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APPLICATION OF HOLLOW CATHODE GLOW DISCHARGE IN THE PROCESSES OF THIN METAL FILMS DEPOSITION

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INTRODUCTION

Thin metal films are widely used in various fields of modern industry. However, the most widespread use thin-film elements have got in the different electronics technological processes, for instance, in manufacturing of semiconductor devices, in laser and nonlinear optics to provide polarizing, illuminating or mirror properties to optical elements etc.

Currently there are two main groups of methods for deposition of thin metal film coatings: chemical vapor deposition (CVD) and physical vapor deposition (PVD) which differentiate from each other with the process of obtaining a film-forming flux of atoms, ions or molecules. Nowadays, much attention is paid to physical vapor deposition (PVD) methods in which the atoms and metal molecules required for the synthesis of coatings are obtained by means of processes involving the evaporation of a target[1]. The main representatives of this group of methods are the sputtering by the cathode`s spots of vacuum arc discharge, electron and ion beams, thermal vacuum evaporation, magnetron sputtering and so on.

The experience of industrial application of such technologies, made it possible to identify along with the advantages their main cons, mainly due to the low deposition rate, poor coating uniformity, poor adhesion to the substrate surface, limited processing surfaces, and the like.

Recently, a gas-discharge plasma of abnormal glow discharge with a cold cathode in crossed electric and magnetic fields at pressures below 1 Pa for generating of necessary fluxes of atoms and molecules in order to obtain metal film layers is been used. The

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[1] Plasma assisted physical vapor deposition processes: A review / R.F. Bunshah, C.V. Deshpandey // J. Vac. Sci. and Technol., A3 (3), 1985, p. 553-560.

results of studies performed in [2] and [3] showed that the usage of low-pressure gas-discharge plasma initiated in a magnetic field allows to obtain of metal coatings in thickness close to homogeneous on substrates with a large surface area. The high sputtering rate, the absence of overheating of the substrate`s surface, the relatively low degree of contamination of the films makes this method a very effective source of directional atoms and metal ions suitable for deposition of coatings.

At the same time, there is no any data regarding the usage as a source of spray particles for the cultivation of metallic films on dielectric substrates of low-temperature ionized plasma of hollow cathode glow discharge without the applied peripheral magnetic field at the traditional pressures of 1 to 100 Pa. The simplicity of the design, the long life expactancy of the target (cathode) between replacements, the low cost of the equipment compared to the magnetron systems have made it quite economically effective and relevant to investigate the usage of hollow cathode glow discharge for thin metal films deposition.

1. Experimental procedure

As a source of ionized plasma of glow discharge (further GD), burning in the hollow cathode is used. A diode plasmoionic discharge scheme in which the voltage was applied between the hollow cathode (material of the future film) and the anode was implemented (Fig. 1).

The processes of formation and growth of thin-film metal coatings using gas-discharge technologies are determined by the coherence of a number of factors: the size and relative spatial position of the target and the substrate surface, the pressure in the vacuum chamber, the current and voltage in the discharge gap, the deposition time, etc [4], [5].

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[2] Болбуков, В.П. Распыление мишени на дне полого катода источника быстрых молекул газа в неоднородном магнитном поле / В.П. Болбуков // Вестник МГТУ «СТАНКИН». 2014. № 2. С. 111–117.

[3] Метель, А.С. Тлеющий разряд с электростатическим удержанием электронов: физика, техника, применения / А.С. Метель, С.Н. Григорьев – М.: ИЦ МГТУ «Станкин», «Янус-К». - 2005. – 296 С.

[4] G.P. Bolotov, M.G. Bolotov, I.O. Prybytko, G.K. Kharchenko, “Diagnosis of plasma glow discharge energy parameters in the processes of treatment small diameter long tubes”, in II International Young Scientists Forum on Applied Physics and Engineering (YSF), Kharkiv., IEEE. 2016, pp.116 – 119. **DOI:** 10.1109/YSF.2016.7753815

[5] CVD-processes by hollow cathode glow discharge / A. Hellmich, T. Jung, A. Kielhorn, M Ribland // Surf. Coat. Technol., 1998, V.98, p. 1541-1546.

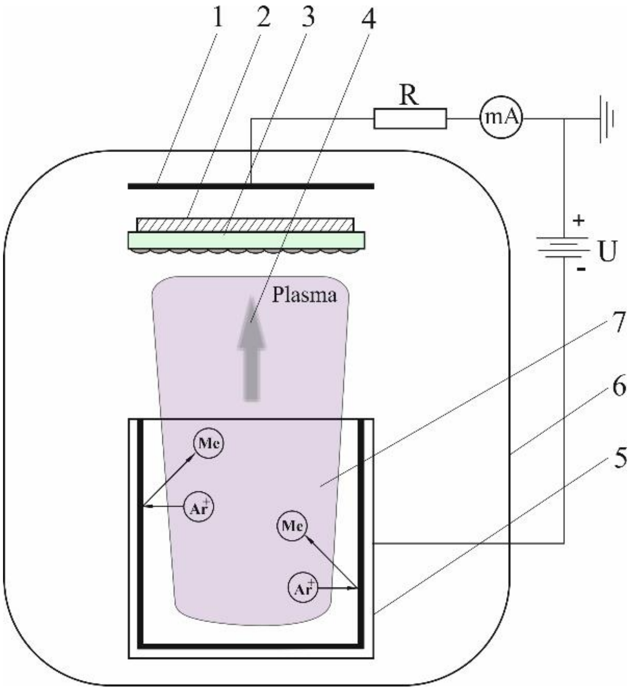

Fig. 1. Experimental scheme: 1 - anode; 2 - support; 3 - glass substrate; 4 - the direction of the evaporated metal; 5 - hollow cathode; 6 - vacuum chamber; 7 - glow discharge plasma.

In this regard, the purpose of our research was determination the influence of these technological parameters on the thickness of the coating layer, the deposition rate and the adhesion value of films with a glass base during deposition in low-temperature plasma of a hollow cathode GD.

The influence of the dimensions of the cathode cavity`s original aperture on the thickness of film layer was determined using cathodes with a diameter of 40 mm and 20 mm with a surface area of 5024 mm^2 and 2512 mm^2, respectively. The height of target (cathode) in both cases was 40 mm. The diameter of the anode ring remained unchanged during the studies and was 50 mm. Thus, the surface anode`s area was 300 mm^2.

The influence of the technological mode parameters of deposition was determined by alternating them within the following limits: the distance of the cathode substrate $L_{c\text{-}s}$ = 5… 30 mm, the discharge current I_d = 25… 100 mA, with the voltage on the electrodes fluctuating within 400… 800 V. The deposition was carried out in argon

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environment at the gas pressures of 26… 60 Pa. The deposition time varied from 15 to 60 min. Before deposition, the training of the emitting surface of the cathode was carried out at a current of 15 mA in argon medium for 2 min. in order to clean it up from various contaminants and creating of free release of the target atoms. The substrate was also pre-treated by the chemical etching in hydrofluoric acid for 1 min. and then washed in distilled water.

The adhesive strength of the copper films with the glass substrate was determined by the method of indentation in accordance with the methodology described in [6]. Fig. 2 show the scheme of installation for determining the adhesion value of thin films with the substrate surface.

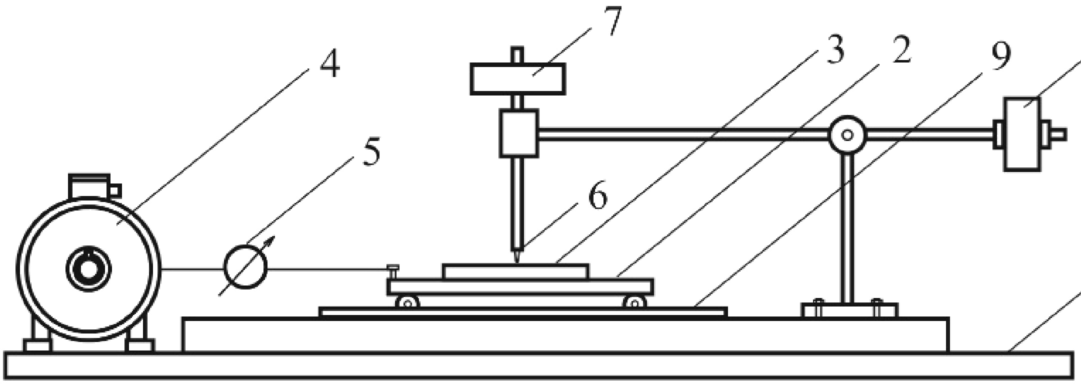

Fig. 2. Scheme of device for determination of coatings adhesive properties: 1 - table; 2 - movable platform; 3 - sample; 4 - electric motor; 5 - dynamometer; 6 - indenter; 7 - vertical load; 8 - counterweight; 9 - directing rails.

The basis of this technique is the assumption that the adhesion of the coating to the substrate is provided by the layers directly adjacent to it. The adhesive strength of the copper film with the glass substrate can be calculated by the following equation:

$$F_{adh} = F_1 - F_2 - F_3, \qquad (1)$$

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[6] Физика тонких металлических и полупроводниковых слоев / И.Д. Конозенко // Успехи физических наук, т. LII. Вып. 4, 1954, с. 561–602.

where F_1 - the force required to move the indenter through the coating under a vertical load P_1, when a coating remains on the trace, the amount of which does not exceed 5% of the total trace area of the indenter; F_2 - the force required to move the indenter through the coating under vertical load P_2 when, on the indenter track, the clear glass is not less than 5% of the total area of the trace; F_3 - force arising from the movement of the indenter head on glass under vertical load P_1 - P_2.

The adhesive strength is determined by the equation:

$$P_{adh} = \frac{F_{adh}}{S}, \tag{2}$$

where S - area released by the indenter on the glass during its passage in 1 sec was determined by follow equation:

$$S = d\frac{L}{t}, \tag{3}$$

where d - the track width from the indenter needle; L - the total length of the track; t – move time.

2. Deposition of thing metal films in low temperature plasma of hollow cathode glow discharge

Fig. 3 shows the dependence of the thickness of the copper film obtained by spraying cathodes with a diameter of 20 mm and 40 mm on a glass substrate. The deposition was carried out at fixed values of the discharge current I_d = 100 mA in argon medium at a pressure in the discharge chamber 26 Pa for 30 minutes. The cathode - substrate distance has been varied within the limits of 10… 30 mm. The voltage at the discharge gap was 600… 800 V, respectively.

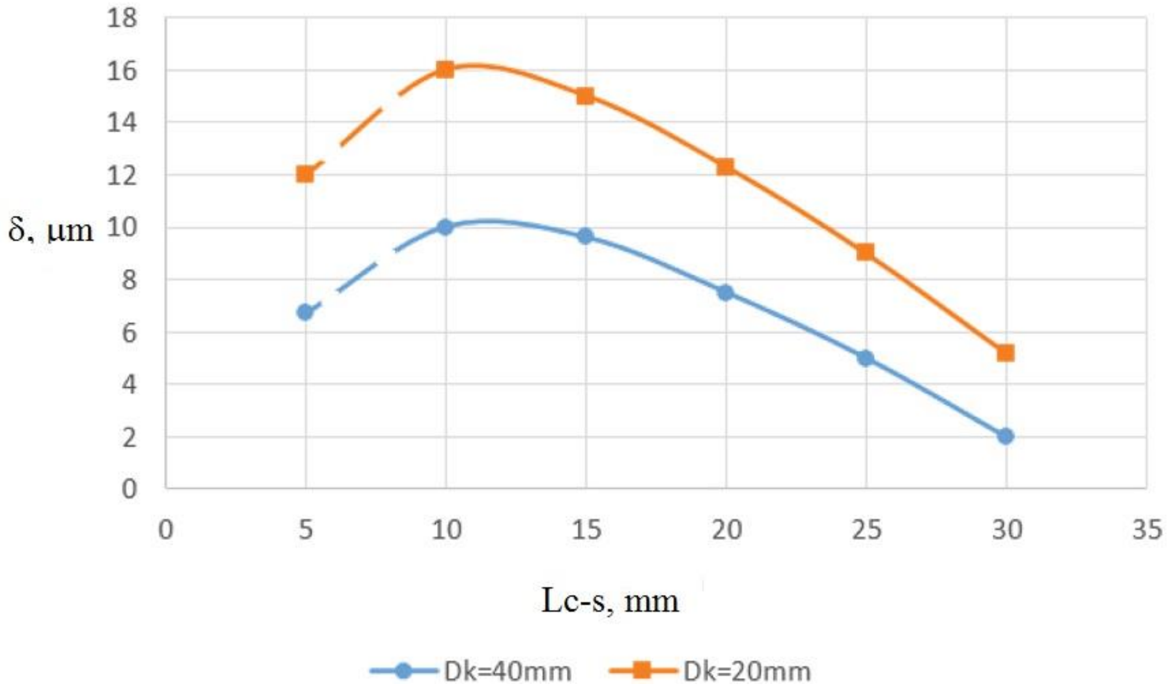

Fig. 3. Dependence of the thickness of the copper film layer δ on the cathode-substrate distance L_{c-s} deposited on glass substrates.

The experimentally obtained dependencies show that the thickness of the condensed layer increases as it approaches the outlet of the cathode cavity. Thus, reducing of L_{c-s} from 30 mm to 10 mm in our experiments inevitable led to increasing of the thickness of the layer of copper film by 3..5 times with a marked deterioration of its homogeneity. The unevenness of the thickness of the films obtained at a distance of 30 mm doesn't exceed 5… 8%, while for films obtained at a distance of 10 mm from the cathode such uniformity is maintained only at a distance of 10… 15 mm from the center of the substrate and at a distance of 20 mm reaches 30%.

Fig. 4 shows the graphs of the radial distribution of the thickness of the metal coating layer on a glass substrate with a size of 60 × 30 mm. Point 0 on the graph is the center of origin of the film. Reducing of the cathode-substrate distance, obviously, leads to a certain increase in the temperature of the substrate, which is primarily due to the increasing of bombardment intensity of its surface by heavy atoms. In turn, substrate heating, causes an increase in the time of diffusive displacement of particles and migration processes on its surface, thereby contributing to the growth of the film layer thickness. However, in our experiments, further reduction of the $L_{c\text{-}s}$ distance to 5 mm entailed the significant slowdown in the growth of the coating layer. The thickness of such films is about 3… 4 μm less than the thickness of the films obtained at $L_{c\text{-}s}$ = 10 mm. This effect, in our opinion, can be associated with two main processes: mechanical separation of deposited metal atoms on the substrate`s surface under bombardment by rapid gas ions, and their evaporation due to local heating by argon`s ion flux (thermal atomization).

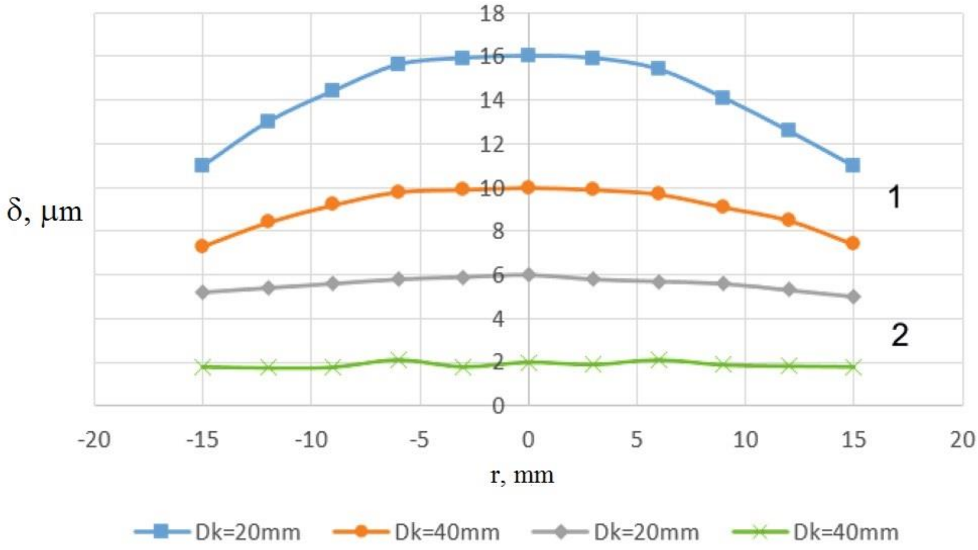

Fig. 4. Radial distribution of copper films thickness on a glass substrate deposited at a distance of: 1) 10 mm from the cathode cavity; 2) 30 mm from the cathode cavity.

Experimentally obtained dependences (fig. 5) show that increasing of the discharge current leads to a rapid raising in the thickness of the metal coating condensed layer. Thus, going up of the I_d value from 25 mA to 100 mA, in our experiments, led to an increase in the thickness of the copper film within 0.5 ... 10 μm and 1 ... 16 μm for cathodes with a diameter of 40 mm and 20 mm, respectively.

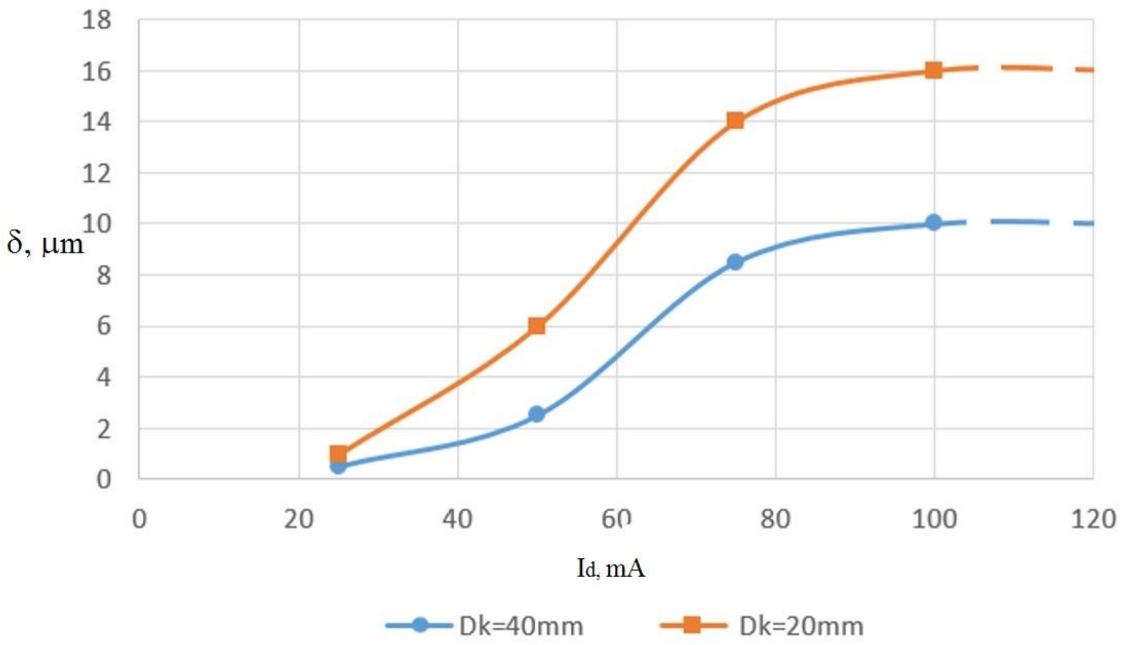

Fig. 5. Dependences of the copper films thickness δ on the magnitude of the discharge current I_d obtained at an argon pressure of 26 PA and a deposition time of 30 min.

The greater thickness of the copper film obtained by sputtering of the cathode with a diameter of 20 mm is due to a more intense ionization and, accordingly, emission processes in the middle of the cavity than to a cathode with a diameter of 40 mm. This is due to the fact that at constant pressure in the discharge chamber, reducing the diameter of the cavity leads to a certain increasing in current density because of decreasing in the length of the dark cathode space d_k [7], [8]. Accordingly, the flow of ions increases, bombarding the surface of the target, thereby intensifying the process of sputtering of its material. In the studied range of deposition mode parameters variation, the discharge current density varied within 5… 20 A/m^2 for a cathode with a diameter of 40 mm and 10… 40 A/m^2 for a cathode with an aperture of 20 mm.

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[7] Bolotov, M.G., Bolotov, G.P. “Criterial definition of the limits of glow discharge energy stability in welding”. 2019 IEEE 2nd Ukraine Conference on Electrical and Computer Engineering, UKRCON 2019 - July 2019, P. 497. **DOI:** 10.1109/UKRCON.2019.8879992

[8] G.P. Bolotov, M.G. Bolotov I.V. Nahorna. “Hollow cathode glow discharge as a heating source in welding and brazing”. 2017 IEEE 1st Ukraine Conference on Electrical and Computer Engineearing, UKRCON 2017. pp.1197-1202. **DOI:** 10.1109/UKRCON.2017.8100441

Fig. 6 shows the dependence of the copper films thickness on the pressure of argon in the discharge chamber deposited at a current I_d = 100 mA for 30 minutes. The pressure in the discharge chamber varied within 26… 66 Pa, with the discharge voltage U_d = 600… 900 V. The distance of the cathode-substrate was 10 mm.

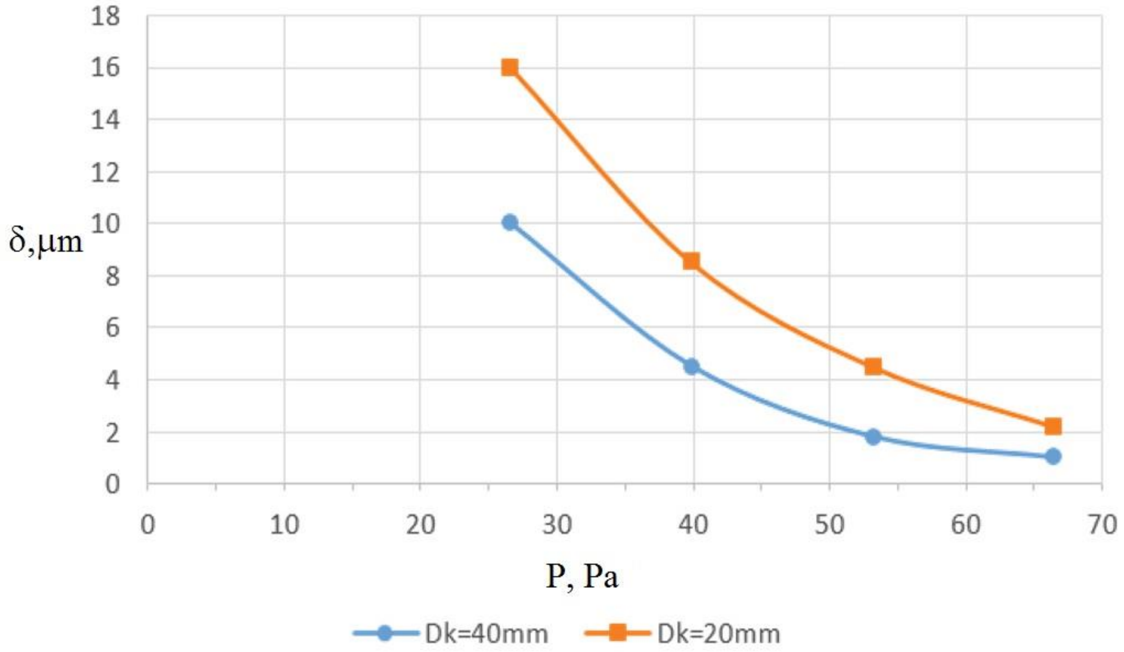

Fig. 6. Dependence of coating thickness δ on pressure in discharge chamber P at discharge current I_d = 100 mA, distance $L_{c\text{-}s}$ = 10 mm and deposition time 30 min.

Changing of argon pressure in discharge chamber from 26 to 66 Pa leads to a rapid decrease in both the thickness of the film itself and the rate of its deposition on the glass substrate for both studied cathodes by 8 ... 10 times. This gives grounds to consider this parameter as the most important in the processes of thin film coating extensions. In this case, the decisive length of the free path of both the atomized target atoms and the working gas ions, whose value decreases with increasing pressure, is crucial. Since, in a result of increasing the collisions number with the residual gas molecules, they lose their energy and change the trajectory of their motion, which leads to a decrease in the coefficient of atomization of the material and to a decrease in the flux density of the deposited particles on the substrate surface accordingly. However, the decrease in the gas gap pressure leads to increasing in the length of the dark cathode space that separates from the walls of the cavity compresses the cathode part of the plasma so that it begins to resemble an electron beam. This causes the decreasing of the condensation area on the substrate surface. The uniformity of such films in thickness does not exceed 15… 25% for cathodes 40 and 20 mm, respectively.

It should be noted that the deposition rate and the thickness of the coating layer also depend on the intensity of the bombardment and the target's material atomization by the positive ions of the working gas. The voltage at the electrode gap determines their energy, or rather by the magnitude of the cathode drop in U_c potentials [9]. This is due to the fact that almost all the discharge voltage is concentrated in its cathode part, since the anode potential drop and the discharge column in the sum do not exceed 15… 20%.

In case of a GD with a hollow cathode, the determination of the magnitude of the cathode drop in U_c`s potentials is due to some difficulties, which are due, first of all, to the cathode geometry. As in the hollow cathode conditions it doesn`t possible to ensure the equidistant of all points of its surface from the anode. In this regard, the current density and the cathode potential drop across the cavity surface are not constant [10].

In [11], a technique that allows considering the ionization processes in the cathode layer of a glow discharge with a hollow cathode to determine the value of U_c with satisfactory agreement with the experiment is proposed.

The proposed expression combines the discharge voltage with the pressure in the discharge gap and looks as follows:

$$\frac{U_c}{U_0} = -\frac{p}{p_0}\ln\left(1-\frac{p}{p_0}\right), \qquad (4)$$

where U_c is the cathode potential drop; U_0 - discharge burning voltage at pressure P; P - pressure in the chamber; P_0 is the critical pressure at which discharge burning becomes impossible.

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[9] Москалев Б.И. Разряд в полом катоде. М: Энергия. 1969, 181 с.

[10] G.P. Bolotov, M.G. Bolotov, "Determination of external stabilizing resistor value in the glow discharge power supply while welding", IEEE 37th International Conference "Electronics and Nanotechnology ELNANO'2017", pp.365-369, April 2017. **DOI:** 10.1109/ELNANO.2017.7939780

[11] Никулин С.П. "Тлеющий разряд с полым катодом в длинных трубках" ЖТФ. 1999. № 6 (69) с. 36-39.

$$p_0 = \frac{\kappa T S_a}{4\gamma V \sigma_i}, \tag{5}$$

where k - the Boltzmann constant; T - gas temperature; S_a - the area of the anode; γ - the ion-electron emission factor; V - the volume of the cavity; σ_i - the ionization cross section.

The discharge`s burning voltage U_0 is determined by the equation:

$$U_0 = \frac{W}{e\gamma} + \frac{E_i}{e} \approx \frac{W}{e\gamma}, \tag{6}$$

where W - the average energy lost per ionization; E_i - the ionization threshold energy.

The calculations performed by this methodology has subsequently allowed us to determine the energy of the fast argon atoms with which they fall to the surface of the cathode and causing its atomization. The deposition rate was defined as the ratio of the thickness of the film to the duration of its production.

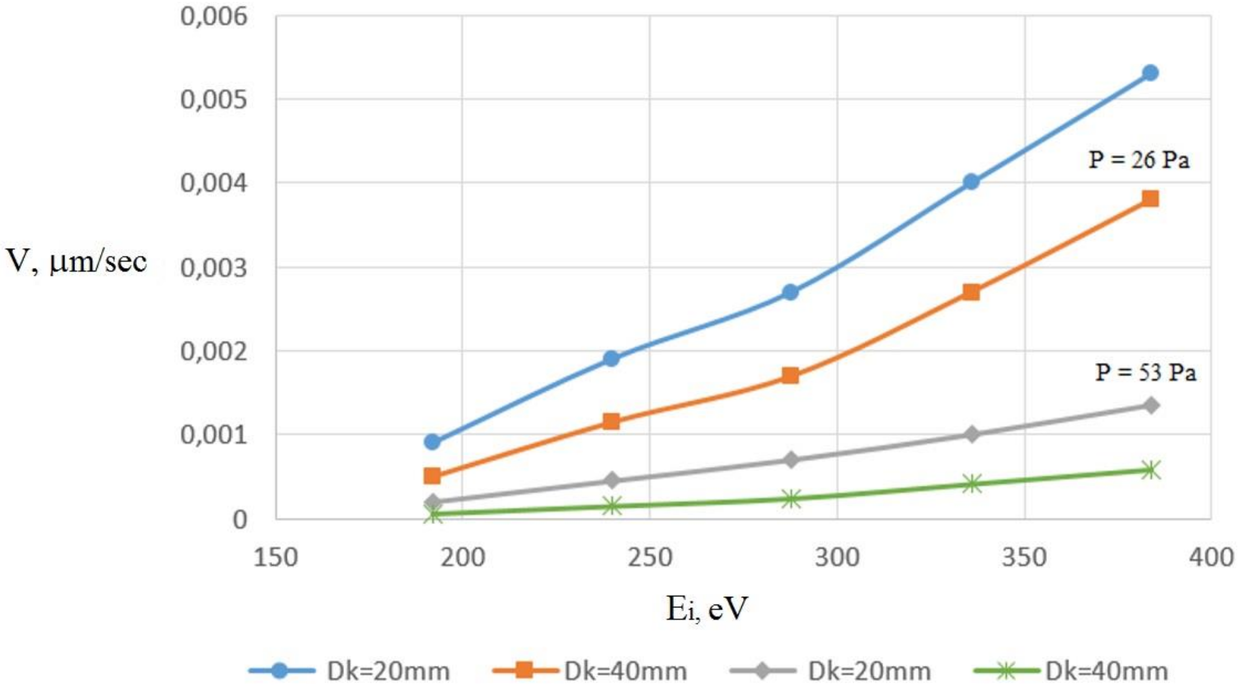

Fig. 7. Dependences of the deposition rate of copper films on a glass substrate on the ions energy which bombarding the cathode surface at argon pressures in a gas chamber of 26 Pa and 53 Pa.

Fig. 7 shows the dependences of the rate of the copper films growth on the energy of argon ions obtained at pressures in a discharge chamber of 26 Pa and 53 Pa for 30 min for cathodes with a diameter of 20 mm and 40 mm. The discharge voltage varied from 400 to 800 V. Dependences show that with increasing of eU value from 192 eV to 384 eV, the deposition rate of metal coating increases almost linearly for cathodes of both diameters. The different slope of the curves also indicates a significant effect of argon pressure in the gas chamber on the film deposition rate.

Fig. 8 shows the microstructure of Cu films deposited on a glass substrate at the energy of atoms of 192 eV for 30 min with the gas pressure of 53 Pa and 26 Pa.

The microstructure of a Cu film obtained at a gas pressure of 53 Pa characterizes by the less homogenious structure, rather than film which we`ve got at a pressure of 26 Pa. So then, at a pressure of 53 Pa the fast argon atoms form structural defects “islands” on the surface of the substrate, which are the condensation centers of the future film.

Also, was established that, increasing of the pressure in the discharge chamber from 26 Pa to 53 Pa leads to a significant slowdown of the film condensation process, which is caused by the partial loss of kinetic energy of both the atomized atoms of the target material and the fast argon ions due to their collisions.

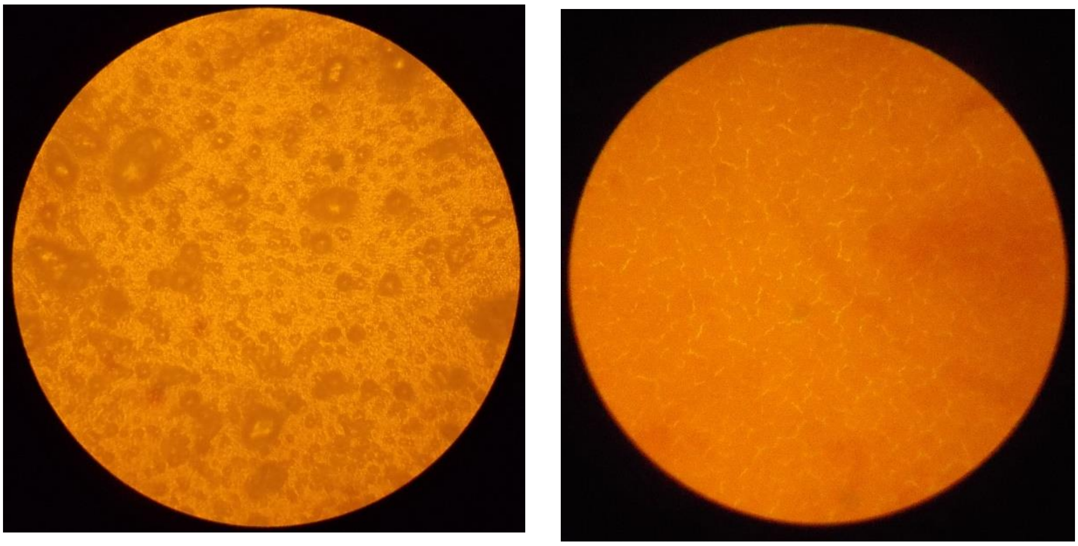

Fig. 8. Microstructure of Cu films deposited on a glass substrate at a energy of atoms of 192 eV for 30 min with the gas pressure of 53 Pa (a) and 26 Pa (b).

Fig. 9 shows the dependence of the adhesion of copper films on their thickness obtained at a deposition rate of 0.0053 μm/sec and a current density of 15 A/m^2 and 29 A/m^2 for cathodes with a diameter of 40 mm and 20 mm, respectively. From fig. 8 follows that in the range of thicknesses of 0.5… 1.2 μm obtained during deposition from a cathode with a diameter of 40 mm, the adhesive strength changes within the range of 38… 32 MPa. With further increase in thickness of the deposited films, their adhesion with the glass surface of the substrate deteriorates dramatically. Thus, in the thickness range of 1.5… 9 μm, for both cathode diameters, the adhesion strength decreases from ~ 17 MPa to 1 MPa, and at δ = 12… 16 μm the adhesion strength drops to almost zero and is approximately 0.41… 0. 24 MPa. Such dependence, in our opinion, is explained by the increase of internal stresses in the body of the coating with rising of film thickness.

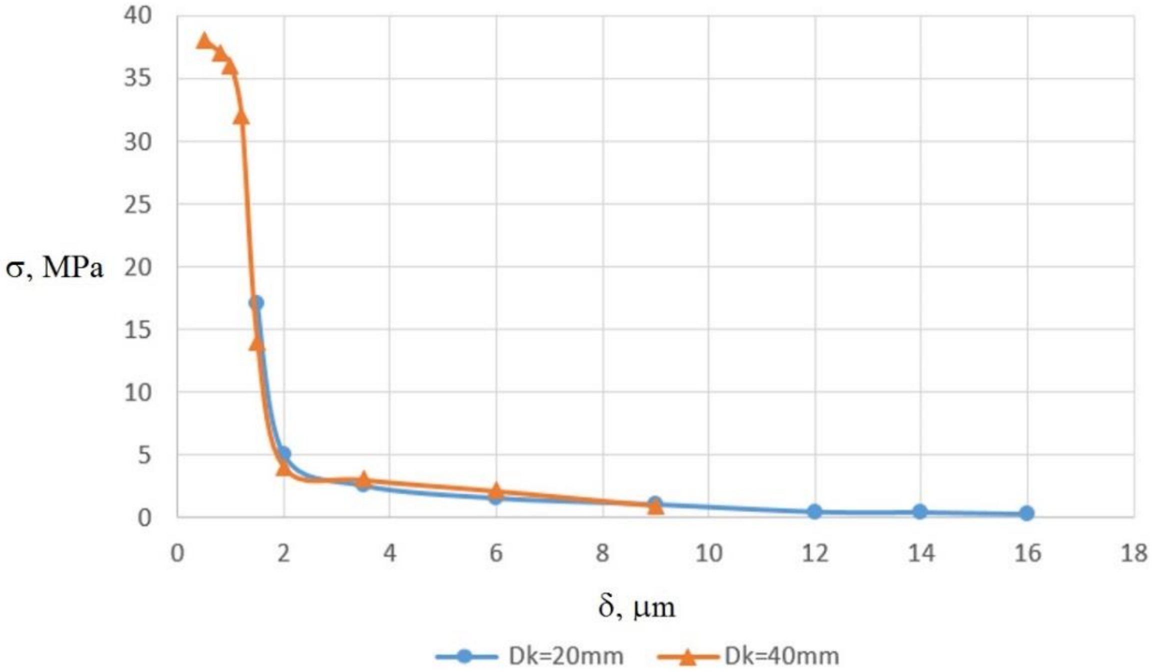

Fig. 9. Dependence of adhesion of copper coatings on their thickness.

3. Optimization of thin metal films deposition process in hollow cathode glow discharge

As mentioned earlier, the formation and growth of metal films in a plasma of glow discharge is multifactorial, ie, defined coherence of a number of parameters, which greatly complicates the control of deposition process. In this regard, the ways of optimizing the conditions of technological process of deposition of thin-film in glow discharge with the hollow cathode effect were considered.

The definition of these parameters in the traditional way in this case is complicated, so their choice was performed by the "swing" method, when the change in the desired parameter was fixed while deviating one of the technological parameter from some average value by 25% in the direction of increasing and decreasing of it [12].

The model was introduced into the set of parameters, taking into account the possibility of determining their quantitative assessment. As the parameter of optimization the thickness of the copper film (δ, μm) deposited on a glass substrate was determined. As the variable factors the cathode-substrate distance $L_{c\text{-}s}$ (X1), discharge current I_d (X2), the magnitude of the pressure in the discharge chamber P (X3) and the deposition time t (X4) were taken.

To minimize the effects of random measurement errors, each experiment attempt was repeated 5 times. The intervals of variation of factors and their numerical values at the upper and lower levels are shown in the table. 1.

Table 1 - Levels and intervals of factors variation

Factors	Variation intervals, ΔX	The levels of variation		
		The lower level, (X_1=–1)	Basic level	The upper level, (X_1=+1)
The cathode-substrate distance ($L_{c\text{-}s}$), mm	10	10	20	30
Discharge current I_d, mA	25	50	75	100
Pressure in the discharge chamber P, Pa	20	20	40	60
Deposition time t, sec	20	20	40	60
The optimization parameter is the thickness of the copper film δ, μm				

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[12] Новик Ф.С. Математические методы планирования экспериментов в металловедении / Ф.С. Новик // – М.: МИСИС, 1972. – 105 с.

In order to determine the influence of the varying factors on the value of the optimization parameter, a full factor experiment, the matrix of which has the form 2^4 was used.

The linear model of this experiment looks like this:

$$y_1 = b_0 + b_1 \cdot x_1 + b_2 \cdot x_2 + b_3 \cdot x_3 + b_4 \cdot x_4; \tag{7}$$

The calculation of the coefficients for the corresponding factors was carried out according to the next equation [13]:

$$b_j = \frac{\sum_{i=1}^{N} x_{ji} y_i}{N}, \; j = 0, 1, \ldots . k \tag{8}$$

where $\sum_{i=1}^{N} x_{ji}$ - the algebraic sum of the elements vector - the column of each factor; *i* - the experiment number; *j* - factor number; *N* - the number of experiments.

The coefficient b_0 was found as the arithmetic mean of the optimization parameter, $b_0 = 5{,}987$, with $b_1 = -1{,}125$; $b_2 = 2.913$; $b_3 = -1.375$; $b_4 = 2.475$.

It is known that the magnitude of the coefficient at the corresponding factor determines its contribution to the value of the optimization parameter, ie, the greater the numerical value of the coefficient, the stronger the factor will affect the optimization parameter. In this regard, it can be concluded that the discharge current and deposition time are the most positively influential parameters, as the optimization parameter (thickness of the deposited layer) increases with their numerical value. Most negatively, among the studied parameters, the process of deposition of the coating in the glow discharge with a hollow cathode is influenced by the increase in the distance of the cathode substrate and the pressure in the discharge chamber, which is evidenced by the minus sign at the corresponding coefficients.

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[13] Адлер Ю.П. Планирование эксперимента при поиске оптимальных условий / Ю.П. Адлер, Е.В. Маркова, Ю.В. Грановский.; Под ред. Ю.П. Адлер. – М.: Наука, 1976. – 278с.

The calculation of the optimization parameters y1 performed by equation 1 showed a significant discrepancy with respect to the experimental data (Table 2).

Table 2 - The results of the experiments

№ Exp.	Level of factors				Experimental value of optimization parameter y, µm	Estimated value of the optimization parameter y_1, µm	The value of the optimization parameter based on pair interactions y_2, µm	The value of the optimization parameter based on triple interactions y_3, µm
	X_1, mm	X_2, mA	X_3, Pa	X_4, sec				
1	10	50	20	60	6	8,05	6,3	5,9
2	30	50	20	20	2,5	0,85	2,5	2,5
3	10	100	20	20	9	8,9	8,74	8,9
4	30	100	60	60	10	9	10,54	10,13
5	10	50	20	20	4	3,1	3,712	4,1
6	30	50	60	20	1	-1,9	0,99	1,12
7	10	100	60	20	3,5	6,2	3,8	3,6
8	10	100	20	60	16	13,9	16,3	16,13
9	30	100	20	20	4,5	6,7	5,04	4,6
10	10	50	60	60	5	5,3	4,7	5,1
11	10	50	60	20	1,4	0,35	1,7	1,3
12	30	100	60	20	2,2	3,93	1,7	2,1
13	30	50	20	60	2,9	5,8	3,6	2,9
14	30	100	20	60	14	11,63	13,5	13,9
15	30	50	60	60	1,8	3,05	1,8	1,8
16	10	100	60	60	12	11,13	11,74	11,26

This means that the analyzed process is a complicated system that cannot be described by a linear model. Therefore, for the sake of purity of calculations, it is necessary to consider not only linear terms b1x1 but also triple interactions of factors.

Thus, given the pairwise interactions of the factors, the regression model will look like this:

$$y_2=b_0+b_1\cdot x_1+b_2\cdot x_2+b_3\cdot x_3+b_4\cdot x_4\cdot b_{12}\cdot x_{12}+b_{13}\cdot x_{13}+b_{14}\cdot x_{14}+b_{23}\cdot x_{23}+b_{24}\cdot x_{24}+b_{34}\cdot x_{34} \qquad (14)$$

The regression equation with the effect of triple factor interactions:

$$y_3=b_0+b_1\cdot x_1+b_2\cdot x_2+b_3\cdot x_3+b_4\cdot x_4\cdot b_{12}\cdot x_{12}+b_{13}\cdot x_{13}+b_{14}\cdot x_{14}+b_{23}\cdot x_{23}+b_{24}\cdot x_{24}+b_{34}\cdot x_{34}+ b_{123}\cdot x_{123}+b_{124}\cdot x_{124}+b_{234}\cdot x_{234}+b_{134}\cdot x_{134}; \qquad (15)$$

The triple coefficients of the equation have the following values: b_{123} = 0,137; b_{124} = 0.387; b_{134} = -0.275; b_{234} = - 0.138.

The results of the calculation of the regression equations, taking into account the effect of paired and triple interactions (y2, y3) of the studied factors are given in Table 3 indicates that the model has a sufficiently high accuracy with regard to triple bonds, as evidenced by the considerable similarity between the experimental results.

Regression and correlation analysis were performed to determine the relationship between the independent variables and the optimization parameter (copper film thickness).

One of the main indicators that determine when conducting correlation analysis is the value of the Pearson multiple correlation coefficient (r), the calculation of which was carried out according to the following equation [14]:

$$r = \frac{\sum_{i=1}^{N}(x_i - \bar{x})\cdot(y_i - \bar{y})}{\sqrt{\sum_{i=1}^{N}(x_i - \bar{x})^2 \cdot (y_i - \bar{y})^2}}; \qquad (16)$$

where $(x_i - \bar{x})^2$ and $(y_i - \bar{y})^2$ - the square of the difference of the arithmetic mean of x and y.

Hence r = 0.916, indicating a statistically strong relationship between the varied parameters.

The second major parameter of the correlation analysis that determines the relationship between the varied factors and the optimization parameter is the multiple determination coefficient (r2), r2 = 0.84, i.e. the variance of the dependent variable is 84%. Thus, we can say that this set of independent variables (variable parameters) 84% affects the optimization parameter, the remaining 16% of the variation is provided by

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[14] Растригин Л.А. Статистические методы поиска / Л.А. Растригин. – М.: Наука, 1968.

the influence of other parameters not taken into account in the model. This is a high result.

The conducted regression analysis is presented in form of a graph in fig. 10.

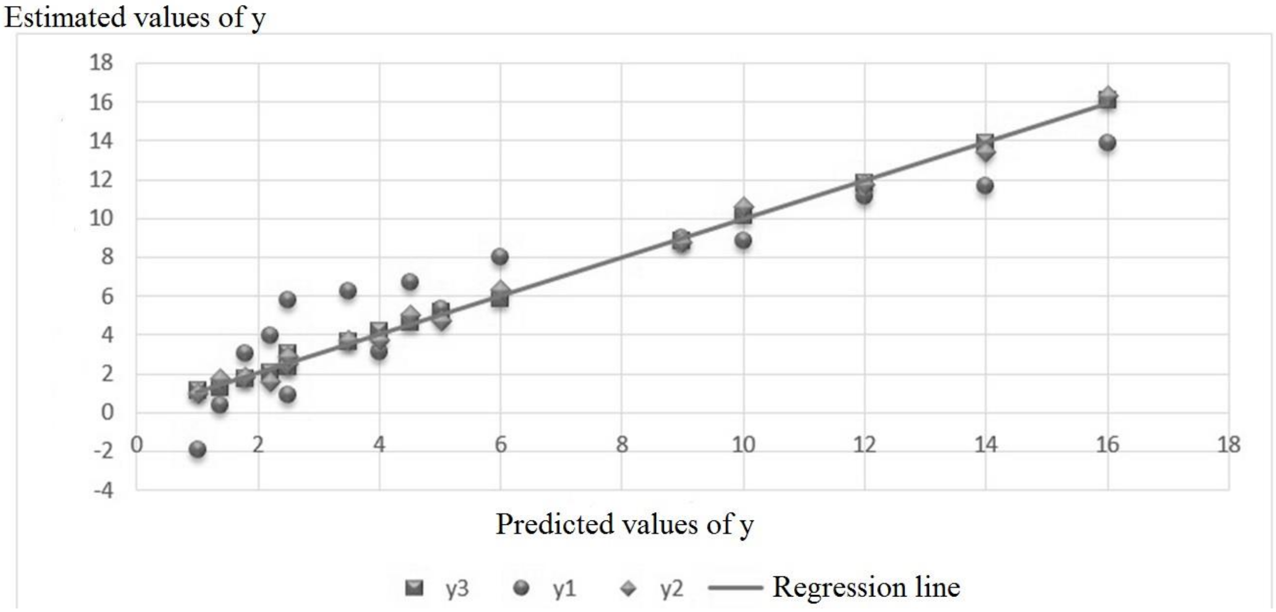

Fig. 10 The diagram of scattering of optimization parameter values

The significance of each of the equation`s coefficients was checked independently using the Student's t-test and constructing a confidence interval. Firstly, it is necessary to find the variance of the regression coefficient, which is calculated by the equation [15]:

$$S_{bj}^{2} = \frac{S_{від}^{2}}{N}; \qquad (17)$$

where S_{rep}^2 - the variance of reproducibility; N is the number of experiments.

Take into account the number of experiments conducted simultaneously, the reproducibility variance is defined as:

$$S_{від}^{2} = \frac{\sum_{u=1}^{NN}(y_u^0 - \bar{y}^0)}{NN-1}; \qquad (18)$$

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[15] Айвазян С.А. Статистическое исследование зависимостей / С.А. Айвазян. - М.: Металлургия, 1966.

where y_u^0 - the arithmetic value of optimization parameter; $\bar{y}^0$ - the arithmetic value of optimization parameter obtained in parallel experiments; NN - the number of parallel experiments. In this regard, in our case NN-1 = 4.

The calculated value of the Student's t-test is determined by the next equation:

$$t_j = \frac{|b_j|}{S_{bj}}; \qquad (19)$$

where $|b_j|$ the absolute value of the coefficient.

The calculations made by the eq. (19) are presented in the form of table 3. Comparison of the results of the calculation with the table value of the Student's t-test shown that for our number of degrees of freedom equal to t = 1,795 it can be said that the coefficients b_2, b_3, b_4 are significant in the range of confidence 0,9. The coefficient b_1 can be neglected. Building a confidence interval confirmed the previously obtained results. The absolute value of the first criterion b_1 is less than the upper bound of the confidence interval, indicating that it is not significant.

Table 3 - Summary table of results

The values of the coefficients	t-statistics, t = 1,795 for a 0.9 confidence	Confidence interval		Coefficient multiple correlations, r	Determination factor, r^2
		The lower boundary bj-Δbj	The upper boundary bj+Δbj		
b_0=5,987	-	-	-	0,916	0,84
b_1=-1,125	1,712	-0,608	-1,642		
b_2=2,913	4,431	1,347	2,742		
b_3=-1,375	2,092	-0,832	-1,286		
b_4=2,475	3,766	1,144	2,265		

CONCLUSIONS

Thus, the principal possibility of obtaining thin-film glass coatings deposited in the plasma of a glow discharge initiated in a hollow cathode at argon pressures in a gas chamber 26…53 Pa is shown.

It was found that at a Cu film thickness of 0.5… 1.2 μm obtained under these conditions, the adhesive strength fluctuates within 38… 32 MPa and decreases rapidly with the subsequent film expansion.

By means of using the statistical methods, it was found that among all the technological parameters, the most significant influence on the processes of formation and growth of thin-film metal coating is the discharge current I_d and the deposition duration t. It was found that the distance of the cathode-substrate $L_{c\text{-}s}$ and the pressure in the discharge chamber P lead to a significant decrease in the thickness of the deposited film.

It is established that the constructed regression mathematical model allows to obtain the results of theoretical calculation with 4 ... 6% error relative to experimental ones.

Conducted correlation and regression analyzes showed that the obtained model is statistically significant within the confidence limits of 0.9. The value of the multiple correlation coefficient is at 0.916, which indicates a close relationship between the input parameters and the optimization parameter.

The analysis found that the optimization parameter (copper film thickness) of 84% is described by the influence of the set of investigated mode parameters, the remaining 16% of the variation is provided by the influence of other parameters not taken into account in the model.

SUMMARY

The work considers the application of low temperature plasma of hollow cathode glow discharge for a thin metal films obtaining on a glass substrates at a gas pressures of 1 to 100 Pa. The substrate - cathode distance varied in a range of 5...30 mm. The discharge current I_d = 25… 100 mA, with the voltage on the electrodes fluctuating within 400… 800 V. It is shown that at a Cu film thickness of 0.5… 1.2 μm obtained under the given conditions, the adhesive strength fluctuates within 38… 32 MPa and decreases rapidly with further film expansion. Also, by using of mathematical modeling with statistical methods showed the feasibility of controlling the process of thin films deposition in plasma of glow discharge by two main technological parameters, namely the strength of the discharge current and the duration of deposition. It is established that the constructed regression mathematical model allows to obtain the results of theoretical calculation with 4 ... 6% error relative to experimental ones.

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