UDC 537.311:322
IRSTI 29.19.22:47.09.48
https://doi.org/10.55452/1998-6688-2023-20-2-27-35

Nussupov K.Kh.[1], Beisenkhanov N.B.[1]*, Kukushkin S.A.[2], Sultanov A.T.[1], Keiinbay S.[1], Shynybayev D.S.[1], Kusainova A.Zh.[1]
[1]Kazakhstan-British Technical University, 050000, Almaty, Kazakhstan
[2]Federal State Budgetary Institution of Science "Institute of Problems of Mechanical Engineering, Russian Academy of Sciences", 199178, St. Petersburg, Russia
*E-mail: beisen@mail.ru

FORMATION OF CRYSTALLINE SiC IN NEAR-SURFACE SILICON LAYERS BY METHOD OF COORDINATED SUBSTITUTION OF ATOMS

Abstract. In this work, monocrystalline films of silicon carbide were synthesized on the surface of a Si(100) silicon wafer using the method of coordinated substitution of atoms. The films were synthesized at temperatures of 1200 °C and 1300 °C for 20 minutes in a CO gas flow at a pressure of 0.8 Pa. The effect of 1200–1300 °C temperatures on the formation of single- and polycrystalline layers, as well as nanostructured SiC phases in the near-surface region of silicon by the method of atom substitution, is analyzed. The formation of a high-quality crystalline silicon carbide film and the influence of synthesis conditions on the total volume of SiC structural phases, microstructure and nanostructure of the surface are shown. It was found that an increase in temperature from 1200 °C to 1300 °C led to a more intensive formation of silicon carbide and an increase in the number of Si–C bonds by 1.9 times due to an increase in the thickness of the synthesized silicon carbide layer. There is an increase in the proportion of the crystalline phase due to a more intense transformation of the nuclei of nanocrystals into micro- and nanocrystals. Intense processes of penetration of carbon atoms deep into silicon at a temperature of 1300 °C with amorphization of its structure and the formation of Si-C, which can transform into crystalline phases at temperatures above 1300 °C, are assumed. The proportion of the SiC crystalline phase increases to 50.2% of the film volume due to the intensive transformation of nanocrystal nuclei into micro- and nanocrystals. It has been experimentally shown that the formation of various SiC structures on Si (100) occurs in full accordance with the main principles of the method of coordinated substitution of atoms.

Key words: silicon carbide, structure, crystallization, synthesis, method of coordinated substitution of atoms.

Нусупов К.Х.[1], Бейсенханов Н.Б.[1]*, Кукушкин С.А.[2], Султанов А.Т.[1], Кейінбай С.[1], Шыныбаев Д.С.[1], Кусайнова А.Ж.[1]
Қазақстан-Британ техникалық университеті, 050000, Алматы қ., Қазақстан
Ресей ғылым академиясының Машина жасау мәселелері институтының Ғылым Федералдық мемлекеттік бюджеттік мекемесі, 199178, Санкт-Петербург қ., Ресей
*E-mail: beisen@mail.ru

АТОМДАРДЫ СӘЙКЕС АЛМАСТЫРУ ӘДІСІ АРҚЫЛЫ КРЕМНИЙ ҚАБАТТАРЫНЫҢ БЕТУСТІНЕ КРИСТАЛДЫҚ SiC ТҮЗІЛУІ

Аңдатпа: Бұл жұмыста Si(100) кремний тілігінің бетінде атомдарды сәйкес алмастыру әдісімен кремний карбидінің монокристалды қабықшалары синтезделді. Қабықшалар 0,8 Па қысымдағы CO газ ағынында 20 минут ішінде 1200 °C және 1300 °C температураларда синтезделді. 1200–1300 °C температураларының атомдарды алмастыру әдісімен кремнийдің бетке жақын аймағындағы моно және поликристалды қабаттардың, сондай-ақ наноқұрылымды SiC фазаларының түзілуіне әсері талданған. Жоғары сапалы кристалды кремний карбидті қабыршақтың түзілуі және синтез жағдайларының SiC құрылымдық фазаларының жалпы көлеміне, бетінің микроқұрылымы мен наноқұрылымына әсері көрсетілген. Температураның 1200 °C-тан 1300 °C-қа дейін жоғарылауы кремний карбидінің қарқынды түзілуіне және синтезделген кремний карбиді қабатының қалыңдығының ұлғаюына байланысты Si–C байланыстарының санының 1,9 есе артуына әкелетіні анықталды. Нанокристалдар ядроларының микро және нанокристалдарға неғұрлым қарқынды түрленуіне байланысты кристалдық фазаның үлес салмағының артуы байқалады. Көміртек атомдарының кремнийге 1300 °C температурада терең енуінің қарқынды процестері оның құрылымының аморфизациялануы және 1300 °C жоғары температурада кристалдық фазаларға айналуы мүмкін Si-C байланыстарының түзілуі болып табылады. SiC кристалдық фазасының үлесі нанокристалдық ядролардың микро және нанокристалдарға қарқынды трансформациясы есебінен қабыршақтың көлемінің 50,2% дейін артады. Si (100) бетінде әртүрлі SiC құрылымдарының түзілуі атомдарды сәйкес алмастыру әдісінің негізгі принциптеріне толық сәйкес келетіні тәжірибе жүзінде көрсетілген.

Тірек сөздер: кремний карбиді, құрылым, кристалдану, синтездеу, атомдарды сәйкес алмастыру әдісі.

Нусупов К.Х.[1]**, Бейсенханов Н.Б.**[1]*****, Кукушкин С.А.**[2]**, Султанов А.Т.**[1]**, Кейінбай С.**[1]**,**
Шыныбаев Д.С.[1]**, Кусайнова А.Ж.**[1]
[1]Казахстанско-Британский технический университет, 050000, г. Алматы, Казахстан
[2]Федеральное государственное бюджетное учреждение науки Институт проблем машиноведения
Российской академии наук, 199178, г. Санкт-Петербург, Россия
*E-mail: beisen@mail.ru

ФОРМИРОВАНИЕ КРИСТАЛЛИЧЕСКОГО SiC В ПРИПОВЕРХНОСТНЫХ СЛОЯХ КРЕМНИЯ МЕТОДОМ СОГЛАСОВАННОГО ЗАМЕЩЕНИЯ АТОМОВ

Аннотация. В работе на поверхности кремниевой пластины Si(100) методом согласованного замещения атомов синтезированы монокристаллические пленки карбида кремния. Синтез пленок осуществлен при температурах 1200 °C и 1300 °C в течение 20 минут в потоке газа CO при давлении 0.8 Па. Анализируется влияние температур 1200–1300 °C на формирование методом замещения атомов моно- и поликристаллического слоев, а также наноструктурированных фаз SiC в приповерхностной области кремния. Показано формирование высококачественной кристаллической пленки карбида кремния и влияние условий синтеза на общий объем структурных фаз SiC, микроструктуру и наноструктуру поверхности. Обнаружено, что увеличение температуры с 1200 °C до 1300 °C привело к более интенсивному формированию карбида кремния и росту количества Si-C-связей в 1,9 раза вследствие увеличения толщины синтезированного слоя карбида кремния. Происходит увеличение доли кристаллической фазы за счет более интенсивной трансформации зародышей нанокристаллов в микро- и нанокристаллы. Предположены интенсивные процессы проникновения атомов углерода вглубь кремния при температуре 1300 °C с аморфизацией его структуры и образованием Si-C-связей, которые могут трансформироваться в кристаллические фазы при температурах выше 1300 °C. Доля кристаллической фазы SiC увеличивается до 50,2% объема пленки за счет интенсивной трансформации зародышей нанокристаллов в микро- и нанокристаллы. Экспериментально показано, что формирование разнообразных структур SiC на Si (100) происходит в полном соответствии с основными положениями метода согласованного замещения атомов.

Ключевые слова: карбид кремния, структура, кристаллизация, синтез, метод согласованного замещения атомов

Introduction

In 2008, a method was discovered for synthesizing thin SiC epitaxial films on Si based on the coordinated substitution of a part of silicon atoms for carbon atoms inside the near-surface Si layer [1]. The term "the coordinated substitution" means that new chemical bonds between Si and C atoms are formed simultaneously and in coordination with the destruction of old Si-Si bonds in silicon, which leads to the preservation of the overall structure of chemical bonds. The Method of Coordinated Substitution of Atoms (MCSA) is based on the use of the chemical reaction $2Si_{solid} + CO_{gas} = SiC_{solid} + SiO_{gas}$, proposed in [1], which proceeds in two stages.

At the first stage of the reaction, the CO molecule interacts with the surface of the silicon substrate and decomposes into a carbon atom and an oxygen atom. The oxygen atom enters a chemical reaction with the Si atom, which results in the formation of SiO gas. The SiO gas is removed from the system, and a vacancy is formed in place of the silicon atom of the substrate, which was included in the composition of the SiO gas. The active carbon atom released because of a chemical reaction from a CO molecule is shifted to an interstitial position in the silicon lattice. As a result of the shift of carbon to the interstitial position, an intermediate phase of the so-called “pre-carbide” silicon is formed. This phase is silicon saturated with defect pairs $C + V_{Si}$ [2,3], where C is a carbon atom in the interstitial position in silicon, V_{Si} is a silicon vacancy formed because of the removal of a silicon atom in the composition of the SiO_{gas} gas. One Si cell contains 4 pairs of $C + V_{Si}$ dilatation defects, i.e. in "pre-carbide" silicon, everything is ready for the transformation of silicon into silicon carbide.

At this stage, the carbon atoms move in a coordinated manner towards the silicon vacancies, forming silicon carbide [1]. A detailed analysis of these processes is described in a series of works summarized in reviews [1,2,4].

The method of coordinated substitution of atoms in the synthesis of epitaxial SiC films has such advantages as the possibility of obtaining high-quality crystalline films on the surface of single-crystal silicon.

Main provisions

In this experiment, silicon carbide SiC is synthesized on the (100) Si face at low CO pressure by the method of coordinated substitution of atoms.

As is known [5,6], the slip planes in crystals with a diamond lattice at temperatures exceeding 0.5 of the melting point of the crystal are the family of (111) planes, and the sliding occurs along [110] directions. Therefore, on a smooth (100) silicon surface, SiC of the (100) orientation cannot be nucleated during growth by the MCSA method. The (100) Si face upon conversion transforms into a SiC face consisting of many facets resembling sawtooth structures, the side faces of which are covered with the (111), (110), and (210) planes.

The theory of growth of SiC on Si (100) by the MCSA method was constructed in [7]. In this paper, a formula was derived that relates the pressure of the CO gas to the synthesis temperature of the SiC layer. According to this formula, only at a high CO pressure, about $3 \cdot 10^3$ Pa and, for example, at a temperature of 1250°C, is it possible to form a SiC film of orientation (100), i.e. at this CO gas pressure, only the SiC(100) face on Si(100) will form. However, at such a high pressure, according to [8], the thickness of the SiC layer cannot grow more than a few nanometers. At lower pressures, (111), (110), (210) and other faces will form. Various nanostructures and SiC polytypes will form on the Si(100) surface. In this case, the film structure will be mainly polycrystalline, although there will also be epitaxial facets of SiC orientations (111) of the cubic SiC modification or hexagonal SiC polytypes, but having a nonpolar orientation (not the 0001 orientation). The formation of this kind of nanocrystals is of interest for various applications. That is why the purpose of this work was to study the processes of formation of various SiC structures on the (100) Si face at low CO pressure, at which CO intensively penetrates deep into Si and is intensively converted into Si.

Materials and Methods

The structure of SiC nanolayers synthesized by the atom substitution method on the surface of c-Si (100) wafers 7 × 7 × 0.3 mm in size and with a resistivity of 4–5 Ω cm was studied using IR spectroscopy and scanning electron microscopy. The SiC films were synthesized in a special electric furnace at a temperature of 1200 and 1300°C for 20 minutes in a CO gas flow at a pressure of 0.8 Pa [9].

To analyze the composition and structure of silicon carbide films, IR absorption spectra were obtained using a Nicolet iS-50 IR spectrometer (ThermoScientific, USA). The software for the spectrometer makes it possible to decompose infrared spectra into components and determine the position, amplitude, and area of the maxima. Main technical characteristics of Nicolet iS50: signal-to-noise ratio - 55000, wavenumber scale error limits - ± 0.01 cm^{-1}; resolution - 0.09 cm^{-1}; beam splitter: XT-KBr (11000–375 cm^{-1}) [10-12].

The surface of the samples was studied using a Quanta 200i 3D scanning electron microscope (FEI Company) with a directly heated tungsten cathode designed for qualitative and quantitative analysis of nanosized objects. The microscope is equipped with a thermionic gun and a station with a focused ion beam with a built-in system for energy dispersive microanalysis and analysis of the structure and texture of materials. Electronic optics: accelerating voltage from 200 V to 30 kV and high vacuum resolution of 3 nm. Ion optics: accelerating voltage from 2 to 30 kV and resolution at 30 kV - 9 nm.

Results and discussion

To analyze the structural quality of the films, the IR absorption spectra of the films were decomposed into components. The position of the component maxima in the spectrum was determined and their area was calculated. These data can be used to estimate the degree of crystallinity and phase composition of the films. The data obtained for a film synthesized at a temperature of 1200°C, a pressure of 0.8 Pa for 20 min are shown in Figure 1.

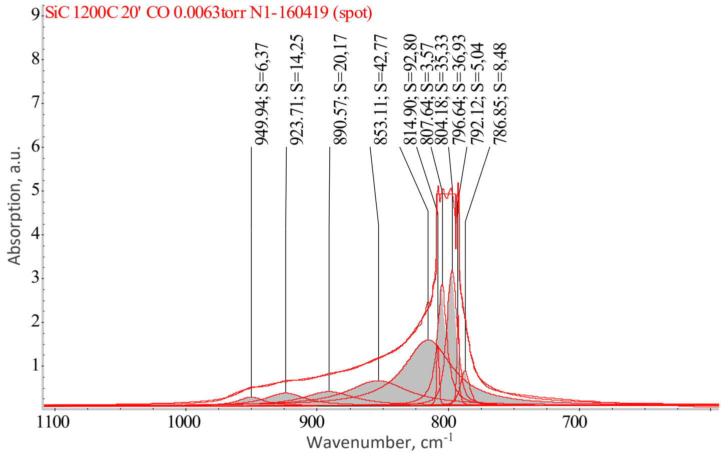

Figure 1 – Mathematical decomposition of the IR spectrum of the SiC film synthesized in an atmosphere of CO gas (1200 °C, 0.8 Pa, 20 min)
by the method of atom substitution

As indicated in [13], the theoretical values of the infrared absorption peaks for cubic, hexagonal and rhombic modifications are 795.9 cm^{-1} for β-SiC (3C-SiC), 799.5 cm^{-1} for 2H-SiC, 797.6 cm^{-1} for 4H-SiC, 797.0 cm^{-1} for 6H-SiC, 797.5 cm^{-1} for 15R-SiC. Since the values obtained in the experiment may have insignificant differences, it can be assumed that the sample contains a monocrystalline phase of SiC (maximum at 796.64cm^{-1}).

The total area of the SiC peak of the IR spectrum was 266 a.u. (Figure 1). If intense IR maxima, namely, intense peaks at 792.12-807.64 cm^{-1}, are identified with the crystalline, micro- and nanocrystalline phases, then their total area is ~81 a.u., or 30.4% of the IR- spectrum and volume of the SiC phase. The amorphous phase, close to the defective crystalline phase (peak 786.85 cm^{-1}) [10,11,14], is 8.48 a.u., or 3.2% of the volume of the SiC phase. There are also nanocrystal nuclei and carbon-vacancy structures (spectrum components in the range 814.90–949.94 cm^{-1}), which total 176 a.u., or 66.4% of the SiC phase [11].

It was found that an increase in temperature to 1300 °C led to a more intense formation of silicon carbide and an increase in the number of Si–C bonds by a factor of 1.9 (from 266 to 499 a.u.). These data do not contradict the provisions of review [4] on an increase in the thickness of silicon carbide layers with an increase in the synthesis temperature. All components grew (Figure 2). As is known [14], even in the case of crystallization of amorphous silicon carbide layers obtained by ion implantation, an increase in the fraction of the crystalline phase is observed from 40–50% at 900°C to 70–80% at 1200°C.

An increase in the volume and proportion of the amorphous component of SiC from ~8.5 a.u. (3.2%) to ~93 a.u. (18.7%) indicates a more intense absorption of carbon atoms and the formation of amorphous structures in the "film-substrate" transition layer. This temperature turned out to be insufficient for the crystallization of these structures. The presence of an intense sharp peak at 785 cm^{-1} (Figure 2) indicates the absorption of radiation by elongated Si–C bonds corresponding to the defective SiC phase, which is transitional between amorphous and crystalline in terms of its structural quality. The presence of a broad component with a maximum at 748.77 cm^{-1} may indicate intense processes of penetration of carbon atoms deep into silicon with amorphization of its structure and the formation of Si-C bonds in addition to dilatation dipoles. However, it should be taken into account that the results of studies of silicon carbide films synthesized by ion implantation indicate the impossibility of obtaining single-crystal layers at temperatures up to 1400 °C. This is due to the formation of superstrong carbon clusters during energetic ion implantation.

These results well confirm the conclusions of [7] about the features of the formation of SiC on the Si (100) surface and about the decisive role of the orientation of dilatation dipoles in the process of formation of the symmetry of the SiC film.

At the same time, the number of sharp maxima in the spectrum increased significantly up to 864.95 cm^{-1}. This may indicate the intense formation of nano- and microcrystals of various sizes [13,15].

If in Figure 2 we identify intense IR maxima from 794.74 to 864.95 cm^{-1} with the crystalline phase, then their integrated intensity is 251 a.u., or 50.2% of the volume of the SiC phase, i.e. share is increasing significantly. Thus, an intensive transformation of the nuclei of nanocrystals into micro- and nanocrystals is observed. The total number of nanocrystal nuclei and carbon-vacancy structures (spectrum components in the range 874.48-958.92 cm^{-1}) add up to 155 a.u., and the fraction is halved to 31.1% of the film volume. However, the high amplitude of the peaks of crystalline SiC suggests a possibility of higher content of the single-crystal component of silicon carbide.

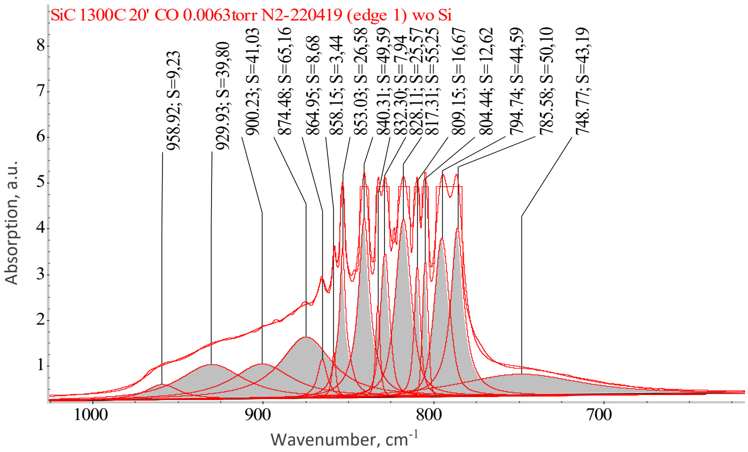

Figure 2 – Mathematical decomposition of the IR spectrum of the SiC film synthesized in an atmosphere of CO gas (1300 °C, 0.8 Pa, 20 min)
by the method of atom substitution

Studies on a Quanta 200i 3D scanning electron microscope showed that with a synthesis duration of 20 minutes, a temperature of 1300°C and a pressure of 0.8 Pa, a microcrystalline surface structure is formed (Figure 3) with grain sizes of 3-20 µm.

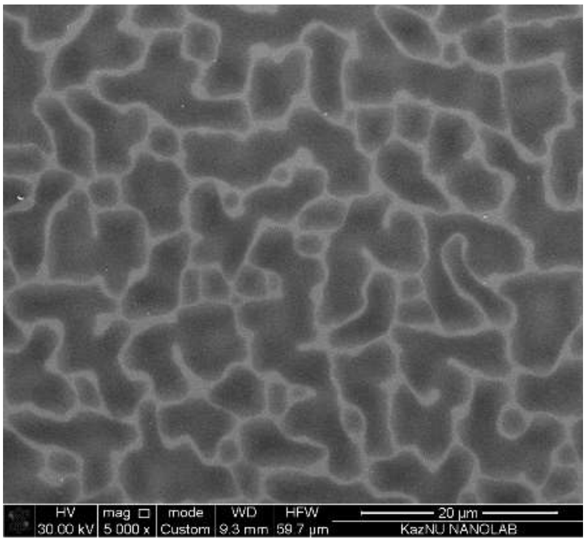

Figure 3 – Scanning electron microscopy (1300 °C, 0.8 Pa, 20 min) of the surface of the SiC film synthesized in a CO gas atmosphere by atom substitution method

Conclusions

The crystalline silicon carbide films containing mono- and microcrystalline phases of SiC were synthesized by the method of coordinated substitution of atoms in a special electric furnace at temperatures of 1200 °C and 1300 °C for 20 minutes in a CO gas flow at a pressure of 0.8 Pa in the near-surface region of a Si(100) wafer.

It was found that an increase in temperature from 1200 °C to 1300 °C led to a more intensive formation of silicon carbide and an increase in the number of Si–C bonds by 1.9 times due to an increase in the thickness of the synthesized silicon carbide layer. The proportion of the SiC crystalline phase increases to 50.2% of the film volume due to the intensive transformation of nanocrystal nuclei into micro- and nanocrystals. However, the high amplitude of the peaks of crystalline SiC suggests a possibility of higher content of the single-crystal component of silicon carbide.

Acknowledgments

This research has been funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AP09058438).

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Information on the authors

Nusupov Kair Khamzaevich (corresponding author)
Doctor of Physical and Mathematical Sciences, Chief Researcher, Laboratory of Alternative Energy and Nanotechnologies, JSC "Kazakhstan-British Technical University", st. Tole bi 59, 050000, Almaty, Kazakhstan
ORCID ID 0000-0001-8200-7510
E-mail: rich-famouskair@mail.ru

Beisenkhanov Nurzhan Beisenkhanovich (corresponding author)
Doctor of Physics and Mathematics, Dean, Chief Researcher, School of Materials Science and Green Technologies, KBTU JSC, st. Tole bi, 59, 050000, Almaty, Kazakhstan
ORCID ID 0000-0002-5908-5614
E-mail: beisen@mail.ru

Kukushkin Sergey Arsenievich
Doctor of Physical and Mathematical Sciences, Head of the Laboratory, Laboratory of Structural and Phase Transformations in Condensed Matter, Institute for Problems of Mechanical Engineering of the Russian Academy of Sciences, St. Petersburg, Vasilievsky Island, Bolshoi Prospekt, 61, Russia, 199178.
ORCID ID 0000-0002-2973-8645
E-mail: sergey.a.kukushkin@gmail.com

Sultanov Asanali Talgatbekuly
Ph.D. student, head of the laboratory, Laboratory of Alternative Energy and Nanotechnologies, KBTU JSC, 59 Tole bi str., 050000, Almaty, Kazakhstan
ORCID ID 0000-0003-0074-431X
E-mail: asanalisultanovs@gmail.com

Keyinbay Symayil
Master, Junior Researcher, Laboratory of Alternative Energy and Nanotechnologies, KBTU JSC, st. Tole bi, 59, 050000, Almaty, Kazakhstan
ORCID ID 0000-0002-7268-7193
E-mail: Keiinbay_symaiyl@mail.ru

Shynybaev Darkhan Serikovich
Bachelor, leading operator of the technological line, Laboratory of Alternative Energy and Nanotechnologies, KBTU JSC, st. Tole bi, 59, 050000, Almaty, Kazakhstan
ORCID ID 0009-0000-7729-7517
E-mail: darhan.86@mail.ru

Kusaynova Aizhan Zhambulovna
Master student, leading chemical technologist, Laboratory of alternative energy and nanotechnologies, KBTU JSC, st. Tole bi, 59, 050000, Almaty, Kazakhstan
ORCID ID 0000-0002-2485-9739
E-mail: a.kusainova@kbtu.kz

Авторлар туралы мәліметтер

Нусупов Каир Хамзаевич
Ф-м.ғ.д., бас ғылыми қызметкер Баламалы энергетика және нанотехнологиялар зертханасы, «Қазақ-Британ техникалық университеті» АҚ, АҚ «КБТУ», Төле би, 59, 050000, Алматы қ., Қазақстан
ORCID ID 0000-0001-8200-7510
E-mail: rich-famouskair@mail.ru

Бейсенханов Нуржан Бейсенханович (корреспонденция авторы)
Ф-м.ғ.д., декан, бас ғылыми қызметкер Материалтану және жасыл технологиялар мектебі, АҚ «ҚБТУ», Төле би 59, 050000, Алматы қ., Қазақстан
ORCID ID 0000-0002-5908-5614
E-mail: beisen@mail.ru

Кукушкин Сергей Арсеньевич
Ф-м.ғ.д., зертхана меңгерушісі, Конденсацияланған заттардағы құрылымдық және фазалық өзгерістер зертханасы РҒА Машина жасау мәселелері институты, Санкт-Петербург қ., ВА, Үлкен даңғылы, 61, Ресей, 199178
ORCID ID 0000-0002-2973-8645
E-mail: sergey.a.kukushkin@gmail.com

Султанов Асанали Талгатбекулы
Ph.D. студенті, зертхана меңгерушісі Баламалы энергетика және нанотехнологиялар зертханасы, АҚ «ҚБТУ», Төле би 59, 050000, Алматы қ., Қазақстан
ORCID ID 0000-0003-0074-431X
E-mail: asanalisultanovs@gmail.com

Кейінбай Сымайыл
Магистр, кіші ғылыми қызметкер Баламалы энергетика және нанотехнологиялар зертханасы, АҚ «ҚБТУ», Төле би 59, 050000, Алматы қ., Қазақстан
ORCID ID 0000-0002-7268-7193
E-mail: keiinbay_symaiyl@mail.ru

Шыныбаев Дархан Серикович
Бакалавр,технологиялық желінің жетекші операторы, Баламалы энергетика және нанотехнологиялар зертханасы, АҚ «ҚБТУ», Төле би 59, 050000, Алматы қ., Қазақстан
ORCID ID 0009-0000-7729-7517
E-mail: darhan.86@mail.ru

Кусайнова Айжан Жамбуловна
Магистрант, жетекші химик-технолог, Баламалы энергетика және нанотехнологиялар зертханасы, АҚ «ҚБТУ», Төле би 59, 050000, Алматы қ., Қазақстан
ORCID ID 0000-0002-2485-9739
E-mail: a.kusainova@kbtu.kz

Информация об авторах

Нусупов Каир Хамзаевич
Д.ф-м.н., главный научный сотрудник, Лаборатория альтернативной энергетики и нанотехнологий, АО «Казахстанско-Британский технический университет», ул. Толе би, 59, 050000, г. Алматы, Казахстан
ORCID ID 0000-0001-8200-7510
E-mail: rich-famouskair@mail.ru

Бейсенханов Нуржан Бейсенханович (автор для корреспонденции)
Д.ф-м.н., декан, главный научный сотрудник, Школа материаловедения и зеленых технологий, АО «КБТУ», ул. Толе би, 59, 050000, г. Алматы, Казахстан
ORCID ID 0000-0002-5908-5614
E-mail: beisen@mail.ru

Кукушкин Сергей Арсеньевич

Д.ф-м.н., заведующий лабораторией, Лаборатория структурных и фазовых превращений в конденсированных средах, Институт проблем машиноведения РАН, г. Санкт-Петербург, Васильевский остров, Большой проспект, 61, Россия, 199178.

ORCID ID 0000-0002-2973-8645

E-mail: sergey.a.kukushkin@gmail.com

Султанов Асанали Талгатбекулы

Ph.D. студент, заведующий лабораторией, Лаборатория альтернативной энергетики и нанотехнологий, АО «КБТУ», ул.Толе би, 59, 050000, г. Алматы, Казахстан

ORCID ID 0000-0003-0074-431X

E-mail: asanalisultanovs@gmail.com

Кейінбай Сымайыл

Магистр, младший научный сотрудник, Лаборатория альтернативной энергетики и нанотехнологий, АО «КБТУ», ул. Толе би, 59, 050000, г. Алматы, Казахстан

ORCID ID 0000-0002-7268-7193

E-mail: Keiinbay_symaiyl@mail.ru

Шыныбаев Дархан Серикович

Бакалавр, ведущий оператор технологической линии, Лаборатория альтернативной энергетики и нанотехнологий, АО «КБТУ», ул. Толе би, 59, 050000, г. Алматы, Казахстан

ORCID ID 0009-0000-7729-7517

E-mail: darhan.86@mail.ru

Кусайнова Айжан Жамбуловна

Магистрант, ведущий химик-технолог, Лаборатория альтернативной энергетики и нанотехнологий, АО «КБТУ», ул. Толе би, 59, 050000, г. Алматы, Казахстан

ORCID ID 0000-0002-2485-9739

E-mail: a.kusainova@kbtu.kz