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Modeling the influence of technological parameters of the magnetron sputtering process using the Caroline D12C system on the proportion of nanocrystallites in the structure of thin silicon films

[1]Tolubaev K.S., [1]Zhautikov B.A., [1*]Zobnin N.N., [2]Dairbekova G.S., [1]Kabieva S.K., [3] Al-Kasasbeh R.T.

[1] Karaganda Industrial University, Temirtau, Kazakhstan;
[2] Satpayev University, Almaty, Kazakhstan;
[3]University of Jordan, Amman, Jordan

** Corresponding author email: zobninnn@mail.ru*

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ABSTRACT
The experimental dependence of the fraction of nano-sized modification of silicon in thin films obtained by magnetron sputtering on the main technological indicators of the process - specific power on the target, pressure in the working chamber, pulsation frequency of the voltage supplied to the target - has been studied. The data was processed using the method of multiple correlation-regression analysis and a corresponding mathematical model was obtained that describes the experimental dependence. It has been established that the specific power at the target does not significantly affect the fraction of nanosilicon in the film. The voltage frequency on the target has only a positive effect and is therefore limited only by the technical capabilities of the sputtering equipment. The pressure in the working chamber has an optimal value because in the mathematical model for this factor there are both positive and negative coefficients. When analyzing the model by calculation, it was found that the largest proportion of nanosilicon in the film, 75.06%, is achieved at a voltage frequency on the target of 100 Hz and pressure in the working chamber of 1.9 Pa. These data are preliminary due to the limited number of experiments.

Keywords: Nanosized silicon, magnetron sputtering, Caroline D12C, film, mathematical modeling, correlation-regression analysis, target.

Information about authors:

Tolubayev Kanat	*Doctoral student, Karaganda Industrial University, Temirtau, Kazakhstan. Email: kanat.tolubayev@tttu.edu.kz*
Zhautikov Bakhyt	*Doctor of Technical Sciences, Professor, Karaganda Industrial University, Temirtau, Kazakhstan. Email: bakhyt_zhautikov@mail.ru*
Zobnin Nikolai	*Candidate of Technical Sciences, Karaganda Industrial University, Temirtau, Kazakhstan. Email: zobninnn@mail.ru*
Dairbekova Guldana	*PhD, Satbayev University, Almaty, Kazakhstan. Email: guldana.01.02.91@mail.ru*
Kabiyeva Sauke	*Candidate of Chemical Sciences, Associate Professor, Karaganda Industrial University, Temirtau, Kazakhstan. Email: kabieva.s@mail.ru*
Al-Kasasbeh Riad	*PhD, Professor, University of Jordan, Amman, Jordan. Email: riad_alkasasbeh@bau.edu.jo*

Introduction

One of the newest applications for thin-film silicon structures is the production of anodes for commercial lithium-ion batteries (LIBs). During the introduction of lithium into nanosilicon, intermetallic compounds are formed Li_xSi_y. In the Li_{12} Si_7 compound, for every 7 silicon atoms, there are 12 lithium atoms, respectively, the volume of lithium is 2.17 times greater than the volume of silicon. The ability to intercalate lithium also influences the high theoretical specific capacity of silicon-based LIB anodes – 4140 mAh/g, which significantly exceeds the values for graphite (372 mAh/g) [[1], [2], [3]]. The literature substantiates that crystalline silicon cannot be used as a negative electrode material, since it is destroyed when lithium is introduced due to an increase in the volume of the crystal cell [[4], [5], [6]]. In this regard, when creating thin-film silicon anodes, it is important to control and influence the ratio of the main polymorphic states of silicon (crystalline, amorphous and nanocrystalline forms) in the finished products. In this case, it is necessary to strive to reduce the proportion of the crystalline form of silicon and increase the proportion of amorphous silicon or nanocrystals [[7], [8], [9]]. The ratio of various polymorphic phases of silicon in thin films can be changed by changing the main technological parameters of sputtering - specific power on the target, pressure in the working chamber, and pulsation frequency of the voltage supplied to the target. In this regard, modeling magnetron sputtering has become relevant to predict the polymorphism of thin silicon films and optimize the process.

Methodology

The most productive of the currently existing equipment options, the Caroline D12C system, was chosen to conduct experimental studies. The Caroline D12C vacuum deposition system (Figure 1) is intended for small- and medium-scale production and research in the field of thin film deposition using magnetron and thermal sputtering. The technical characteristics of the system are given in Table 1 [10].

Table 1 - Technical characteristics of the Caroline D12C magnetron sputtering system [10]

Number of substrates processed per 1 cycle (pcs.)	12pcs. Ø100 mm. 24 pcs. 60x48
Starting pressure in the working chamber, Pa	10^{-3}
Flow rate of working gases supplied to the chamber through one channel (l/hour)	0÷9
Quantity of supplied (non-aggressive) gases up to (pcs.)	3
Number of thermal evaporators up to (pcs.)	2
Number of magnetrons (pcs.)	1÷4
Type of magnetrons for film deposition	Pulse high frequency
Operating current of magnetrons, adjustable (A)	0.5÷23
Operating voltage of magnetrons (V)	300÷650
Target material and size: metals, alloys, silicon (composite targets not directly cooled can be used), mm	Ø 100×4÷12
Working pressure in the vacuum chamber, Pa	0.1 ÷ 3.0
Witness resistance control range (kOhm)	0.2÷20
Resistance measurement error (%)	±3
Recommended substrate heating temperature, °C	10÷550
Substrate temperature instability (%)	±15
Limit residual pressure in the working chamber, (Pa)	$2x10^{-4}$
Time to prepare the installation for operation, taking into account the “overclocking” of the cryopump, no longer (min.)	110
Weight with power and control stand, kg.	Up to 2250

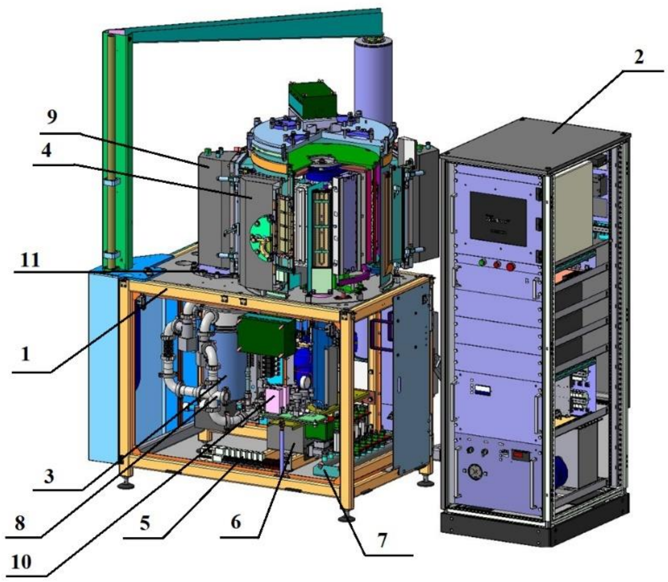


Fig. 1 - Diagram of the Caroline D12C magnetron sputtering system [6] 1 - technological module, 2 - control cabinet, 3 - fore-vacuum pump, 4 - working chamber, 5 - vacuum system, 6 - pneumatic equipment panel, 7 - water block, 8 - hydraulic equipment block, 9 - lifting and turning mechanism, 10 - gas supply panel, 11 – shutter

The system includes a process module (1), a control cabinet (2) and a foreline pump (3) (Figure 1). The main part of the technological module is the working chamber (4) with technological and internal chamber devices. These elements are installed on a plate, which is fixed in a frame structure. Devices to ensure the operation of the system are located under the supporting structure. The vacuum system (5), as part of the cryogenic pump, is installed on the plate below. The shutter (11) separates the vacuum system from the working chamber. The pneumatic equipment panel (6) is designed to distribute compressed air among the pneumatic actuators. The water block (7) ensures the distribution of water cooling to individual parts of the system. The hydraulic unit (8) is designed to create pressure in the hydraulic cylinder of the lifting and turning mechanism. Lift and rotate mechanism (9) for opening and rotating the chamber cover. The gas supply panel (10) is installed isolated from the housing on a special bracket under the stove.

When preparing samples of silicon films, the following technological parameters of the magnetron sputtering process were varied:

1) Specific power on targets. The variation was carried out in the maximum possible range from 1 to 100 W/cm^2, taking into account the available technical capabilities of modern production equipment;

2) Operating pressure in the spray chamber. Chamber pressure is regulated by changing the amount of inert gas supplied to the chamber. The pressure control range was from 0.5 to 3.0 Pa. It was assumed that increasing the gas flow rate would promote better sputtering of the silicon flow from the target to the substrate and the formation of silicon nanoparticles, as well as improved heat removal from condensing particles to avoid coalescence on the substrate;

3) The frequency of the voltage pulses applied to the target. At carrying out experiments applied constant (without pulse) voltage and two voltage frequency options were 20 and 100 kHz.

Silicon crystals were used as targets for magnetron sputtering. To produce silicon crystals, we used TCR-5C-1k/t equipment manufactured by Techno Search Corporation (Japan). The crystals were grown according to the Czochralski method using commercial high-purity silicon of SoG-Si grade 6-7N.

A preliminary assessment of the film morphology was carried out using the ELLIPS-1891 spectral ellipsometer designed for precision measurements of the thickness of thin films, the optical parameters of thin-film structures and the spectral dependences of the optical constants of the surfaces of various materials (metals, semiconductors, dielectrics, etc.). When the uniform morphology of the silicon film was revealed and the absence of a substrate surface not covered by the film, further determination of the thickness of the resulting film was carried out using a Jeol JSM-6490LA scanning electron microscope with a magnification range of up to 300,000 times.

The ratio of various polymorphic states of silicon in the film was assessed using Raman spectroscopic analysis on a Horiba system Jobin – Yvon HR800UV (France).

Results and Discussions

During the study, six silicon films were obtained under different technological conditions of magnetron sputtering. Each of the resulting films was studied using Raman spectroscopy and electron microscopy at several different points. This test showed the uniformity of polymorphism across the entire surface of each film, which indicates the advantage of magnetron sputtering technology over the CVD method in terms of ensuring film uniformity. The technological parameters of spraying and the results obtained are presented in Table 2.

Table 2 - Technological parameters of deposition and the proportion of polymorphic modifications in the structure of silicon films

# experience	Magnetron sputtering parameters and results					
	Specific power at the target, W/cm^2 (X1)	Pressure, Pa (X2)	Voltage frequency, kHz (X3)	Proportion of polymorphic state, volume. %		
				A	C	n-C (Y)
1	1	0.5	0	76.1	23.9	0
2	50	0.5	0	56.2	43.8	0
3	50	2.0	0	49.4	5.0	45.6
4	100	3.0	0	2.8	82.1	15.9
5	50	2.0	20	34.2	1.8	64.0
6	50	3.0	100	58.7	5.4	35.9

A – amorphous, C – crystalline, n-C – nanocrystalline

Figure 2 shows micrographs of two types of films that were obtained during the experiments. The first type of films (Figure 2 a), obtained in experiments No. 1-2, do not contain nano-sized silicon, the second type of films (Figure 2 b), obtained in experiments No. 3-6, contains it in varying quantities. Films similar to those presented in Figure 2a were previously obtained by the authors of [11].

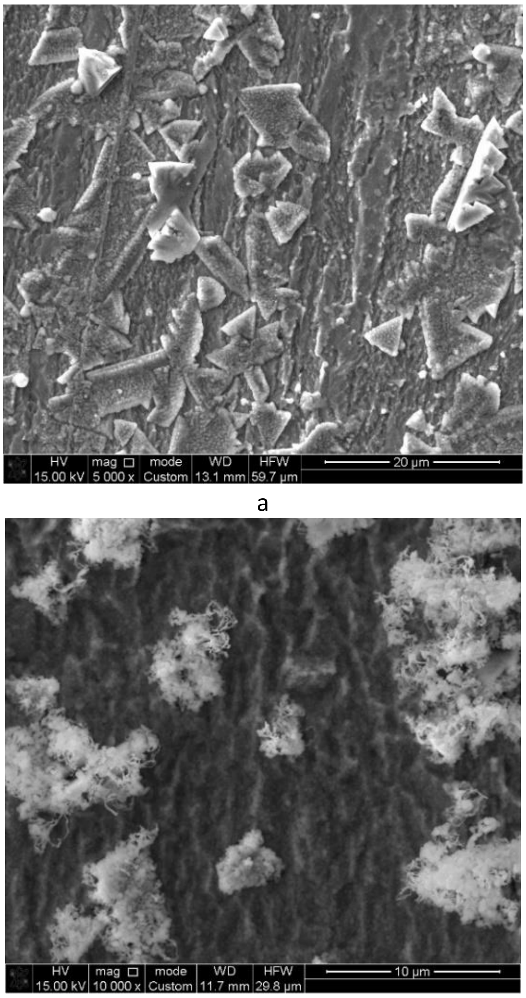
Structures similar to Figure 2b were obtained by the authors of, however, the diameter of these structures (about 5 μm) is much larger than those obtained in this work (20-100 nm) [12]. They can be defined more as microtubes rather than nanofibers as in our case. At a higher magnification of microtubes, it was established that at the nano level, these tubes are represented by spherical nanocrystallites with a diameter of 100-150 nm [12]. These structures are also described by theoretical researchers. One of the options for a visual description of nanosilicon is the statement that this polymorphic structure has a spherical shape with a diameter of 3-10 nm [13]. The same data is provided from other sources regarding the size of silicon nanospheres - 3.5-20 nm [14]. However, the authors did not provide the corresponding microphotographs. Results most similar to the image in Figure 2b were obtained by a number of authors [[15], [16], [17], [18], [19], [20], [21]]. However, unlike these studies, we were able to obtain thinner fibres, which, when interwoven, formed a cellular porous structure at the microlevel. In addition, previous studies did not address the question of what is the proportion of silicon nanocrystallites in the structure of the resulting material. Are there, and in what quantities, amorphous and crystalline structures? Also, no works were found in which the influence of technological parameters of the industrial deposition process on the shape of nanocrystallites and their proportion in the composition of thin films was studied.

The preliminary data obtained (Table 1) were subjected to mathematical processing using the method of multiple correlation-regression analysis with the inclusion of linear, quadratic and cubic terms of the regression model, excluding paired and triple interactions between factors. As a result, a regression model (formula 1) was obtained that relates the parameters of magnetron sputtering (X1, X2, X3) with the fraction of nanosilicon in the composition of the resulting silicon film (Y).

$$Y = -30.7846 + 62.897 \times X2 + 0.2277 \times X3 - 5.3108 \times X2^2 \qquad (1)$$

The resulting model is characterized by the following statistical characteristics:

Dispersion of inadequacy (S^2) = 49.8456
Reduced sum of squares = 3275.6 from 3375.3
F-ratio = 21.905
Multiple correlation coefficient = 0.9851



a

B

Fig. 2 - Microphotographs of the surface of a thin silicon film of magnetron sputtering
a – first type, b – second type

Table 3 presents an assessment of the adequacy of the data found by calculation using the resulting model. As can be seen from the table data, the maximum relative deviation of the calculated data from the experimental values is 15.18%. In combination with the above statistical characteristics of the model, this indicates a high level of adequacy of the resulting model of the magnetron sputtering process.

Table 3 - Assessment of the adequacy of the model of magnetron sputtering of silicon on a copper substrate

# Experiment	Proportion of nanosilicon in the film (Y), %		Deviation	
	Calculation	Experiment	Absolute	%
1	0.00	0.00	0.00	0.00
2	0.00	0.00	0.00	0.00
3	45.60	52.52	-6.92	15.18
4	15.90	14.51	1.38	8.71
5	64.00	57.07	6.92	10.82
6	35.90	37.28	-1.38	3.86

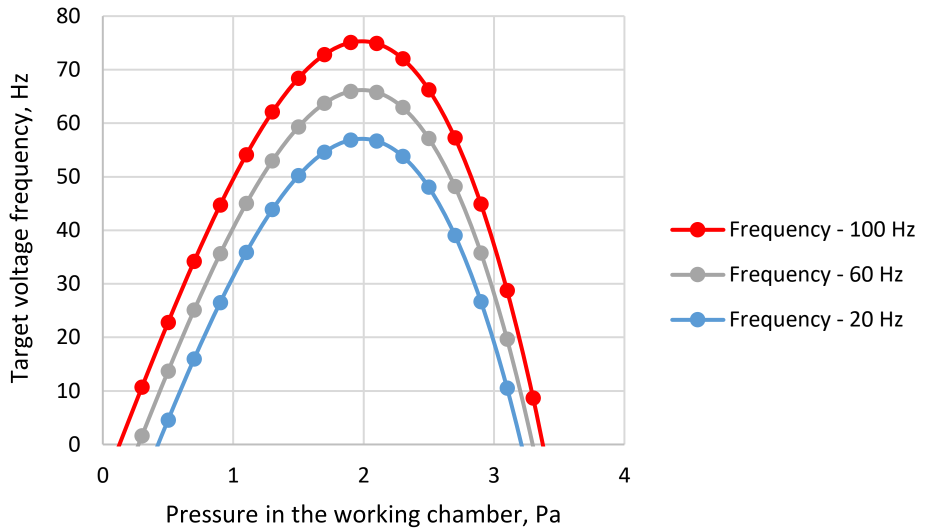


Fig.3 - **Calculated** graph of the dependence of the fraction of nanosilicon in the film on the pressure in the working chamber at different voltage frequencies on the target

When analyzing the resulting model, the assumption is confirmed that the specific power at the target (X1) does not significantly affect the fraction of nanosilicon in the film. The voltage frequency on the target has only a positive effect and is therefore limited only by the technical capabilities of the sputtering equipment. The pressure in the working chamber must be at its optimal value, because in the model for factor X2 there are both positive and negative coefficients.

Figure 3 shows a graph of the dependence of the fraction of nanosilicon in the film on the pressure in the working chamber, calculated using the model obtained here, at different voltage frequencies on the target.

In accordance with Figure 3, the largest proportion of nanosilicon in the film, 75.06%, is achieved at a voltage frequency on the target of 100 Hz and a pressure in the working chamber of 1.9 Pa. Of course, it should be understood that due to data limitations, this result may not be accurate. To improve the process model and clarify the optimal values of technological parameters of magnetron sputtering, more experimental data are needed.

Conclusions

In the process of studying magnetron sputtering of silicon films, it was found that the greatest influence on increasing the proportion of nano silicon in the film composition is exerted by an increase in the pressure in the working chamber and the frequency of voltage pulses on the target. An increase in the specific power at the target leads to a reduction in the proportion of the amorphous phase and an increase in the crystalline phase, but this indicator does not affect the increase in the proportion of nanocrystalline silicon. Increasing the pressure in the working chamber and the voltage frequency helps to increase the proportion of nanosilicon. However, there is probably a limit to these parameters, beyond which a further increase in their values reduces the share of nanosilicon. The silicon film obtained under experimental conditions acquires a porous cellular structure resulting from the interweaving of silicon nanofibers. Based on experimental data, a correlation-regression model of the process of magnetron sputtering of silicon on a copper substrate was constructed with a high level of adequacy within the limits of the experiments. Using the obtained model, preliminary optimal parameters of the sputtering process were calculated. The calculated maximum fraction of nanosilicon in the film - 75.06%, is achieved at a voltage frequency on the target of 100 Hz and pressure in the working chamber of 1.9 Pa. Additional experiments are needed to identify more accurate optimal values of magnetron sputtering parameters and to test the resulting silicon films as anodes of lithium-ion batteries.

CRediT author statement: **K.Tolubaev**: Preparation and implementation of the magnetron sputtering process, generalization of the research results. **B.Zhautikov**: Growing silicon crystals to prepare a target for a magnetron. **N.Zobnin**: Mathematical data processing and establishment of a model of the magnetron sputtering process, working with the resulting model to optimize the process. **G.Dairbekova**: Electron microscopy of silicon film samples obtained by magnetron sputtering. **S. Kabieva**: Raman spectroscopy and interpretation of the obtained spectra. **R. Al-Kasasbeh**: deconvolution of the Raman spectra of silicon nanofilms, comparison of the results obtained with available literature sources.

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Магнетронды шашырату процесінің технологиялық параметрлерінің Caroline D12C жүйесіндегі жұқа кремний қабықшаларының құрылымындағы нанокристаллиттер үлесіне әсерін модельдеу

[1]Толубаев Қ.С., [1]Жаутиков Б.А., [1*]Зобнин Н.Н., [2]Даирбекова Г.С., [1]Кабиева С.К., [3] Al-Kasasbeh R.T.

[1] Қарағанды индустриялық университеті, Теміртау, Қазақстан
[2] Сәтбаев университеті, Алматы, Қазақстан
[3]Иордания университеті, Амман, Иордания

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ТҮЙІНДЕМЕ
Магнетронды бүрку әдісімен алынған жұқа пленкалардағы кремнийдің нано өлшемді модификациясының үлесінің процестің негізгі технологиялық көрсеткіштеріне - нысанадағы меншікті қуатқа, жұмыс камерасындағы қысымға, нысанаға берілетін кернеудің пульсация жиілігіне эксперименттік тәуелділігі зерттелді. Деректер бірнеше корреляциялық-регрессиялық талдау әдісімен өңделді және эксперименттік тәуелділікті сипаттайтын сәйкес математикалық модель алынды. Нысанадағы меншікті қуат пленкадағы нано кремнийдің үлесіне айтарлықтай әсер етпейтіні анықталды. Нысанаға кернеу жиілігі тек оң әсер етеді, сондықтан тек бүрку жабдықтарының техникалық мүмкіндіктерімен шектеледі. Жұмыс камерасындағы қысымның оңтайлы мәні бар, өйткені математикалық модельде бұл фактор оң және теріс коэффициенттерге ие. Модельді есептеу арқылы талдау кезінде пленкадағы нано кремнийдің ең үлкен үлесі 75,06% 100 Гц нысанадағы кернеу жиілігінде және 1,9 Па жұмыс камерасындағы қысымда қол жеткізілетіні анықталды. Бұл деректер эксперименттердің шектеулі санына байланысты алдын ала берілген түрі болып табылады.

Түйін сөздер: Нано өлшемді кремний, магнетронды бүрку, Caroline D12C, пленка, математикалық модельдеу, корреляциялық-регрессиялық талдау, нысан.

	Авторлар туралы мәліметтер:
Толубаев Қанат	*Докторант, Қарағанды индустриялық университеті, Теміртау, Қазақстан. Email: kanat.tolubayev@tttu.edu.kz*
Жаутиков Бахыт Ахатович	*Техника ғылымдарының докторы, профессор, Қарағанды индустриялық университеті, Теміртау, Қазақстан. Email: bakhyt_zhautikov@mail.ru*
Зобнин Николай Николаевич	*Техника ғылымдарының кандидаты, Қарағанды индустриялық университеті, Теміртау, Қазақстан. Email: zobninnn@mail.ru*
Даирбекова Гулдана Сиюндыковна	*PhD, Сәтбаев университеті, Алматы, Қазақстан. Email: guldana.01.02.91@mail.ru*

Кабиева Сауле Казжановна	*Химия ғылымдарының кандидаты, доцент, Қарағанды индустриялық университеті, Теміртау, Қазақстан. Email: kabieva.s@mail.ru*
Al-Kasasbeh Riad	*PhD, профессор, Иордания университеті, Амман, Иордания. Email: riad_alkasasbeh@bau.edu.jo*

Моделирование влияния технологических параметров процесса магнетронного распыления на системе Caroline D12C на долю нанокристаллитов в структуре тонких пленок кремния

[1]Толубаев К.С., [1]Жаутиков Б.А., [1*]Зобнин Н.Н., [2]Даирбекова Г.С., [1]Кабиева С.К., [3] Al-Kasasbeh R.T.

[1] Карагандинский индустриальный университет, Темиртау, Казахстан
[2] Сатпаев университет, Алматы, Казахстан
[3]Университет Иордании, Амман, Иордания

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АННОТАЦИЯ
Изучена экспериментальная зависимость доли наноразмерной модификации кремния в тонких плёнках, полученных методом магнетронного напыления от основных технологических показателей процесса - удельной мощности на мишени, давления в рабочей камере, частоте пульсации напряжения, подаваемого на мишень. Данные обработаны методом множественного корреляционно-регрессионного анализа и получена соответствующая математическая модель, описывающая экспериментальную зависимость. Установлено, что удельная мощность на мишени не влияет существенным образом на долю нано кремния в плёнке. Частота напряжения на мишени влияет только положительным образом и потому ограничивается только техническими возможностями оборудования напыления. Давление в рабочей камере имеет оптимальное значение, т.к. в математической модели при этом факторе имеются как положительные, так и отрицательные коэффициенты. При анализе модели расчётным путём установлено, что наибольшая доля нано кремния в плёнке 75,06% достигается при частоте напряжения на мишени 100 Гц и давлении в рабочей камере 1,9 Па. Эти данные предварительные в силу ограниченного количества экспериментов.

Ключевые слова: Наноразмерный кремний, магнетронное напыление, Caroline D12C, плёнка, математическое моделирование, корреляционно-регрессионный анализ, мишень.

Информация об авторах:

Толубаев Канат	*Докторант, Карагандинский индустриальный университет, Темиртау, Казахстан. Email: kanat.tolubayev@tttu.edu.kz*
Жаутиков Бахыт Ахатович	*Доктор технических наук, профессор, Карагандинский индустриальный университет, Темиртау, Казахстан. Email: bakhyt_zhautikov@mail.ru*
Зобнин Николай Николаевич	*Кандидат технических наук, Карагандинский индустриальный университет, Темиртау, Казахстан. Email: zobninnn@mail.ru*
Даирбекова Гулдана Сиюндыковна	*PhD, Сатпаев университет, Алматы, Казахстан. Email: guldana.01.02.91@mail.ru*
Кабиева Сауле Казжановна	*Кандидат химических наук, доцент, Карагандинский индустриальный университет, Темиртау, Казахстан. Email: kabieva.s@mail.ru*
Al-Kasasbeh Riad	*PhD, Профессор, Иорданский университет, Амман, Иордания. Email: riad_alkasasbeh@bau.edu.jo*

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