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Investigation of the influence of technological parameters on the copper deposition process

Abstract. *The possibility of electroplating copper coatings on chemically and chemical-galvanically nickel-plated acrylic fibers has been studied. The effect of electrolyte pH, its composition, current strength at the 1st and 2nd cathodes, as well as the metallization time on the electrophysical, physical and mechanical properties of copper-containing fibers have been studied.*
Electrically conductive fibers with different copper contents have been obtained. Regularities have been established for the change in the electrical properties of fibers depending on the content of copper. The studies have shown that with an increase in the copper content, the electrical conductivity, the uniformity of the coating and the uniformity of the electrophysical properties (for chemical-galvanically nickel-plated fiber) increase. In the case of copper plating of chemically nickel-plated fiber, the coefficient of variation in electrical resistance increases with increasing plating time, despite the fact that the copper content increases, and the coefficient of variation in copper content and electrical resistance decreases.
Keywords: *electrically conductive chemical fibers, electrical conductivity, electrical resistance, electrolyte, electrodeposition.*

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Introduction

Today, all leading manufacturers of metallized fabrics use nickel as a metal coating. This metal is a ferromagnet, so it reflects well the magnetic component of electromagnetic radiation [1].

Personal protective equipment against electromagnetic radiation includes head protection equipment (protective helmets, hats), eye and face protection (goggles and protective shields), respiratory protection equipment (gas masks and respirators), hand protection (mittens), means of protection against falling (safety belts and safety ropes) and special protective clothing (suits, gowns, aprons, vests) [2].

Electrically conductive chemical fibers, which are marketed by several leading countries, differ significantly in the way they are obtained, the nature of the conductive components used and of their distribution in the polymer mass. The areas of application of such fibers are constantly expanding and their demand is increasing. The development of new technologies to produce electrically conductive fibers and the study of their properties are urgent tasks of modern polymer chemistry and materials science [3].

Metal coatings are applied to dielectrics either by vacuum spraying or by precipitation from solutions, and the latter method is more versatile and can be used for metallizing products of complex shape and, if necessary, obtaining metal coatings with specified functional properties [4].

In the scientific article, antimicrobial silver ions were introduced into knitted and non-woven fabrics using the methods of dry curing with a swab and spraying. The absorbency of nonwoven fabric is significantly higher than that of other knitted fabrics before and after antimicrobial treatment [5].

The work "Development of electroless silver plating on Para-aramid fibers and growth morphology of silver deposits" investigated the electrodeless silvering of para-aramid fibers and the morphology of the growth of silver deposits. The results showed that a higher silver mass gain was beneficial for improving the conductivity of paraaramide fibers deposited with silver [6].

Electromagnetic Shielding Fabric (ESF) is a new electromagnetic shielding product with portability, flexibility and good mechanical properties [7].

The occurrence of static charges on the clothes of medical workers is facilitated by many electrical appliances in modern medical centers, low air humidity, as well as the background level of ionizing radiation. This problem is most relevant for clinical laboratories, physiotherapy and diagnostic rooms. The possibility of using the method of magnetron sputtering of metals on textile materials was studied for the manufacture of medical clothing to impart antistatic properties, provided that their physical and hygienic characteristics are preserved [8].

Applying a metal coating to fibers using supercritical fluid (SCF) is a manufacturing technology used to produce highly conductive fibers; further research is currently being actively conducted on synthetic fibers such as aramid, polyester and nylon fibers [9].

The revision of literature and patent data on the production, properties and use of electrically conductive fibers and materials based on them shows that a lot of attention is paid to the problem of obtaining electrically conductive fibers and products. This is primarily due to the fact that such valuable properties as low density, elasticity, chemical resistance, electrical conductivity, relatively low cost, successfully compete with metal fibers and allow solving urgent problems in various sectors of economy. Currently, to produce metallized fibers, chemical and electroplating metallization methods are most often used, which make it possible to give the fibers a sufficiently high electrical conductivity while maintaining or slightly reducing their strength. However, there are a limited number of publications on the possibility of applying galvanic copper coatings to chemical fibers. The purpose of the current work is to study the possibility of applying a copper coating by electroplating on a pre-chemically and chemical-galvanically nickel-plated fiber, as well as to conduct a set of experiments to study the properties of the resulting copper-containing fiber and comparing the data obtained with the original sample. Sufficiently detailed studies of the influence of technological parameters on the deposition of copper on chemically and chemical-galvanically nickel-plated fiber were carried out [10].

Materials and Methods

The fabric used is pre-chemically and chemically electroplated nickel-plated acrylic fiber. The purpose of this study was to study the possibility of applying a copper coating by electroplating on a pre-chemically and chemically-galvanically nickel-plated acrylic fiber and to study the properties of copper-containing fibers.

From the whole variety of electrolytes, we selected the most commonly used thre electrolytes. The compositions of electrolytes and the recommended parameters of their operation are presented in Table 1.

Table 1. **Electrolyte compositions and recommended parameters**

Electrolyte №1 sulfate	Electrolyte №2 pyrophosphate	Electrolyte №3 ethylenediamine
$CuSO_4 \cdot 5H_2O$ - 200 g/L H_2SO_4 (density 1.84 g/cm^3) - 50 g/L pH = 1 T= 20-30ºC	$CuSO_4 \cdot 5H_2O$ – 30 g/L $Na_4P_2O_7 \cdot 10H_2O$ - 120 g/L $Na_2HPO_4 \cdot 12H_2O$ - 60 g/L pH = 7.5-8.9 T = 20-30ºC Current density - 0.3-0.4 A/dm^3 Currentoutput-75-80%	$CuSO_4 \cdot 5H_2O$ – 95 g/L $C_2H_4(NH_2)_2$ - 50 g/L $(NH_4)_2SO_4$ - 50 g/L $Na_2SO_4 \cdot 10H_2O$ - 50 g/L pH = 6-7.8 T = 20-30ºC

Investigation of the influence of technological parameters on the deposition of copper on chemical and chemical-galvanic nickel-plated fiber. The properties of copper coatings and their structure depend on the deposition conditions. In terms of physical properties, electrolytic copper differs from copper in an equilibrium state. Copper obtained by electrodeposition has increased hardness, electrical resistance and internal voltage.

Depending on the composition, type of electrolyte and structure of copper precipitation, its physical and mechanical properties can vary widely. In most cases, copper coatings are used when applying multilayer coatings such as copper-nickel-chromium or nickel-copper-nickel, etc.

Results and Discussion

Table 2 presents the results obtained in the study of the process of applying copper to chemically nickel-plated acrylic fiber in the above electrolytes. The data presented in Table 2 indicate that, other parameters being equal, the most favorable conditions for the deposition of copper on a nickel-containing fiber are created when using a pyrophosphate electrolyte. It was also found that when copper is deposited on a chemically nickel-plated fiber, the deposited metal is rather quickly oxidized in air with the formation of CuO and Cu_2O. When copper is deposited on chemical-galvanically nickel-plated fiber, the copper coating is more uniform and more resistant to oxygen. Based on the analysis of the data obtained, when studying the influence of various parameters on the process of electrolytic deposition of copper, further experiments were carried out using a pyrophosphate electrolyte.

Table 2. **Results of the study of the process of applying copper to nickel-plated acrylic fibers in various electrolytes**

Type of electrolyte and fiber	Maximum current on the 1st cathode, A/ voltage, V	Maximum current on the 2nd cathode, A/ voltage, V	Metalliza-tiontime, min	Coppercon-tentinfiber, %	Note
№1, pH=1 Chemical nickel plated Chemical-galvanically nickel-plated	1.5/6 2.0/10	1.5/10 3/16	12 6	2.3 3.9	1) Copper is evenly deposited on the chemical-galvanically nickel-plated fiber. On chemically nickel-plated fiber, copper oxidizes rapidly. 2) Over time, copper begins to deposit on the cathode roller.
№2, pH=7.5 -8.9 Chemical nickel plated Chemical pebbles nickel-plated	3/16 3/10	6/17 5/16	12 6	4.3 5.6	1) Copper is evenly deposited on the chemical-galvanically nickel-plated fiber. On chemically nickel-plated fiber, copper oxidizes rapidly. 2) No copper deposition on cathode rollers
№3, pH=6-7.8 Chemical nickel plated Chemical pebbles nickel-plated	1/15 1/15	1.5/18 1.5/18	12 6	Copper is not deposited on the fiber, it is deposited on the cathode rollers	High voltage on the cathode rollers, copper is not fixed on the fiber. Copperisdeposit edonthecathoderollers

The effect of current on the 1st and 2nd cathodes on the amount of copper deposited on the fiber, on the electrical resistivity of the fiber and on the uniformity of the copper coating was studied. The data obtained are presented in Table 3.

An analysis of the results obtained indicates that with an increase in the current strength at the 2nd cathode, the amount of copper deposited on the fiber increases, and the electrical resistivity of the fiber decreases accordingly. This improves the uniformity of the coating (KV_{Cu} decreases) and increases the uniformity of electrical properties ($KV\ \varrho_v$ also decreases).At the same time, the uniformity of the coating improves (KV_{Cu} decreases) and the uniformity of the electrophysical properties increases (KV_{pv} also decreases).

Table 3. **The effect of current on the 1st and 2nd cathodes on the properties of the fibers, subjected to electrolytic copper plating**

Fibertype	Current strength on the 1st cathode, i1, A	Current strength on the 2nd cathode, i2, A	Copper content on fiber, Ccu, %	Coefficient of variation of copper content, KV $_{Cu}$, %	Specific electrical resistance of the fiber, ϱv·10⁻⁶Ohm·m	Coefficient of variation, KV ϱv, %
Chemical-galvanically nickel-plated fiber	1 1 1	3 4 5	3.74 4.42 4.74	41.2 33.3 26.4	12.3 7.67 7.39	51.4 41.3 32.8
Chemical-galvanically nickel-plated fiber	2 2 2	3 4 5	3.85 5.25 6.90	28.3 24.5 23.2	11.7 6.4 6.06	40.6 40.0 39.4
Chemical-nickelplatedfiber	1 1 1	3 4 5	4.06 5.2 5.6	19.8 33.7 42.1	592.9 553.9 507.2	52.8 38.6 27.4

It should be noted that when applying a copper coating on a chemical-galvanically nickel-plated fiber, increasing the current on the 2nd cathode to more than 4–5 A is not advisable, since this does not lead to a further decrease in resistance (Figure 1). Increasing the current on the 1st cathode up to 2 A, makes it possible to slightly increase the amount of copper deposited on the fiber and the electrical conductivity of the resulting fibers.

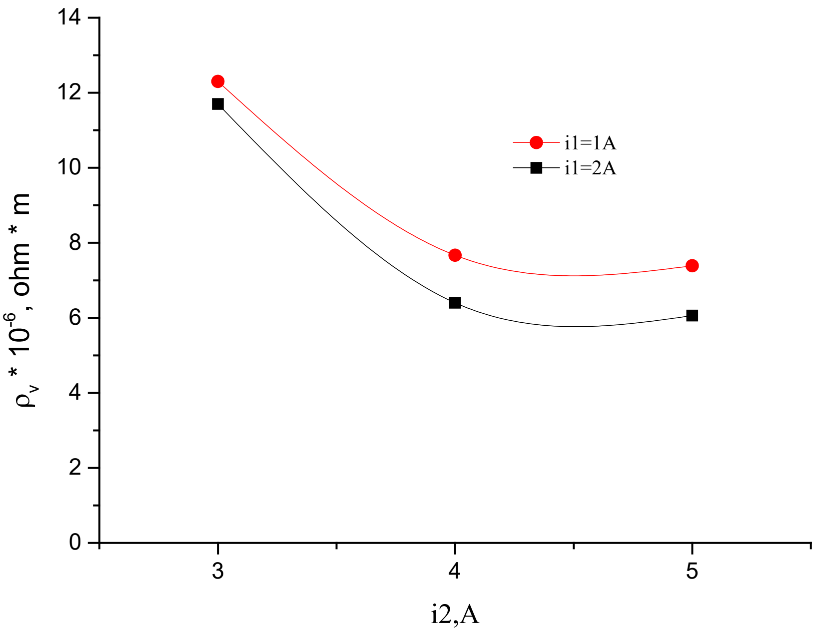

Figure 1. Influence of the magnitude of the current on the 1st and 2nd cathodes on the properties of chemical-galvanic nickel-plated fiber subjected to electrolytic copper plating, at a) i1 = 1 A; b) i1 = 2 A

For chemically nickel-plated fiber (Figure 2), the electrical conductivity of the fiber increases linearly with an increase in the current strength at the 2nd cathode. At the same time, to increase the current strength above 5 A, it is necessary to increase the voltage to values exceeding 16 V, which negatively affects the process of galvanic copper plating.

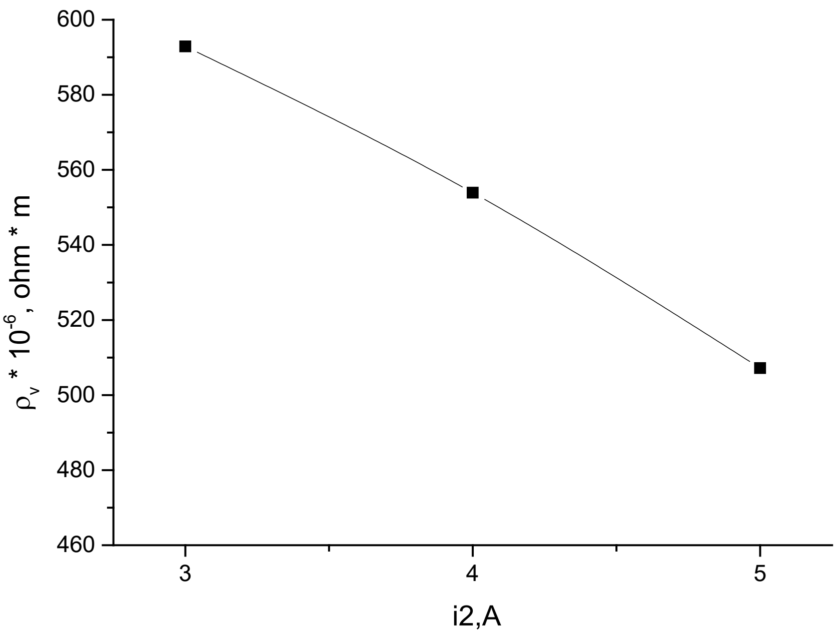

Figure 2. Influence of the current value on the 2nd cathode on the properties of chemical nickel-plated fiber subjected to electrolytic copper plating, at i1 = 1 A

It should also be noted that in the case of copper plating of chemically nickel-plated fiber, the electrical conductivity is 1.5–2 decimal orders lower than in the case of copper coating of chemical-galvanically nickelized fiber with an approximately equal content of galvanic copper. This is due to the different electrical resistance of the original fibers subjected to metallization ($ϱv_{\text{chemically nickel-plated fiber}}$ = $5·10^{-5}$ Ohm·m, $ϱv_{\text{chemically nickel-plated fiber}}$ =$5·10^{-6}$ Ohm·m). As in the preliminary experiments, it was observed that the copper coating on chemically nickel-plated fiber is less resistant to weathering and oxidizes rather quickly ($pv_{\text{chemically nickel-plated fiber}}$ =$5×10^{-5}$ Ohm·m, $pv_{\text{chemically nickel-plated fiber}}$ =$5×10^{-6}$ Ohm·m). It should also be noted that when coppering a chemically nickel-plated fiber, the electrical conductivity is 1.5-2 decimal orders of magnitude lower than in the case of a copper coating of a chemically galvanically nickel-plated fiber with an approximately equal content of galvanic copper. This is due to the different electrical resistance of the initial fibers subjected to metallization ($pv_{\text{chemical-galvanic nickel-plated fiber}}$ = $5×10^{-5}$ Ohm·m, pv_{hcv} = $5×10^{-6}$ Ohm·m).

The influence of the electrolysis time on the amount of deposited copper and, accordingly, on the electrical volume resistance of nickel-plated fibers was also studied. The results obtained (Table 4) indicate that for chemical-galvanically nickel-plated fiber, with an increase in the copper content, the electrical conductivity increases (Figure 2), as well as the uniformity of the coating and the uniformity of the electrical properties (Figure 3).

Table 4. **Effect of plating time on the properties of fibers subjected to electrolytic copper plating**

Fibertype	Metallizationtimemin.	Copper content on fiber, Ccu, %	Coefficient of variation of copper content, KV_{Cu}, %	Specific electrical resistance of the fiber, ϱv·10^{-6}Ohm·m	Coefficient of variation, KV ϱv, %
Chemical-galvanically nickel-plated fiber	4 6 8 10	3.4 5.25 8.3 11.7	27.7 24.5 16.8 13.8	10.1 6.4 5.9 5.2	45.3 38.1 34.0 26.3
Chemical-nickelplatedfiber	10 12 18 24	4.83 6.2 13.2 21.2	28.9 30.0 27.2 28.0	386.7 320.4 216.8 206.0	35.5 52.0 86.6 107.7

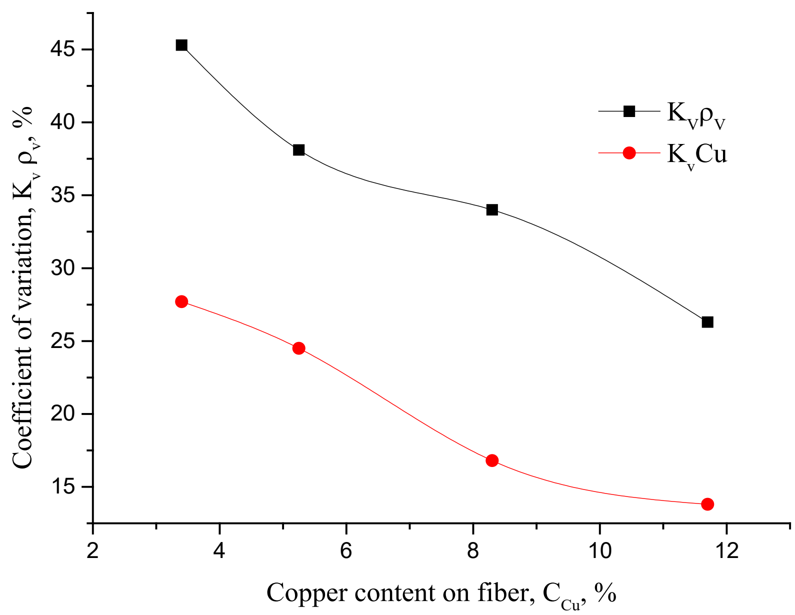

Figure 3. Dependence of the coefficients of variation for copper content (KVCu) and electrical resistance (KV ϱv) on the amount of copper (C_{Cu}) deposited on chemical-galvanic nickel-plated fiber

During copper plating of chemically nickel-plated fiber, the coefficient of variation in electrical volume resistance increases with increasing copper content, while the coefficient of variation in copper content and electrical resistance decrease (Figures 2 and 4). This can apparently be explained by the fact that the oxidation of galvanic copper during the drying process is more intense, the greater its amount, and this is due to the direct contact of galvanic copper with nickel sulfide, which is always present in chemically nickel-plated fiber.

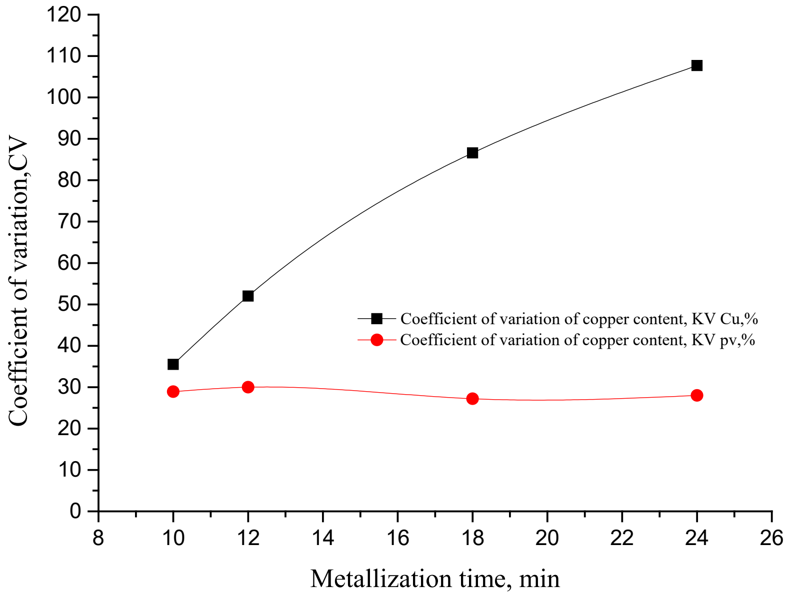

Figure 4. Dependence of coefficients of variation for copper content (KVCu) and electrical resistance (KV ϱv) on the amount of copper (C_{Cu}) deposited on nickel-plated fiber

In the case of copper plating of chemical-galvanically nickel-plated fibers, there is no such contact, since galvanized copper is in contact with the galvanized nickel coating, which explains the higher resistance of the copper coating.

Conclusions

According to the results of this study, electrically conductive fibers with different copper content were obtained. The regularities of changes in the electrophysical properties of fibers depending on the copper content in them are established;

Studies have shown that with an increase in the copper content, the electrical conductivity, the uniformity of the coating and the uniformity of the electrophysical properties (for chemically galvanically nickel-plated fiber) increase. In the case of coppering of chemically nickel-plated fiber, the coefficient of variation in electrical resistance increases with increasing metallization time, despite the fact that the copper content increases, and the coefficient of variation in copper content and electrical resistance fall.

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Мыстың тұндыру процесіне технологиялық параметрлердің әсерін зерттеу

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Андатпа. Химиялық және химиялық гальваникалық никельмен қапталған акрил талшықтарына мыс жабындарын қолдану мүмкіндігі зерттелді. Құрамында мыс бар талшықтардың электрофизикалық, физика-механикалық қасиеттеріне электролит pH, оның құрамы, 1-ші және 2-ші катодтардағы ток күші, сонымен қатар металдану уақытының әсері зерттелді.

Құрамында әртүрлі мыс бар электр өткізгіш талшықтар алынды. Мыстың құрамына байланысты талшықтардың электрлік қасиеттерінің өзгеру заңдылықтары белгіленді. Зерттеулер көрсеткендей, мыс мөлшері артқан сайын электр өткізгіштік, жабынның біркелкілігі және электрлік қасиеттерінің біркелкілігі (химиялық-гальваникалық никельмен қапталған талшықтар үшін) артады. Электрсіз никельмен қапталған талшықты мыс жалату кезінде мыс мөлшерінің жоғарылауына және мыс мөлшерінің өзгеру коэффициенті мен электрлік кедергінің төмендеуіне қарамастан, мыс жалату уақыты ұлғайған сайын электр кедергісінің өзгеру коэффициенті артады.

Түйін сөздер: электр өткізгіш химиялық талшықтар, электр өткізгіштік, электр кедергісі, электролит, электротұндыру.

Исследование влияния технологических параметров на процесс осаждения меди

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Аннотация. Изучена возможность нанесения медных покрытий на химически и химико-гальванически никелированные акриловые волокна. Изучено влияние pH электролита, его состава, силы тока на 1-м и 2-м катодах, а также времени металлизации на электрофизические, физико-механические свойства медьсодержащих волокон.

Получены электропроводящие волокна с различным содержанием меди. Установлены закономерности изменения электрических свойств волокон в зависимости от содержания меди.

Исследования показали, что с увеличением содержания меди увеличивается электропроводность, однородность покрытия и однородность электрофизических свойств (для химико-гальванически никелированного волокна). В случае меднения химически никелированного волокна коэффициент вариации электрического сопротивления увеличивается с увеличением времени меднения, несмотря на то, что содержание меди увеличивается, а коэффициент вариации содержания меди и электрического сопротивления уменьшается.

Ключевые слова: электропроводящие химические волокна, электропроводность, электрическое сопротивление, электролит, электроосаждение.

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