.3	742.7	91.0	130.0	185.3	300.0	723.0
M1000	x_{5j}	91.0	123.3	180.7	291.7	651.0	91.3	124.7	173.7	270.7	627.7
M3000	X_{6j}	98.3	129.3	178.3	274.7	647.3	101.7	126.0	168.7	263.0	625.7

An additive convolution of indicators was used, in which all indicators are disincentives (1)

$$F(x_i)=\sum_{j=k+1}^{m}\omega_j\frac{x_{\min j}}{xij}. \quad (1)$$

We assume that the weight of each indicator is the same, then $\omega_i = 1/n$.

The utility functions of the alternatives are shown in Fig. 17.

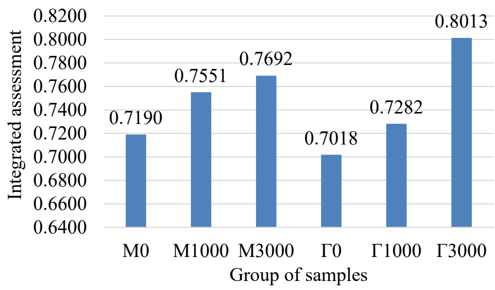


Fig. 17. Results of multifactorial assessment

According to the comprehensive evaluation, a trend towards increasing the efficiency of the samples with increasing corona discharge treatment power becomes visible.

6. Discussion of results related to the influence of corona treatment on conductive layers of electronics

Our results allow us to explain in detail the influence of corona discharge treatment parameters on the electrical and operational properties of conductive layers applied by screen printing on paper substrates.

The increase in surface energy and microroughness after corona discharge treatment (Fig. 2–4, Table 4) provided improved wetting and penetration of graphene ink into the micropores of the paper substrate. For matte paper, treatment at a power of 3000 W (M3000) gave the greatest effect: the resistivity of tracks with a width of 1 mm decreased by more than 30%, and 5 mm – by more than 25% compared to untreated samples. For glossy paper, the effect was less pronounced, which is explained by the lower ability of the smooth surface to retain the effect of functional groups after corona treatment.

The stability of conductivity after mechanical tests (Fig. 6–10) was the highest for M3000, where the increase in resistance did not exceed 5%. Analysis after thermal cycling (Fig. 11–15) confirmed that pre-corona reduces conductivity degradation, while in untreated samples (M0, G0) significant changes were observed due to densification and microcracks. Chemical tests (Fig. 16) showed a decrease in the failure rate in glossy samples after treatment, but for matte samples this effect was not unambiguous, indicating a specific interaction of the base material and the ink.

In contrast to the results reported in [14], in which the improvement of adhesion of metal layers after corona discharge treatment was demonstrated for magnetron sputtering, our data confirm the effectiveness of the method for screen printing of graphene pastes as well. Compared to the data from [9, 10], in which the optimization of printing was carried out mainly for polymer substrates, in this study, a decrease in resistance on paper substrates was achieved without complicating the technological process. This was made possible by combining corona activation of the surface with the properties of a highly viscous graphene ink that adapts to the microrelief of the paper.

Our results allow us to specify those aspects that remained unresolved in previous works. The literature noted the lack of studies combining morphological analysis of the surface with the assessment of electrical characteristics on paper substrates [11–13]; in our work, this relationship was traced by comparing the roughness parameters after corona treatment (Fig. 2–4) with the resistivity of the tracks (Table 4). Also, previous reviews [9, 10, 15] emphasized the limitations of studies on the long-term stability of conductive tracks, especially on cellulose carriers. The mechanical, thermal, and chemical tests we conducted (Fig. 6–16) confirmed that corona treatment at 3000 W enables the preservation of conductivity even after load cycles. Thus, the results of the work directly correlate with the problems identified in Section 2 – the lack of an interdisciplinary approach and the lack of applied research on the durability of conductive layers on paper – and demonstrate their partial solution for the conditions of screen printing of graphene pastes.

Comparison of our data with the literature results [4, 8] confirms that the effectiveness of corona discharge treatment is associated with an increase in the surface energy and wettability of the material; however, unlike studies on polymer substrates, our work traced the specific influence of paper heterogeneity and its microrelief on the stability of conductive tracks. While studies [9, 10] focused on the possibilities of screen printing of graphene inks, we showed that the combination of this method with optimal corona treatment (3000 W) provides not only high conductivity but also its preservation after thermal and mechanical loads. Thus, the results of our study not only confirm some conclusions of previous authors but also expand them, demonstrating the practical effectiveness of corona treatment specifically for cellulose substrates.

The issue of low adhesion and instability of conductive layers on paper substrates was resolved by using corona discharge treatment, which is confirmed by a decrease in resistivity (Table 4), stability after loads, and a decrease in the failure rate. The goal of the study – to determine effective treatment modes – has been achieved: it was found that the use of a power of 3000 W for matte substrates is effective. Thus, the proposed approach fills the existing niche in the production of flexible electronics on paper.

The results were obtained for a specific type of plastisol graphene ink and two types of paper with fixed technological printing modes. Changing the ink composition, type of substrate, or drying parameters may affect the effectiveness of corona treatment. Also, the study did not cover the effect of long-term storage of samples and aging under the influence of ultraviolet light.

The limited volume of series for statistical verification of some results, especially in terms of chemical tests, does not allow us to draw fully generalized conclusions for all types of paper substrates. For matte paper, the effect of reducing the proportion of failures after treatment was not stable, which requires additional explanation.

In the process of our studies, certain shortcomings were identified that may affect the completeness and universality of the results.

First, when measuring the electrical resistance, contact connection with copper terminals was used, which, despite caution, may introduce an error due to instability of the clamping force and microdamage to the surface of the conductive tracks. This drawback can be eliminated by applying non-contact measurement methods or using standardized spring clamps with controlled force.

Second, the study did not take into account the possible influence of non-uniformity of ink application within one sample, which could lead to local differences in conductivity. In the future, this drawback can be eliminated by automated control over layer thickness and the use of visual inspection systems during printing.

Third, during the simulated chemical wear tests, the pH and temperature of the working solutions were not controlled throughout the test, which could change the aggressiveness of the environment. To increase the reproducibility of the results, it is worth using climatic chambers with automatic adjustment of the solution parameters.

In addition, the test series were performed on a relatively small number of samples, which reduces the statistical reliability of the conclusions. Further studies should be conducted with a larger sample size and multifactorial experimental design to identify possible interactions between technological parameters.

Further work should be directed toward:

– expanding the range of paper and ink types under study, including different ink bases;

– studying the effect of combinations of corona treatment with other modification methods (plasma treatment, nanocoating);

– modeling the wear process to predict durability under actual operating conditions;

– optimizing printing parameters for ultra-thin conductive lines (< 0.5 mm) taking into account the effect of corona discharge activation.

Our results form a scientific and technological basis for adapting screen printing of graphene conductors on paper to the requirements of flexible electronics, as well as open up prospects for scaling the process in roll production.

7. Conclusions

1. Pretreatment of paper substrates with corona discharge changes their microrelief and increases wettability. The greatest effect was observed for matte paper at a power of 3000 W, when there was a moderate increase in average roughness and a stable increase in the height of irregularities, which created favorable conditions for ink fixation. The mechanism is the formation of polar functional groups on the surface and an increase in adhesion potential.

2. Surface modification with corona discharge provided a decrease in the resistivity of conductive tracks on matte paper by up to 30% for narrow lines and more than 25% for wide ones compared to untreated samples. This was possible due to the combination of increased microroughness and stable adhesion, which reduced the number of contact defects and improved layer uniformity.

3. Conductive layers applied to matte paper pre-treated at 3000 W demonstrated the smallest increase in resistance after mechanical and thermal tests – within 2–5%, while for untreated analogs this indicator reached 46%. For glossy paper, the effect was also positive but less pronounced. The increased stability is explained by a closer adhesion of the conductive layer to the base and a lower tendency to the formation of microcracks.

4. A comprehensive analysis of the operational characteristics revealed that the corona treatment mode with a power of 3000 W for matte paper is the optimal one. It provides a balance between low resistivity, high wear resistance, and stability after loads. Our result is attributed to the optimal depth of surface modification while maintaining the structural integrity of the base, which makes it possible to integrate the process into roll production without complicating the technological line.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study, as well as the results reported in this paper.

Funding

The study was conducted without financial support.

Data availability

The data will be provided upon reasonable request.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

Acknowledgments

The authors express their gratitude to Mykhailo Dusheyk, leading engineer at the Faculty of Electronics, and Oleksandr Anatoliyovych Plivak, head of the research (experimental) laboratory of measuring equipment at NN MMI, NTUU KPI named after Igor Sikorsky, for technical support in conducting the research.

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