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K.S. Tolubayev¹, B.A. Zhautikov¹, N.N. Zobnin¹, G.S. Dairbekova², S.K. Kabiyeva¹,

¹Karaganda Industrial University, Temirtau, Kazakhstan

E-mail: kanat.tolubayev@ttu.edu.kz

E-mail: bakhyt_zhautikov@mail.ru*

E-mail: zobninmn@mail.ru

E-mail: kabieva.s@mail.ru

²Satpayev University, Almaty, Kazakhstan;

E-mail: guldana.01.02.91@mail.ru

IDENTIFICATION AND QUANTIFICATION OF SILICON POLYMORPHISM IN MAGNETRON SPUTTERED THIN FILMS

ЖҰҚА МАГНЕТРОНДЫ БҮРІККІШ ПЛЕНКАЛАРДАҒЫ КРЕМНИЙ ПОЛИМОРФИЗМІН ИДЕНТИФИКАЦИЯЛАУ ЖӘНЕ САНДЫҚ АНЫҚТАУ ИДЕНТИФИКАЦИЯ И КОЛИЧЕСТВЕННАЯ ОЦЕНКА ПОЛИМОРФИЗМА КРЕМНИЯ В ТОНКИХ ПЛЁНКАХ МАГНЕТРОННОГО НАПЫЛЕНИЯ

Abstract. The article raises the problem of disagreements between different approaches to studying silicon polymorphism in silicon nanofilms using Raman spectroscopy. Here, the polymorphism assessment was carried out using a Horiba brand device Jobin – Yvon HR 800 UV (France). An argon-cadmium laser with a wavelength of 315 nm was used as the excitation source. The measurements were carried out in the range from 0 to 3200 cm^{-1} . Two different types of film were studied. Parallel measurements of Raman spectra revealed the homogeneity of the polymorphic composition of films of both types, which indicates the advantage of magnetron sputtering technology over the CVD method in this part. The analysis revealed: a split peak with a maximum in the region of 120 cm^{-1} interpreted as a manifestation of amorphous silicon, a peak in the region of 210 cm^{-1} attributed to the crystalline form of silicon. Peaks were found in the region of 408 and 520 cm^{-1} , which can be assessed as signs of nanosilicon. Also, in the spectrogram there are peaks in the range of 600-660 cm^{-1} (presumably copper silicide, an intermediate layer between the substrate and the film), as well as peaks at 900-1000 cm^{-1} and 1500-1660 cm^{-1} , identified as manifestations of copper. It was found that in the case of silicon film with an amorphous and crystalline structure, a laser wavelength of 315 nm is sufficient for the beam to penetrate the substrate and intermediate layers and record their spectrum. In this case, the copper of the substrate is identified by a peak in the region of 1500-1660 cm^{-1} . The nanocrystalline structure is less permeable and the identification peak of copper in this case is in the region of 900-1000 cm^{-1} . To the greatest extent, the results obtained correlate with the results obtained by Levitsky V.S. It is recommended to use his approaches to the identification of silicon films, as they most adequately describe the relationship between the results of Raman spectrometry and the polymorphism of silicon films.

Keywords: SoG - Silicon, magnetron sputtering, Caroline D12C, nano silicon, amorphous silicon, crystalline silicon, film, Raman spectrum, polymorphism.

Аңдатпа. Мақалада Раман спектроскопиясы арқылы кремний нано пленкаларындағы кремний полиморфизмін зерттеудің әртүрлі тәсілдерінің келіспеушілігі мәселесі көтеріледі. Мұнда полиморфизмді бағалау Horiba Jobin–Yvon hr800uv (Франция) маркалы құрылғының көмегімен жүргізілді. 315 НМ толқын ұзындығы бар аргон-кадмий лазері қозу көзі ретінде пайдаланылды.

Өлшеу 0-ден 3200 см⁻¹ аралығында жүргізілді. Қабықшаның екі түрі зерттелді. Раман спектрлерін параллель өлшеу барысында екі типтегі пленкалардың полиморфты құрамының біртектілігін анықтады, бұл магнетронды бүрку технологиясының осы бөліктегі CVD әдісінен артықшылығы туралы айтады. Талдау кезінде мыналар анықталды: аморфты кремнийдің көрінісі ретінде түсіндірілген максимумы 120 см⁻¹ болатын бөлінген шың, кремнийдің кристалды түріне жатқызылған 210 см⁻¹ шыңы. 408 және 520 см⁻¹ аймағында нано кремнийдің белгілері ретінде бағалауға болатын шыңдар табылды. Сондай-ақ, спектрограммада 600-660 см⁻¹ (мыс силицидi, субстрат пен пленка арасындағы аралық қабат), сондай-ақ мыстың көрінісі ретінде анықталған 900-1000 см⁻¹ және 1500-1660 см⁻¹ шыңдары бар. Аморфты және кристалды құрылымы бар кремний пленкасы жағдайында 315 нм лазердің толқын ұзындығы сәуленің субстратқа және аралық қабаттарға еніп, олардың спектрін тіркеуі үшін жеткілікті екендігі анықталды. Бұл жағдайда субстраттың мысын 1500-1660 см⁻¹ шыңы анықтайды. Нано кристалды құрылымы аз өткізгіш және мыстың идентификациялық шыңы бұл жағдайда 900-1000 см⁻¹ аймағында. Алынған нәтижелер Левицкий В.С. – мен ең көп корреляцияланады. Раман спектрометриясы мен кремний пленкаларының полиморфизмі нәтижелерінің байланысын барынша барабар сипаттайтын кремний пленкаларын анықтау тәсілдерін қолдану ұсынылады.

Түйін сөздер: SoG-Silicon, магнетронды бүрку, Caroline D12C, нано кремний, аморфты кремний, кристалды кремний, пленка, раман спектрі, полиморфизм.

Аннотация. В статье поднимается проблема разногласий различных подходов к изучению полиморфизма кремния в кремниевых нано плёнках методом рамановской спектроскопии. Здесь оценка полиморфизма проводилась с помощью прибора марки Horiba Jobin–Yvon HR800UV (Франция). В качестве источника возбуждения использовался аргон-кадмиевый лазер с длиной волны 315 нм. Измерения проводились в диапазоне от 0 до 3200 см⁻¹. Изучены два различных типа плёнки. Параллельные замеры спектров Рамана выявили однородность полиморфного состава плёнок обоих типов, что говорит о преимуществе технологии магнетронного напыления перед методом CVD в этой части. При анализе установлены: расщепленный пик с максимумом в районе 120 см⁻¹, интерпретированный как проявление аморфного кремния, пик в районе 210 см⁻¹, отнесенный к кристаллической форме кремния. В районе 408 и 520 см⁻¹ найдены пики, которые можно оценивать как признаки нано кремния. Также на спектрограмме имеются пики в диапазоне 600-660 см⁻¹ (предположительно силицид меди, промежуточный слой между подложкой и плёнкой), а также пики 900-1000 см⁻¹ и 1500-1660 см⁻¹, идентифицированные как проявления меди. Установлено, что в случае кремниевой плёнки с аморфной и кристаллической структурой длины волны лазера 315 нм достаточно, чтобы луч проникнул к подложке и промежуточным слоям и зарегистрировал их спектр. При этом медь подложки идентифицируется пиком в районе 1500-1660 см⁻¹. Нано кристаллическая структура менее проницаема и идентификационный пик меди в этом случае в районе 900-1000 см⁻¹. В наибольшей степени полученные результаты коррелируют с результатами, полученными Левицким В.С. Рекомендуется использовать его подходы к идентификации кремниевых плёнок, как наиболее адекватно описывающие связь результатов рамановской спектрометрии и полиморфизма кремниевых плёнок.

Ключевые слова: SoG-Silicon, магнетронное напыление, Caroline D12C, нано кремний, аморфный кремний, кристаллический кремний, плёнка, рамановский спектр, полиморфизм.

Introduction. Silicon, in its physicochemical properties, is the closest analogue of carbon, and has been of interest as a material for micro- and optoelectronics for more than 50 years (Daliev K.S., 2005; Abdurakhmanov K.P., 1998). The most promising direction of silicon technologies is the growth of thin films obtained by deposition of amorphous, crystalline and nanocrystalline silicon on substrates. Silicon-based thin films are fabricated using chemical vapor deposition (CVD), magnetron sputtering and vacuum evaporation (Catchpole K.R.; Yoshihiro H.; Bergmann R.B., 2002).

Diagnostics of silicon films is an important element in the technological chain of production of high-quality solar cells based on amorphous hydrogenated silicon (a-Si:H) and microcrystalline silicon (μc-Si). Raman spectroscopy is an effective local non-destructive method for structural analysis. Using this technique, it is possible to determine the size of crystallites, changes in the short- and medium-range orders of the structure, the presence of stresses in the structure, as well as the ratio of the shares of crystalline and amorphous

components in thin films containing nanocrystalline inclusions (Levitsky V.S., 2001). However, the interpretation of Raman spectra of thin silicon films still does not have a unified approach. Different researchers use equipment with different wavelengths and laser powers, samples prepared in different ways, and different Raman shift limits in spectroscopy. Therefore, the results of studying these objects differ significantly.

In the work of Geisler S.V. et al. a study of thin silicon films obtained by gas-jet electron-beam plasma-chemical gas-phase deposition was conducted (GJEB P.E. CVD). Equipment Triplemate SPEX was used for Raman spectroscopy of films based on an argon laser with a wavelength of 488 nm and a power of 5 mW. According to the authors, this laser wavelength was chosen in order to reduce the beam penetration depth and prevent recording of the substrate spectrum. The power was chosen to avoid crystallization (the polymorphic transformation of amorphous and nanocrystalline silicon into crystalline silicon) under the action of a laser beam (Gaisler S. V., 2004). In this case, the spectra presented in Figure 1 were obtained.

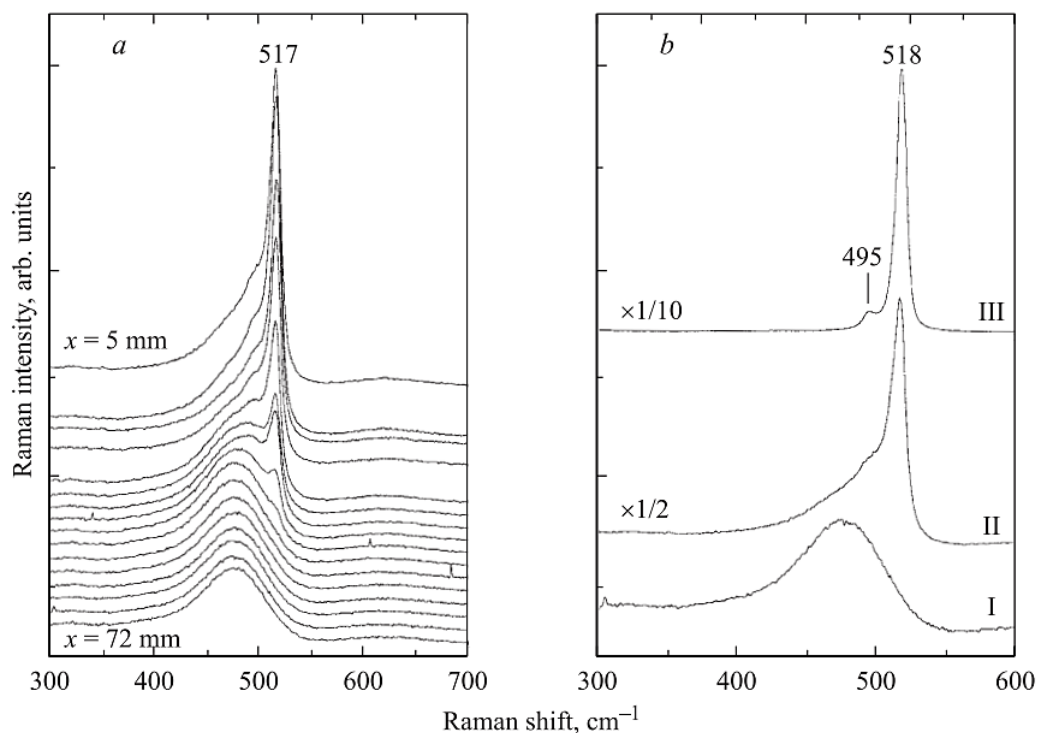


Figure 1. Raman spectra obtained at different points of the silicon film

Note – compiled by the author based on (Gaisler, 2004)

The authors identified the resulting spectrogram as follows. The peak with a maximum at 475 cm^{-1} indicates the presence of an amorphous state of silicon. The peak with a maximum in the region of $514\text{--}518\text{ cm}^{-1}$ indicates the presence of nanoparticles. The authors also note a peak at 495 cm^{-1} , which they could not identify (Gaisler S. V., 2004). Given these assumptions, it should be noted that the method GJEB PE CVD results in a film that is uneven in terms of silicon polymorphism. One sample contains both areas consisting entirely of nanosilicon and areas containing only amorphous silicon. Such heterogeneity will lead to instability of the technological properties of the film and can also lead to an erroneous assessment of the film morphology. At the same time, there is no mention of the possibility of the presence of areas with crystalline silicon, although it is not at all excluded. The Raman shift range is only from

300 to 700 cm^{-1} , which may not provide enough data to evaluate film polymorphism. Finally, the authors did not present electron microscopy results in the paper to support the analysis of the Raman spectroscopy data. Although there is an indication here that nanocrystalline silicon has a spherical shape with a diameter of 3-10 nm. Data from other sources regarding sizes of 3.5-20 nm are also provided (Kamei T., 1999).

Similar studies were carried out by Utemuradova Sh. B. et al. In their work, they used Raman spectroscopy to study crystalline n-type silicon grown using the Czochralski method, i.e. a material that obviously does not contain either amorphous or nanocrystalline silicon. Here the authors provide photographs of crystalline silicon obtained using a scanning electron microscope. The Raman spectrum and micrograph are shown in Figure 2 (Utemuradova S.B., 2002).

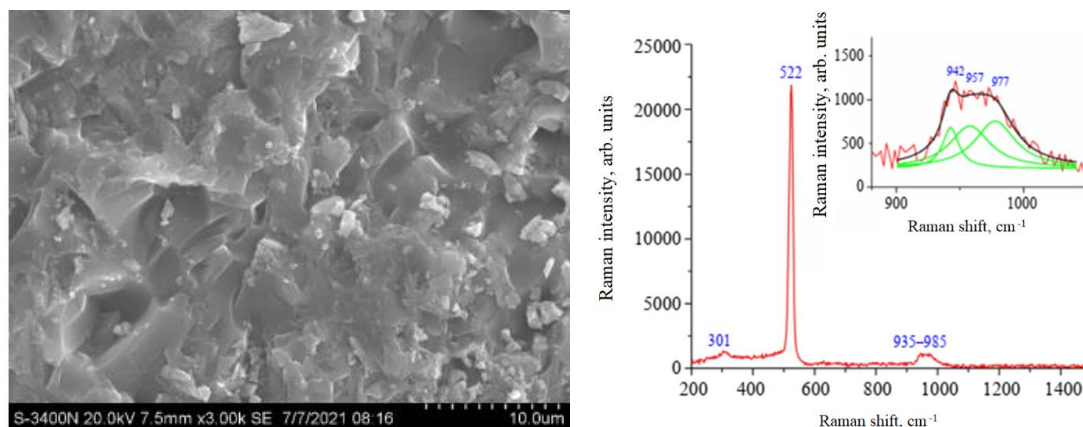


Figure 2. Raman spectrum and micrograph of a sample of single-crystalline silicon grown using the Czochralski method

Note – compiled by the author based on (Utemuradova, 2002)

Raman spectroscopy was carried out on a CARS Raman instrument Spectrometer. No further details are provided. The microphotograph data in Figure 2 contradicts the data of Geisler S.V., because in the presence of a dominant peak at 522 cm^{-1} , the micrograph does not show spherical silicon particles with a size of 3-10 nm, which are mentioned in the article by Geisler S.V. Meanwhile, in her work, Utemuradova Sh. B. indicates the presence of nanoparticles in the sample, although it is obvious from the micrograph that there are no nano-sized particles in the structure of the sample. Moreover, the magnification $\times 3000$ (3.00 k) indicated in the photo cannot reveal nano-sized objects.

It is also indicated that the Raman spectrum has peaks at 301 and 935-985 cm^{-1} . The presence of these peaks is explained as follows. As a result of the long-range translational symmetry of crystalline Si, additional peaks in the range of 1000–1100 cm^{-1} may appear in the Raman spectra, which are much less intense than the first-order peak (Temple P. A., 1973). The vibration at 301 cm^{-1} in many works is attributed to the LA (longitudinal acoustic) mode (Wellner A., 2004; Zhigunov D. M., 2018; Raczykowski B., 2017). It is assumed that this peak is due to the overtones TA (2TA(X)) at the critical point X as a result of the occurrence of a superposition of transverse and longitudinal acoustic modes. The intensity of the peak at 522 cm^{-1} is 17 times greater than the second-order peak at 301 cm^{-1} (Utemuradova S. B., 2002). The peak at 935–985 cm^{-1} (Utemuradova S. B., 2002) was identified by analogy with the works (Temple P. A., 1973; Raczykowski B., 2017; Sachat E., 2017) as a manifestation of nanocrystalline silicon, caused by the scattering of several transverse optical phonons (2TO) and

their overtone state. Thus, from the work of Utemuradova Sh. B. we can conclude that in the studied samples there is only nanocrystalline silicon and no crystalline or amorphous silicon.

A different interpretation of Raman spectra is given in his work by V.S. Levitsky. He gives his vision of the Raman spectra of amorphous (a-Si), crystalline (cryst. Si) and microcrystalline silicon (nc-Si) Figure 3.

Under microcrystalline silicon Levitsky V.S. understands a certain superposition of amorphous silicon and nano-sized inclusions of crystalline silicon. Moreover, he claims that the spectrum of microcrystalline silicon consists of a peak with a maximum near 480 cm^{-1} associated with the amorphous phase, and a much narrower peak near 520 cm^{-1} associated with the nanocrystalline phase (Levitsky V. S., 2001). At the same time, the author also does not provide photographs obtained as a result of electron microscopy, which would confirm these data.

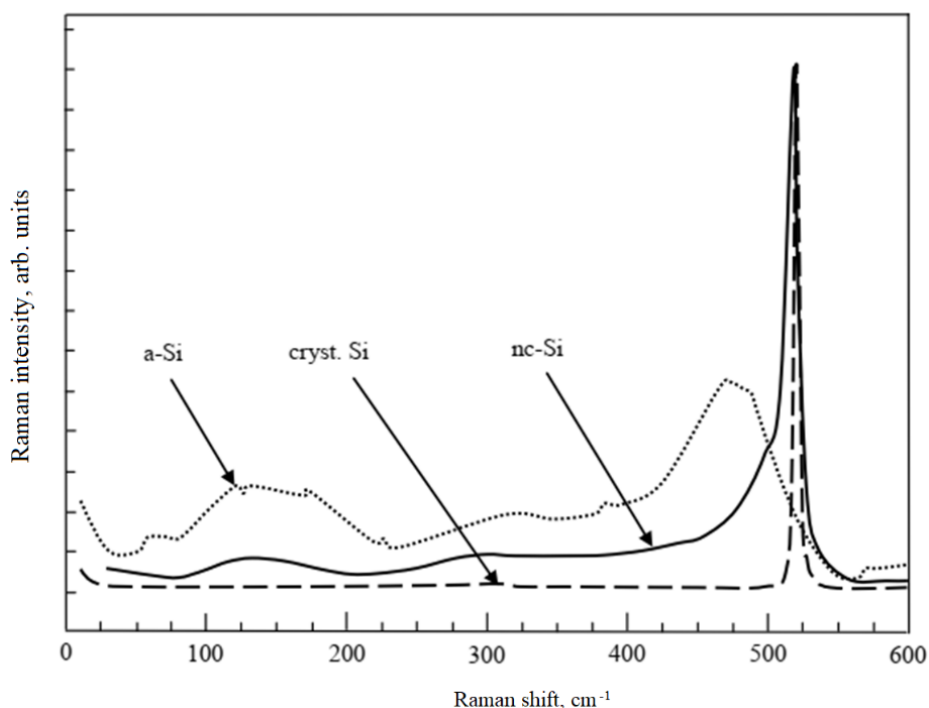


Figure 3. Raman spectra of amorphous, crystalline and microcrystalline silicon

Note – compiled by the author based on (Levitsky, 2001)

As can be seen when considering the existing attempts to identify the polymorphism of silicon structures, there are many internal and external contradictions in this issue that require resolution. The proposed solutions are not complete enough; additional research is needed to give a more accurate and reasonable interpretation of the Raman spectra of thin silicon films.

Materials and methods of research. Silicon nanofilms were obtained by magnetron sputtering using the Caroline D12C system. Silicon crystals grown by the Czochralski method using commercial high-purity silicon of SoG-Si grade 6-7N were used as targets for magnetron sputtering. The substrate was copper foil 0.5 mm thick. The thickness of the silicon film was 300-400 nm.

Raman spectroscopic analysis was carried out on a Horiba brand system Jobin – Yvon HR800UV (France). An argon-cadmium laser with a wavelength of 315 nm was used as the

excitation source. The laser power on the sample was ~25 mW for Ar / Cd . The holographic diffraction grating had 2200 lines/mm for a 315 nm laser and was focused onto a CCD detector. The lens was Olympus 412 UV for Ar / Cd. The measurements were carried out in the range from 0 to 3200 cm^{-1} . No filter was used to reduce the radiation entering the detector from the samples.

The essence of the method is that when a laser beam with a wavelength of 315 nm (wave number about 31000 cm^{-1}) passes through the silicon film under study, the wave energy is absorbed and, accordingly, the wavelength increases (the wave number decreases by about 2-5%). The detector estimates the energy of all scattered particles reflected from the film based on the wave number. The device data processing program performs deconvolution according to the method described in the literature (Kemmer G.,2010; Mysen B.,1982; Ferraro J. R., 2003), i.e. subtracts the wave numbers of scattered particles from the original wavenumber, estimates the statistical distribution of this difference, and calculates the fractional content of each polymorphic silicon phase in the film identified by the peak in the spectrogram, proportional to the area of the peak relative to the base line of the spectrogram. Baseline correction was previously performed on the original spectra before data normalization. Data normalization helps to compare small deviations in spectra. The formula for data normalization is described in the literature (Mysen B.,1982).

Results and Discussions. During the research, two types of silicon films were studied, obtained under different conditions of magnetron sputtering. For each type of film, several parallel measurements of Raman spectra were carried out at several different points along the entire surface of the film. This test showed the uniformity of the Raman spectra over the entire surface of each film, which indicates the advantage of magnetron sputtering technology over the CVD method in terms of ensuring film uniformity.

Figure 4 shows the Raman spectrogram of the first type of silicon film.

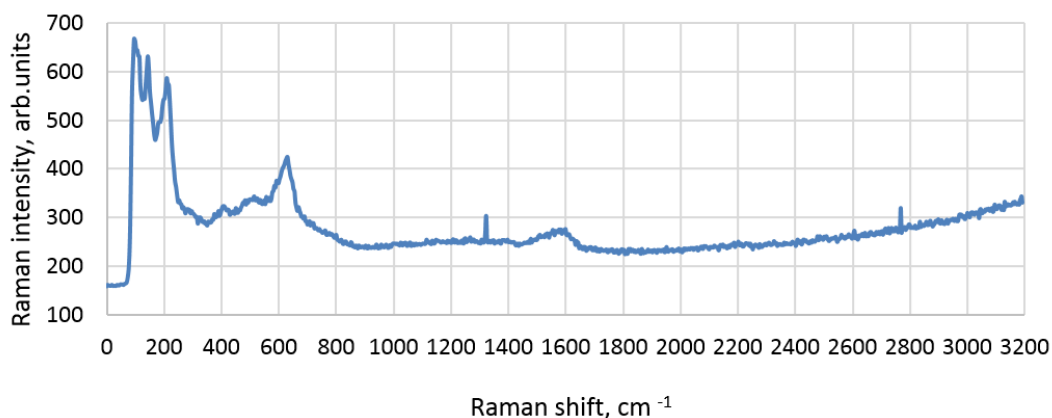


Figure 4. Raman spectrogram of silicon film of the first type

Note – compiled by the author

We will interpret the spectrum based on the data in Figure 3. We will consider the split peak with a maximum in the region of 120 cm^{-1} as a manifestation of amorphous silicon. The peak with a maximum around 210 cm^{-1} is interpreted as a crystalline form of silicon. In the region of 408 and 520 cm^{-1} there are minor peaks of the second order, which can be assessed as trace amounts of nanosilicon. The spectrogram also has peaks in the range 600-660 cm^{-1} and 1500-1660 cm^{-1} . According to the literature, the second peak corresponds to copper (Lopes C. S. D.,2018) and copper can also give a peak in the region of 900-1000 cm^{-1} , which casts doubt on

the reasoning given by Sh.B. Utemuradova regarding the belonging of this peak to crystalline silicon. This means that the laser wavelength of 315 nm was sufficient for the beam to penetrate the substrate and record its spectrum. It is possible that if the studies discussed here (Levitsky V. S., 2001; Gaisler S. V., 2004; Utemuradova S. B., 2009) had examined the Raman spectrum over a wider range, they might also have captured the substrate material. As for the peak at 600-660 cm^{-1} , it can be assumed that it refers to transitional copper silicides between the substrate and the silicon film, which could arise during the deposition of the first portions of silicon on the copper substrate. Data on the formation of thin films of copper silicides a-Si (50 nm)/ Cu (200 nm)/a-Si (50 nm) are available in the literature (Buchin E. Yu., 2019). However, it is not possible to compare the results, because the authors studied copper silicide films using diffractometry rather than Raman spectroscopy.

The software package integrated into the spectroscopy equipment provides automated deconvolution, but for additional verification, the spectrum obtained from the analysis of the first type of film was subjected to manual deconvolution. The normalized Raman spectrum curves for deconvolution are presented in Figure 5.

Integrating the fit function of the normalized Raman spectra in the peak range over the baseline allows one to find the area under the curve. We sum up the areas of two peaks (amorphous and crystalline phases) and take them as 100%. Accordingly, we calculate the share of each phase in proportion to the area under the peak of this phase. For amorphous silicon this would be 77.7% and for crystalline silicon 22.3%, which is close to the values obtained from automated deconvolution of 76.1 and 23.9%, respectively. As can be seen in the normalized spectrogram, the peak probably related to nanosilicon (520 cm^{-1}) was practically leveled to the noise level. In this regard, we do not take this component into account in the calculation and take the fraction of the nanosilicon phase equal to zero.

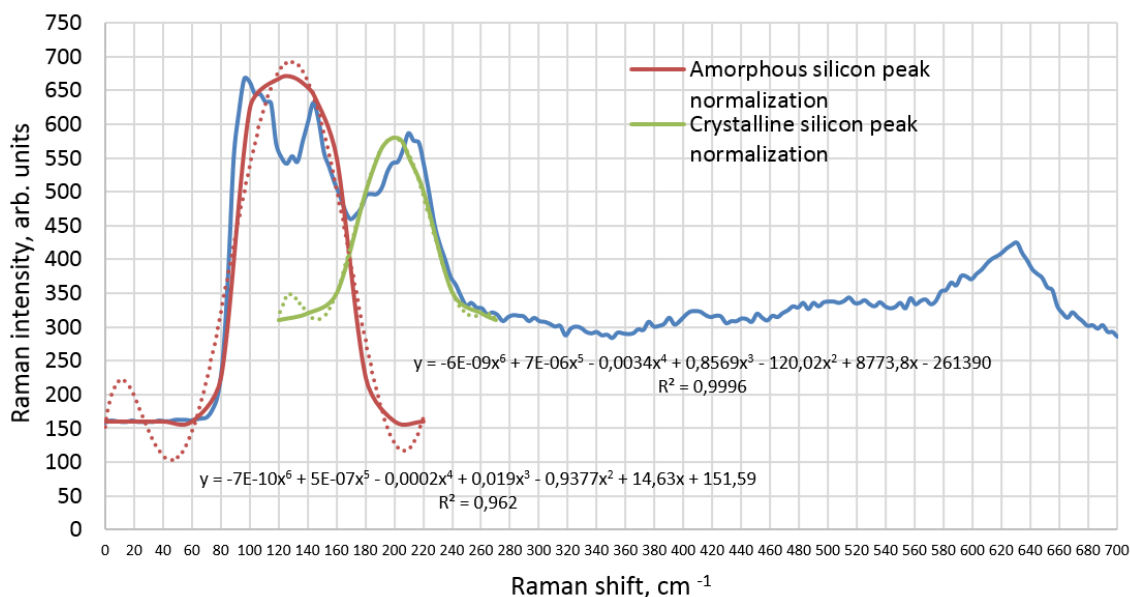


Figure 5. Normalized curves of the Raman spectrum, silicon films of the first type

Note – compiled by the author

Figure 6 shows the Raman spectrogram of the second type of silicon film. The height of the peaks of amorphous silicon in the region of 120 cm^{-1} , as well as of crystalline silicon in the region of 210 cm^{-1} , decreased sharply. At the same time, their splitting was preserved. The peak

at 408 cm^{-1} completely disappeared, and the nanosilicon peak with a maximum at 519 cm^{-1} increased significantly. However, at high altitudes, the peak width is relatively small. At the same time, the peak in the range of $600\text{-}660\text{ cm}^{-1}$ remained, but its height decreased. The peak corresponding to copper at $1500\text{-}1660\text{ cm}^{-1}$ disappeared completely, but was preserved to a limited extent in the region of $900\text{-}1000\text{ cm}^{-1}$.

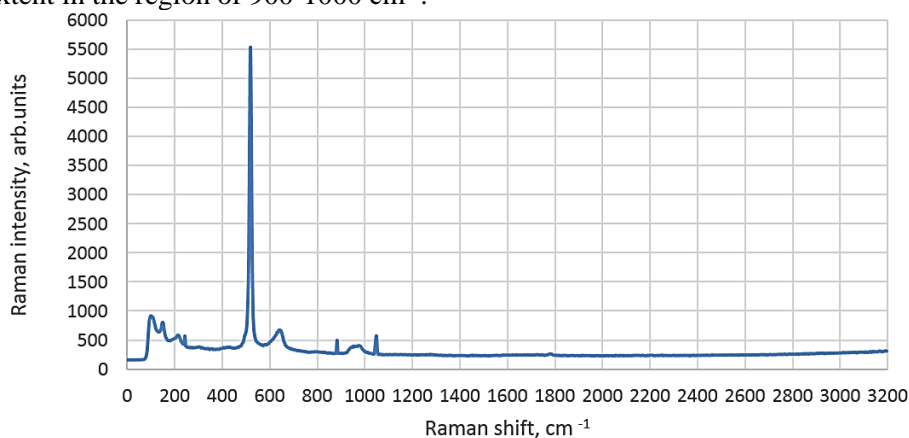


Figure 6. Raman spectrogram of a second type silicon film

Note – compiled by the author

Considering that the wavelength and laser power of Raman spectroscopy were the same when studying both films and the thickness of the films was the same, we can conclude that the nature of the film itself has fundamentally changed. The second type of film is less transparent to the laser beam and does not allow identification of the copper substrate, but still makes it possible to assess the presence of an intermediate layer of copper silicide located between the substrate and the silicon film.

Figures 7 (nano-silicon peak) and 8 (amorphous and crystalline silicon peaks) present the normalized Raman spectrum curves of the second type film used for deconvolution and determination of the ratio of various polymorphic states. The results of manual deconvolution calculations are confirmed by the data of automatic calculations. As can be seen from the calculation results, the film of the second type is represented mainly by the nanosilicon phase against the background of a reduction in the amorphous and, to a greater extent, crystalline phases.

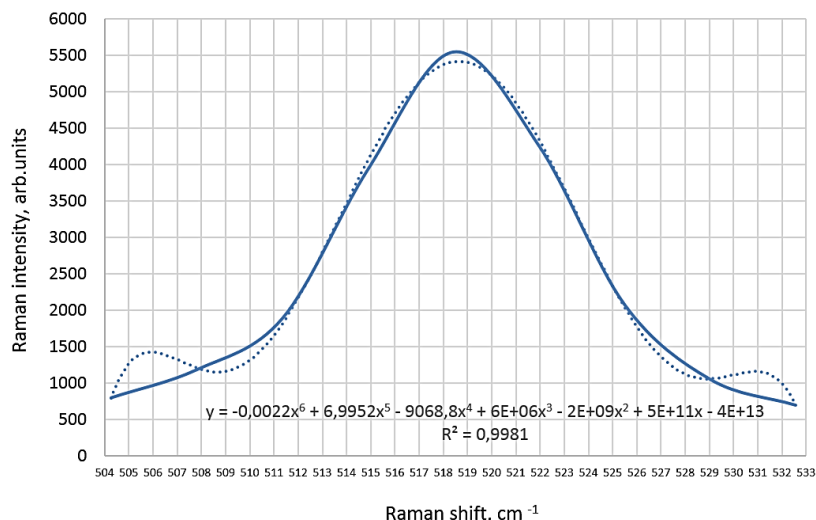


Figure 7. Normalized curve of the Raman spectrum (nano-silicon peak) of a second type silicon film

Note – compiled by the author

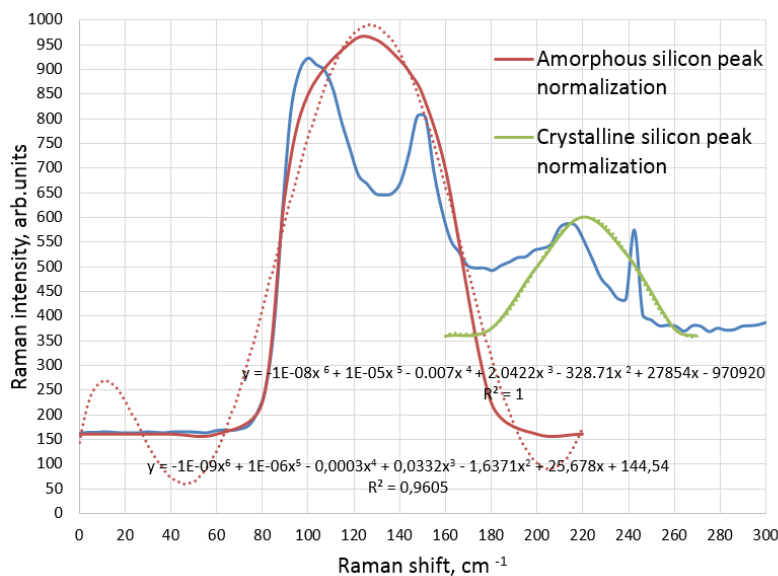


Figure 8. Normalized curves of the Raman spectrum, silicon films of the second type (peaks of amorphous and crystalline silicon)

Note – compiled by the author

Conclusions. Two different types of films were investigated. Parallel measurements of Raman spectra showed the homogeneity of the polymorphic composition of both types of films, which confirms the advantages of magnetron sputtering technology over the CVD method. The analysis revealed a split peak at about 120 cm^{-1} , interpreted as amorphous silicon, and a peak at about 210 cm^{-1} corresponding to crystalline silicon. Peaks around 408 and 520 cm^{-1} may indicate nanosilicon. The spectrogram also contains peaks in the range of $600\text{--}660\text{ cm}^{-1}$, presumably copper silicide, and peaks at $900\text{--}1000\text{ cm}^{-1}$ and $1500\text{--}1660\text{ cm}^{-1}$ identified as copper. For silicon films with an amorphous and crystalline structure, a laser wavelength of 315 nm is sufficient to

penetrate the substrate and record its spectrum, while copper is identified by peaks in the region of 1500-1660 cm^{-1} . The nanocrystalline structure is less transparent, and copper in this case is identified by peaks around 900-1000 cm^{-1} . The results obtained are most consistent with the data obtained by V.S. Levitsky. [6], whose methods for identifying Raman spectra of silicon films are recommended to be used as the most accurate in comparison with other studies (Gaisler S. V., 2004; Kamei T., 1999; Utemuradova S. B., 2022) describing the relationship between Raman spectroscopy and polymorphism of silicon films.

Conflict of interest. The authors declare that there is no conflict of interest.

Notification of the use of generative AI and technologies using it in the process of writing a manuscript. In preparing this work, the authors did not use generative AI and technologies based on its use in the process of writing the manuscript.

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Information about the authors

Tolubayev Kanat – Doctoral student, Karaganda Industrial University, Temirtau, Kazakhstan. E-mail: kanat.tolubayev@ttu.edu.kz, ORCID: 0009-0007-0868-7088

Zhautikov Bakhyt – Doctor of Technical Sciences, Professor, Karaganda Industrial University, Temirtau, Kazakhstan. E-mail: bakhyt_zhautikov@mail.ru, ORCID: 0000-0002-0908-4533

Zobnin Nikolai – Candidate of Technical Sciences, Karaganda Industrial University, Temirtau, E-mail: zobninnn@mail.ru, ORCID: 0000-0002-8556-2971

Dairbekova Guldana – PhD, Kazakh National Research Technical University, Almaty, Kazakhstan. E-mail: guldana.01.02.91@mail.ru, ORCID: 0000-0002-1528-0637

Kabiyeva Saule – Candidate of Chemical Sciences, Associate Professor, Karaganda Industrial University, Temirtau, Kazakhstan. E-mail: kabieva.s@mail.ru, ORCID: 0000-0002-4868-5278
