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THE NICKEL PLATING OF 3D-PRINTED PLA-PLASTIC PARTS

This work demonstrates the possibility of obtaining a finely crystalline and well-adhered conductive layer of copper sulfide, the value of the resistivity of which is comparable to the corresponding parameter for conductive layers obtained from graphite by a mechanical method.

The effect of various commercial surface-active additives to the nickel-plating electrolyte on the quality of the nickel coating obtained at the galvanic metallization stage was investigated. It has been established that the addition of an anti-pitting additive, a dispersant-wetting agent, accelerates the process of nickel electrodeposition on the copper sulfide activated dielectric surface. A significant improvement in the uniformity of the coating and obtaining a fine-crystalline structure is ensured by the use of a complex of bright-forming additives based on saccharin, which provides a high throwing power of the nickel-plating electrolyte in the range of low current densities up to 0,5 A/dm^2.

Keywords: *chemical-galvanic metallization; conductive layer; copper sulfide; nickel coating; PLA-plastic; 3D-printing. Fig.: 9. Table: 1. References: 15.*

Relevance of the research. Electrodeposition of galvanic coatings on dielectric surfaces is carried out to give the surface of plastic patrs a decorative appearance, light-reflecting properties, as well as to create electrical contact in printed circuit boards and radio-electronic devices [1; 2]. In addition, the dielectrics metallization process can be applied to obtain various nanomaterials with a highly developed surface [3; 4].

Problem statement. In the case of chemical-galvanic metallization of dielectric materials, it is necessary to create catalytic properties in order to selectively deposit the metal on the surface of the corresponding material. For this purpose, an activation process is used, which involves the use of high-cost palladium-based chemical compounds [5]. Thus, replacing the expensive palladium catalyst during activation with palladium-free conductive compounds is relevant. An alternative to this are conductive layers based on metal sulfides such as zinc, copper, and nickel.

Analysis of recent research and publications. In work [6] it was proposed to obtain conductive layers based on copper sulfides by the adsorption-diffusion method using higher polythionium acids. In [7], the synthesis of copper sulfide nanowires is proposed using a hydrothermal method using thiourea as a sulfur source. Also known [8] is the production of nanostructured sulfide from solutions in which the source of sulfur is thiosulfate. In all cases, copper-containing solutions for the synthesis of sulfides contain complex compounds of the latter, and the synthesis of sulfide materials involves the use of hard-to-reach components and additional heating of solutions to a temperature of 50…90 °C.

On the other hand, the next main stage of chemical-galvanic metallization of dielectrics is the electrodeposition of a high-quality, continuous and fine-crystalline galvanic coating on the formed conductive layer. Despite the high conductivity of the materials used to create conductive layers, due to the relatively small thickness of the latter, their conductivity can be an order

of magnitude lower than that of the corresponding bulk material. In particular, a study of the mechanism of the nickel electrodeposition process on a paraffin-impregnated graphite electrode is proposed in [9]. The electroplating conditions in this case may be similar to those implemented during electrodeposition of nickel onto a surface activated by a graphite conductive layer. In addition, in some cases [10], copper sulfides, similar to graphite, can exhibit hydrophobic properties.

Uninvestigated parts of a common problem. In [11], to create a conductive layer, a simpler method is presented, which involves the deposition of copper sulfide with the use of ammonium copper complexes and sodium sulfide solutions, which also does not require additional heating of the solutions. However, it is important to establish the sequence of operations and the duration of processing to form a homogeneous fine-crystalline conductive layer with a sufficient level of conductivity. The introduction of the latest technology for producing plastic products by 3D-printing is becoming widespread, however, the influence of appropriate processing on the adhesion and quality of conductive layers obtained on such products is poorly studied. In addition, it is important to study the influence of the electrolyte composition on the nickel electrodeposition process on the surface of the conductive layer formed from copper sulfide.

Research objective. The purpose of this work is to investigate the possibility of obtaining a highly conductive copper sulfide layer on PLA-plastic parts obtained by 3D-printing, as well as to establish the influence of surface-active additives in the nickel-plating electrolyte on the electrochemical parameters and quality of the nickel coating on a copper sulfide-activated dielectric base.

The statement of basic materials

1.1 Deposition of a copper sulfide conductive layer. The deposition of a copper sulfide conductive layer was carried out by a two-stage treatment, which consisted of the initial treatment of the plastic product in a solution of ammonium copper (II) complex, followed by rinsing and treatment in a solution of sodium sulfide [11]. For primary treatment, two types of copper ammonium complex solutions were used, which contained 0.4 M divalent copper (in the form of sulfate or basic copper carbonate) and NH_4OH (25%) 100 ml/l. The alkalinity of the corresponding solution was around pH 11. According to the data described in [11], after initial treatment in an ammonium complex solution and rinsing, the surface of plastic samples was treated in a sodium sulfide solution with a concentration of 100 g/l. The mechanism of formation of the sulfide conductive layer can be described as follows. At the first stage, the solution containing copper complex compounds is mechanically captured by the surface. As a result of further rinsing, which leads to a decrease in pH, the ammonium complex is destroyed with the formation of colloidal copper hydroxide according to the reaction [12]:

$$[Cu(NH_3)_4]SO_4 + 4H_2O = Cu(OH)_2 + (NH_4)_2SO_4 + 2NH_4OH.$$

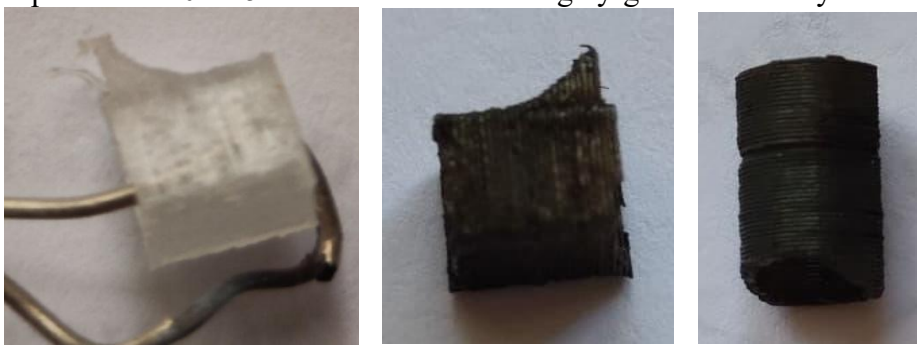
The formed copper hydroxide has a colloidal form, as a result of which it binds well to the surface of plastic samples. As a result of further treatment in a sodium sulfide solution, the following reaction occurs, which leads to the formation of a sulfide layer:

$$Cu(OH)_2 + Na_2S = CuS + 2NaOH.$$

Copper sulfides are also formed by direct interaction of residues of unhydrolyzed complex copper ions with a sodium sulfide solution.

Since the main focus of this work was to improve the technology of metallization of plastic products obtained by 3D-printing, fragments of products printed from transparent PLA-plastic were used as the test samples. On average, the area of the samples studied was 2...4 cm^2. The studied plastic samples, placed on a copper wire, were sequentially immersed in solutions of the ammonium complex of copper and sodium sulfide with intermediate washing in distilled water.

Due to the high alkalinity of the solutions, preliminary degreasing was not used. The above operations were repeated for 10…15 min until a continuous gray-green sulfide layer was formed.



a *b* *c*

Fig. 1. Initial sample made of PLA-plastic (a) and with deposited copper sulfide conductive layer (b, c)

Source: developed by the authors.

An important parameter of the conductive layer is its resistance; the lower the corresponding parameter, the more uniform is the current distribution over the surface of the sample and the faster the process of electrodeposition of the primary metal layer. The resistance of the obtained conductive layer was estimated based on the measurement of the resistance of the samples using a Baku BK-9205A digital multimeter. When measuring the resistance, the distance between the electrodes of the multimeter was 5 mm. The results of measuring the resistance of the conductive layer of copper sulfide on eight identical samples are shown in Fig. 2.

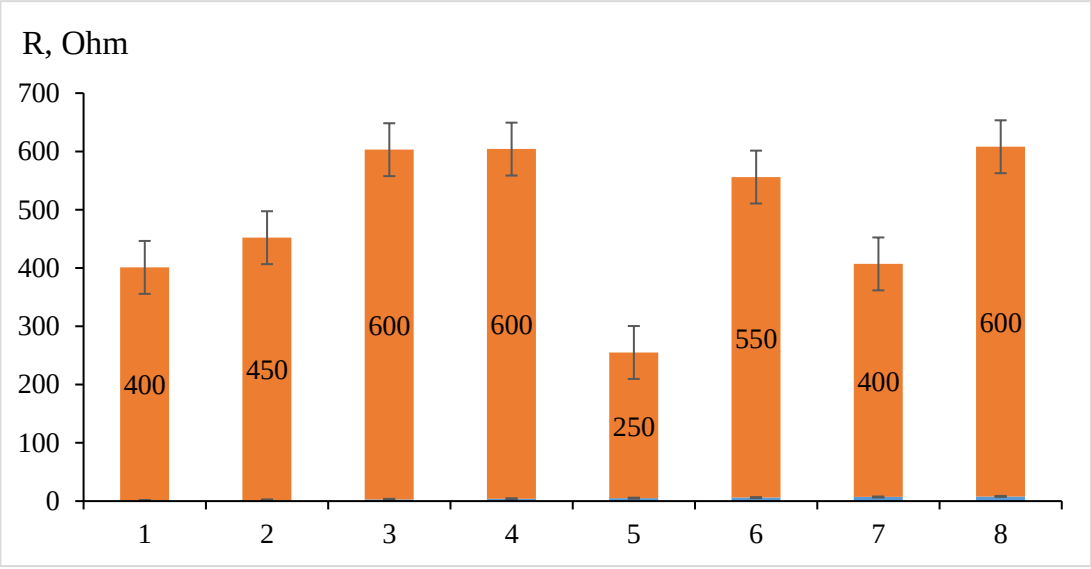


Fig. 2. Histogram of the resistances of the conductive layer of the studied samples (1-8)

Source: developed by the authors.

From Fig. 2 it can be seen that for most of the obtained samples the resistance value is on average about 500 Ohms, given the value of the interelectrode distance, it can be concluded that the approximate resistivity of the samples can be of the order of 10^3 Ohm·cm. This resistivity value is comparable to the corresponding value for graphite conductive layers obtained by mechanical rubbing of graphite [13].

1.2 The influence of electrolyte composition on the nickel electrodeposition process. In this work, industrial nickel-plating electrolytes were studied, the electrolyte of basic composition № 1 contained (g/l): $NiSO_4 \cdot 7H_2O$ – 200; $NiCl_2 \cdot 6H_2O$ – 60; H_3BO_3 – 40. Electrolyte № 2

additionally contained the wetting additive Nitech Netzmittel – 2 ml/l. Electrolyte № 3 additionally contained the wetting additive Nitech Netzmittel – 2 ml/l and a complex of brightener additives: saccharin – 5 g/l; main brightener additive TSL – 3 ml/l; additive that increases the throwing power in the range of low current densities TRSL – 3 ml/l. Electrolyte № 4 additionally contained a complex of brightener additives: saccharin – 1 g/l; butenediol – 1 ml/l [14].

To study the influence of surface-active additives on the structure and quality of electroplated nickel coatings, linear, cyclic, and chronoamperometric current-voltage measurements were performed on nickel and graphite electrodes. The corresponding measurements were performed on a digital potentiostat PGStat500n in a standard three-electrode cell. The ohmic losses were leveled by using the Luggin capillary placed close to the surface of the nickel electrode. The working electrodes were nickel and graphite rods pressed into dielectric housings, respectively. The working surface – the end face of the nickel electrode had an area of 0,28 cm^2, and the graphite electrode had an area of 0,4 cm^2, respectively. An electrode made of nickel foil of the nickel Grade 1 was used as an auxiliary electrode, which was placed along the contour of the cylindrical cell. A saturated silver-chloride electrode served as a reference electrode, the potentials on the current-voltage curves are given in the scale of the reference electrode. The measurement temperature was 18 °C. The potential scanning rate during the recording of cyclic and linear current-voltage dependences was 2 mV/s.

The result of cyclic current-voltage measurements on a nickel electrode is shown in Fig. 3.

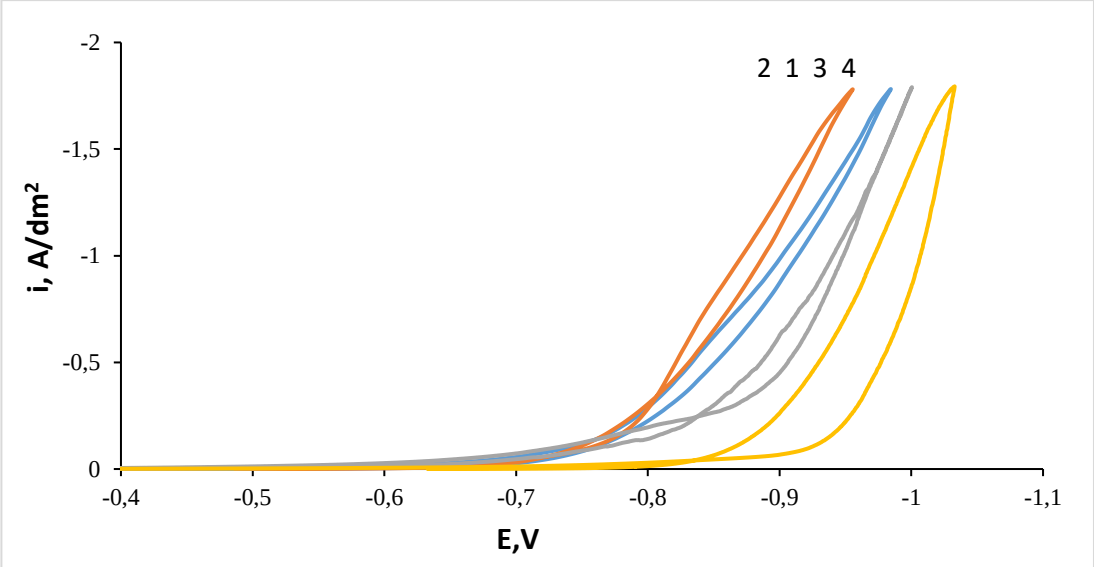


Fig. 3. Cyclic current-voltage curves on a nickel electrode in the studied nickel-plating electrolytes:
1 – electrolyte № 1; 2 – electrolyte № 2; 3 – electrolyte №3; 4 – electrolyte № 4
Source: developed by the authors.

From the polarization curves (Fig. 3.) the following conclusions can be drawn. Depending on the type of additive, the overvoltage of nickel electrodeposition changes. The lowest overvoltage is observed in electrolyte solutions № 1 and № 2. Moreover, the presence of an antipitting wetting additive (electrolyte № 2) leads to an additional reduction in cathodic overvoltage, which is greater the higher the cathodic current density. This may be a consequence of both a reduction in the overvoltage of the side cathodic hydrogen evolution process and a facilitation of the formation of a new metallic phase. In electrolyte № 3, the overvoltage increases slightly and a region of moderate increase in current density is observed in the potential range of -0.6…-0.9 V to cathodic current density values of 0.5 A/dm^2. This may indicate an earlier

onset of the nickel electrodeposition process and an increased throwing power of the corresponding electrolyte in the range of low current densities, caused by the action of the corresponding complex of bright-forming additives. For the other studied electrolytes, such a region is not observed. In electrolyte № 4 with the addition of saccharin and butenediol, the highest overvoltage of nickel deposition is observed, however, the course of the curve is similar to those obtained in solutions № 1 and № 2 without additives.

Given that the resistance of the copper sulfide conductive layer is comparable to the resistance of the graphite conductive layer, in addition, the resulting surface was quite hydrophobic. In view of this, for a simulation study in the process of nickel electrodeposition, polarization measurements were carried out on a graphite electrode, the surface of which is also poorly wetted. The cathodic polarization curves obtained on a graphite electrode in the studied nickel-plating electrolytes are shown in Fig. 4.

As can be seen from Fig. 4, the introduction of surface-active additives into the nickel-plating electrolyte affects the value of the stationary potential of the graphite electrode. However, the process of active electrodeposition or electroreduction of nickel ions in all the electrolytes studied begins at very close potential values within -0.7…-0.75 V. And at current densities of 0.5…1.5 A/dm^2 the difference between the course of the polarization curves is insignificant. Since the formation of a metallic phase on the surface of a graphite electrode can be significantly difficult, for a deeper understanding of the course of electrodeposition processes, the cathodic overvoltage values at a current density of 1 A/dm^2 were calculated and are given in Table 1.

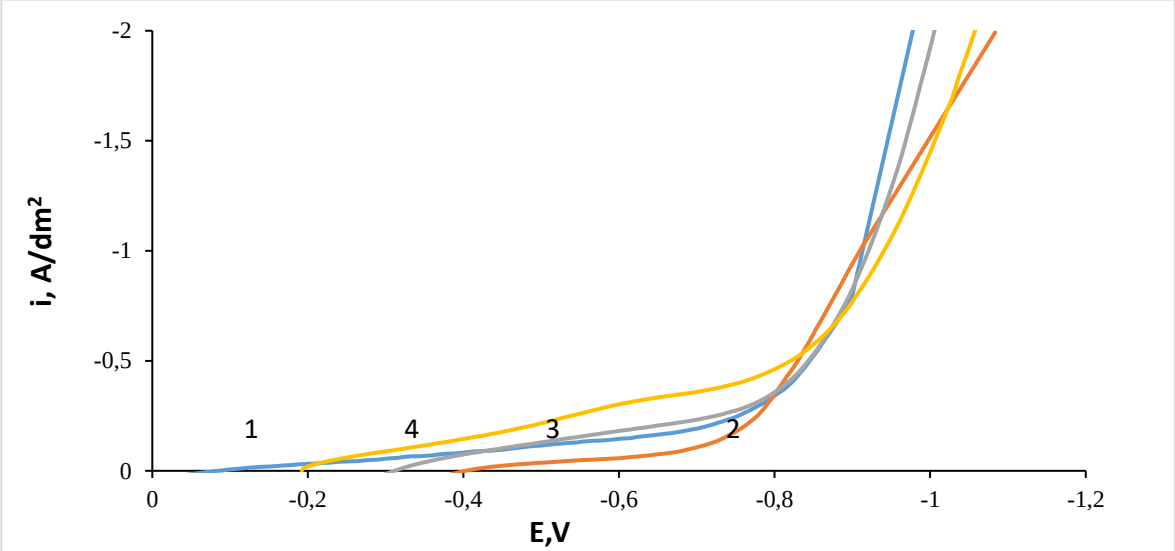


Fig. 4. Cathodic polarization curves on a graphite electrode:
1 – electrolyte № 1; 2 – electrolyte № 2; 3 – electrolyte № 3; 4 – electrolyte № 4
Source: developed by the authors.

Table 1 – Cathodic overvoltage values on a graphite electrode

№ of electrolyte	Ec, V	E (i =1 A/dm^2), V	η (i =1 A/dm^2), V
1	-0,08	-0,93	-0,85
2	-0,4	-0,92	-0,52
3	-0,3	-0,92	-0,62
4	-0,19	-0,94	-0,75

Table 1 shows that the largest cathodic overvoltage is observed for the basic nickel-plating electrolyte № 1 and the electrolyte with the introduced bright-forming complex containing butenediol (electrolyte № 4). It should also be noted that these electrolytes do not contain a dispersant-wetting additive. The corresponding additive affects the surface tension and wettability of the electrode surface and, as a result, the overvoltage of the formation of a new metallic phase

on the surface of the graphite electrode. This may be the reason for the increased values of the total overvoltage of nickel electrodeposition on the graphite electrode, in addition to the known suppressive effect of butenediol. An alternative explanation may also be the following. Simultaneously with nickel, electrochemical reduction of hydrogen occurs. Hydrogen is released primarily in more energetically favorable areas (micro-unevennesses) and blocks them as a separate phase. The wetting agent, by reducing the surface tension, promotes the desorption of hydrogen from the surface and its conditional unlocking, which also accelerates the process of electrodeposition and the formation of a new nickel phase on the free electrode surface [6]. Considering that the values of the cathode potential at a current density of 1 A/dm^2 are close to -1 V (see Table 1), chronoamperometric measurements were additionally carried out at the corresponding value of the cathode potential, the results of which are shown in Fig. 5.

From Fig. 5 it is seen that for electrolytes № 2 and № 3 a characteristic form of chronoamperometric curve with two clearly expressed maxima is observed [15]. The presence of a pronounced second maximum (electrolytes № 2 and № 3) indicates the activation of the nucleation process and their spread over the surface of the graphite electrode with the formation of a new nickel phase. In contrast, in the chronoamperometric dependences obtained in electrolyte № 4 the second maximum is absent at all, and for electrolyte №. 1 it is weakly expressed. Photographic images of the graphite electrode surfaces after chronoamperometric measurements are shown in Fig. 6.

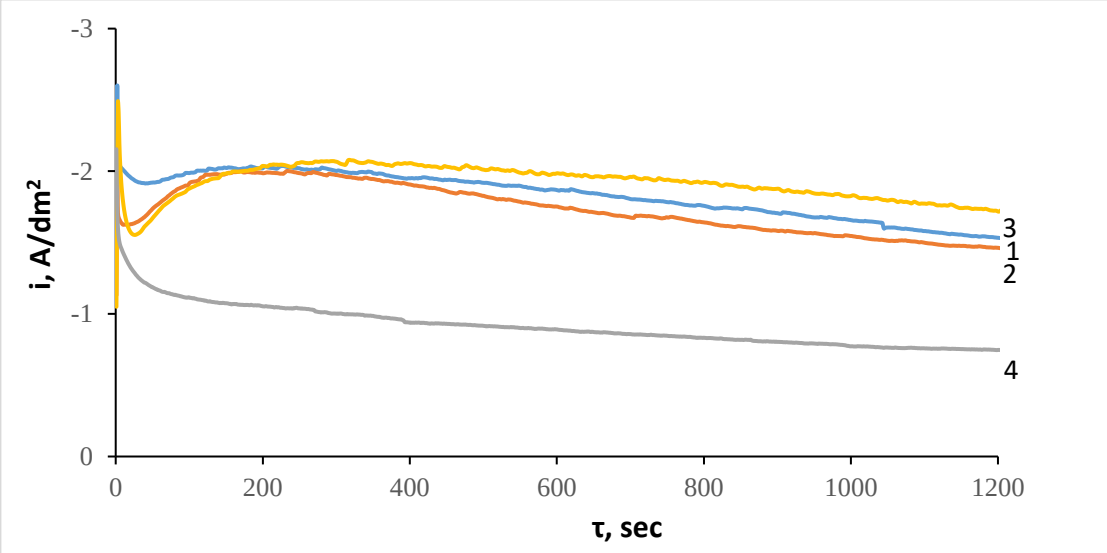


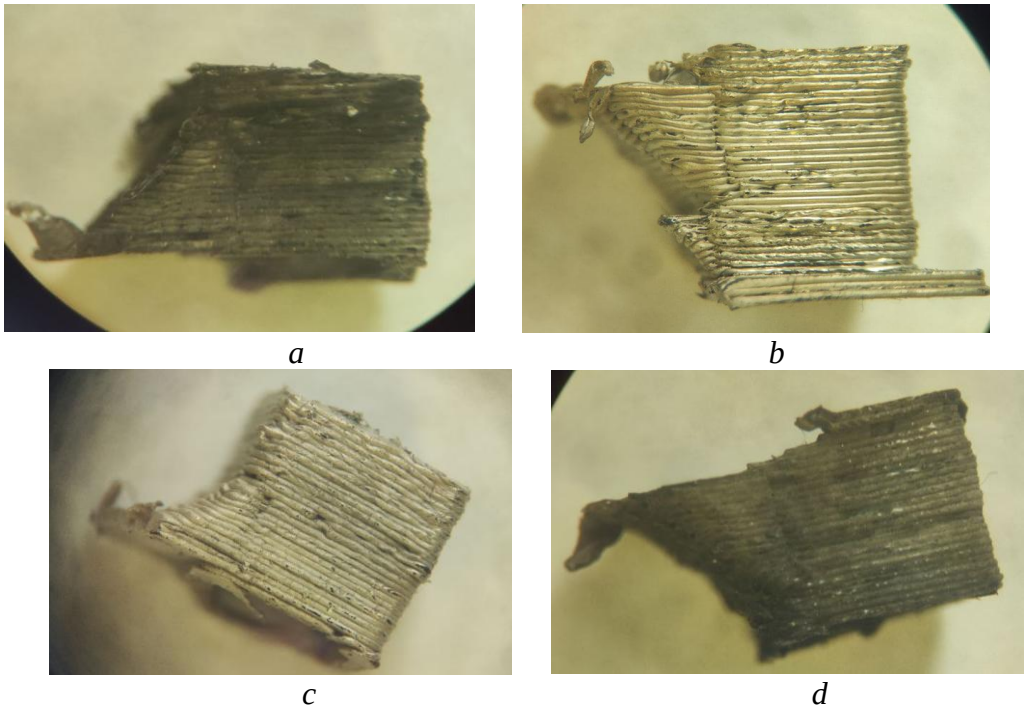
Fig. 5. Chronoamperometric dependences on a graphite electrode at a potential of E = -1 V: 1 – electrolyte №1; 2 – electrolyte № 2; 3 – electrolyte № 3; 4 – electrolyte № 4
Source: developed by the authors.

A *b* *c* *d*

Fig. 6. Photos of the graphite electrode surface after chronoamperometric measurements conducted for 1200 s (E = -1 V):
a – electrolyte № 1; b – electrolyte No. 2; c – electrolyte No. 3; d – electrolyte No. 4
Source: developed by the authors.

From Fig. 6 it is seen that in electrolyte № 1 nickel deposition occurs very slowly, the deposit is present only in the central part fragmentarily. In electrolyte № 2 matte nickel is deposited over the entire surface of the electrode. As expected, a fine-crystalline nickel coating is deposited in electrolyte № 3. At the same time, at the corresponding potential in electrolyte № 4 nickel deposition does not occur.

1.3. Electrodeposition of nickel coatings on PLA plastic samples. The next stage of the research was the testing of electrodeposition of galvanic nickel coatings on PLA-plastic samples with a copper sulfide conductive layer. The nickel coating process was carried out in a polymer electrochemical cell with a volume of 250 cm^3. A PLA-plastic sample with deposited conductive layer on a copper wire power supply was placed at the same distance between two parallel flat nickel anodes, the interelectrode distance was 35 mm. A DC source B5 – 43A was used for electrodeposition. In all experiments, electrodeposition was carried out at a voltage of 1.2 V. During experimental studies, it was found that this voltage corresponds to the optimal current density of the electrodeposition process of the primary nickel layer - about 1 A/dm^2. Electrodeposition was carried out for 20 min. The temperature of electrodeposition of coatings was 18 °C. The morphology of the obtained coatings was studied using an optical microscope MBS-9 at 36x magnification. The results of experiments on electrodeposition of nickel coatings are shown in Fig. 7.



a *b* *c* *d*

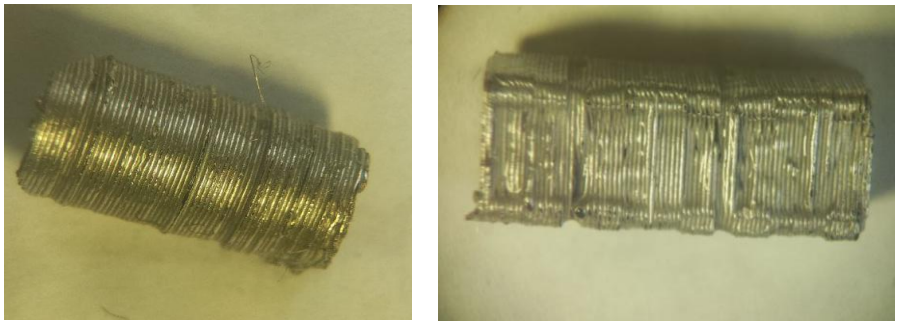
Fig. 7. PLA-plastic sample after electrodeposition of nickel coating in electrolytes: a – № 1; b – № 2; c – № 3; d – № 4. The geometric surface area of the samples is about 2 cm^2

Source: developed by the authors.

From Fig. 7, the following conclusions can be drawn. The results of nickel electrodeposition on a graphite electrode (Fig. 6) are actually reproduced on PLA plastic samples covered with a sulfide conductive layer. When nickel is plating in electrolyte № 1, nickel deposition on the surface occurred to a very small extent. When a wetting additive was introduced into the electrolyte (electrolyte № 2), a fine-crystalline nickel coating was deposited on the surface of the sample after 20 min of electrolysis. However, gaps and partial delamination of the coating were observed on the surface of the sample, which indicates the tension of the resulting nickel deposit. Also, the coating in the inner part of such samples was dull or partially absent, which

indicated a low throwing power of the corresponding electrolyte. In electrolyte №3, the highest quality fine-crystalline solid nickel coatings were obtained, delamination was not observed at all, and the number of gaps was insignificant. The percentage of coverage of the inner surface was also higher than in the previous two electrolytes. The use of industrial bright nickel-plating electrolyte №4 also did not bring a positive result, the surface of the sample after 20 min of electrolysis was also almost not covered with nickel. In this case, similar to the experiments with a graphite electrode, the voltage of 1.2 V and, accordingly, the cathode potential that was set at the same time, is not sufficient for the electrodeposition of a nickel coating.

As a final test, nickel coatings were electrodeposited on samples with a slightly larger surface area and electrolysis continued for 50 min (Fig. 8). The chronoamperometric dependence was also recorded during electrodeposition (Fig. 9).



a *b*

Fig. 8. Samples of PLA plastic after electrodeposition of a nickel coating in electrolyte № 3, electrodeposition time - 50 min, geometric surface area of the sample: a – 3.76 cm^2; b – 3.84 cm^2

Source: developed by the authors.

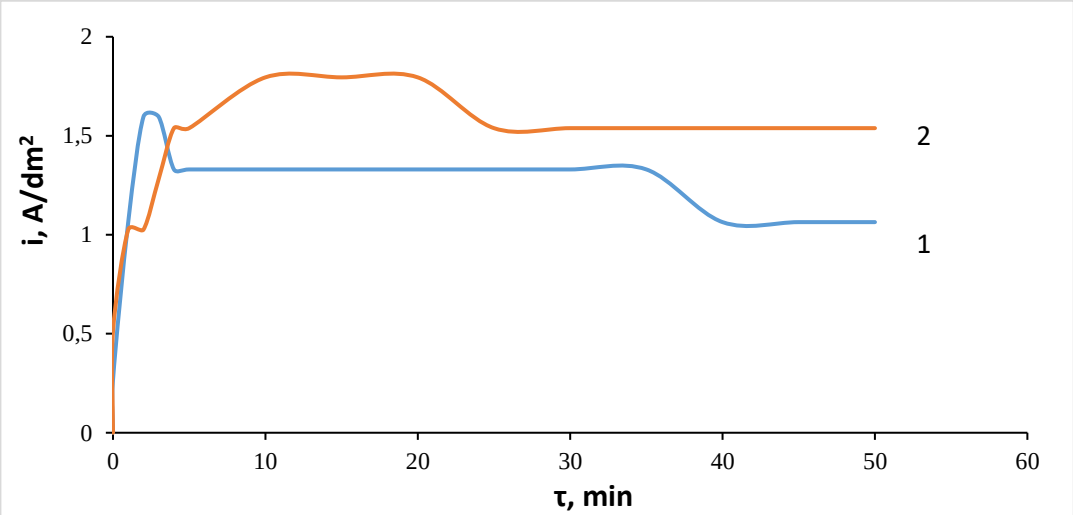


Fig. 9. Chronoamperometric dependences of electrodeposition of nickel coating in electrolyte № 3 for 50 minutes: 1 – sample a (Fig. 8); 2 – sample b (Fig. 8)

Source: developed by the authors.

In the course of the conducted research, high-quality fine-crystalline coatings were obtained (Fig. 8), the inner surface of the corresponding samples was also completely covered. Given the appearance of the chronoamperometric dependences (Fig. 9), the sharp increase in current density ends after 5 min of electrodeposition, respectively, which coincides with the completion of the formation of a solid metal layer on the outer surface of the sample with the

corresponding area. The average values of the current densities (calculated on the basis of Fig. 9) for the sample from Fig. 8 a – 1.26 A/dm^2, for the sample from Fig. 8 b – 1.6 A/dm^2, the thickness of the nickel coatings is about 12 μm and 15 μm. These results are consistent with the generally known statement that the porosity of the galvanic coating is significantly reduced when the thickness exceeds 10 μm.

Thus, the possibility of chemical-galvanic nickel metallization of PLA plastic products obtained by 3D printing, with the involvement of the stage of deposition of a conductive layer from copper sulfides, was established, which enables the direct electrodeposition of a nickel coating on the conductive layer without the involvement of the stage of chemical deposition of metal.

Conclusions. The nickel-plating process of PLA-plastic parts obtained by 3D printing was investigated. The possibility of electrodeposition of a high-quality fine-crystalline nickel coating directly onto the conductive layer of copper sulfide was established.

During the investigation of the influence of the nickel-plating solution composition on the surface of the studied plastic samples with a conductive sulfide layer, it was established that an important component that activates the process of electrodeposition of the coating on the surface of the conductive layer is the wetting additive Nitech Netzmittel at a concentration of 2 ml/l. Significant grinding of the crystalline structure and improvement of the quality of nickel coatings was achieved by additional introduction of a complex of gloss-forming additives with a saccharin content of 5 g/l.

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ХІМІКО-ГАЛЬВАНІЧНА МЕТАЛІЗАЦІЯ НІКЕЛЕМ ВИРОБІВ З PLA-ПЛАСТИКУ ОТРИМАНИХ СПОСОБОМ 3D-ДРУКУ

Показана можливість отримання дрібнокристалічного та високопровідного струмопровідного шару із сульфіду міді на поверхні виробів з PLA-пластика, отриманих способом 3D-друку. Величина питомого опору отриманого провідного шару є сумірною із відповідним параметром для струмопровідних шарів, отриманих з графітового порошку механічним способом.

З огляду на встановлені подібні властивості поверхні струмопровідного шару із сульфіду міді та графітового, модельні дослідження механізму процесу електроосадження нікелю проведені на графітовому електроді. Зокрема, досліджено вплив різних поверхнево-активних добавок, введених у електроліт нікелювання на якість нікелевого покриття, отриманого на етапі гальванічної металізації. У процесі електроосадження нікелевих покриттів на поверхні виробів з PLA-пластика з мідно-сульфідним струмопровідним шаром була досягнута відтворюваність результатів отриманих на графітовому електроді. Встановлено, що додавання антипітингової добавки диспергатора-змочувача пришвидшує процес електроосадження нікелю на активованій сульфідом міді поверхні діелектрика. Введення відповідної добавки призводить до зменшення поверхневого натягу. Це з одного боку призводить до полегшення змочування електролітом гідрофобної електродної поверхні та зменшення перенапругу утворення нової металевої фази. З іншого боку, паралельно із електроосадженням нікелю виділяється водень, який блокує активні ділянки електродної поверхні і утруднює процес утворення нової нікелевої фази на них. Наявність відповідної добавки сприяє інтенсифікації десорбції водню і, як наслідок, утворенню нової нікелевої фази. Суттєве поліпшення рівномірності та отримання дрібнокристалічної структури покриття забезпечується використанням комплексу блискоутворюючих добавок на основі сахарину, що зумовлює підвищення розсіювальної здатності електроліту нікелювання в області малих значень густин струму до 0,5 А/дм^2.

Ключові слова: *хіміко-гальванічна металізація; струмопровідний шар; сульфід міді; нікелеве покриття; PLA-пластик; 3D-друк.*

Рис.: 9. Табл.: 1. Бібл.: 15.

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