O‘ZBEKISTON MILLIY
UNIVERSITETI
XABARLARI, 2025, [3/2]
ISSN 2181-7324

FIZIKA
http://journals.nuu.uz
Natural sciences

UDK: 539.216.2:621.793.1

Sardor ESHBOBOEV,
Researcher of Karshi State University
E-mail:sardoreshboboyev919602944@gmail.com
Yokub ERGASHOV,
Professor of National University of Uzbekistan, DSc
Allanazar TASHATOV,
Professor of Karshi State University, DSc

Rewiew Associate professor N.Norkulov

SURFACE MORPHOLOGY AND STRUCTURAL FEATURES OF NICKEL SILICIDE THIN FILMS FABRICATED BY ION-PLASMA DEPOSITION

Annotation

It has been determined that excessive heating at 800 K may induce agglomeration effects; therefore, the optimal annealing temperature was set at 750 K. Furthermore, the initially amorphous or fine-grained crystalline structure evolved into larger crystalline domains, suggesting the onset of $NiSi_2$ phase formation. Energy-dispersive X-ray spectroscopy (EDX) revealed an almost complete absence of oxygen, carbon, and other impurity peaks, thereby confirming the high purity of the sample. The results indicate that silicide phases, most likely $NiSi_2$, were formed at the Ni/Si interface as a consequence of ion-plasma deposition followed by thermal diffusion.

Keywords: metal, silicide, thin films, nanoscale, contact resistance, ion-plasma deposition, thermal diffusion, energy-dispersive X-ray spectroscopy.

МОРФОЛОГИЯ ПОВЕРХНОСТИ И СТРУКТУРНЫЕ ОСОБЕННОСТИ ТОНКИХ ПЛЕНОК НИКЕЛЬСИЛИЦИДА, ПОЛУЧЕННЫХ МЕТОДОМ ИОННО-ПЛАЗМЕННОГО ОСАЖДЕНИЯ

Аннотация

Установлено, что чрезмерный нагрев при 800 K может вызвать эффекты агломерации; поэтому оптимальная температура отжига была установлена на уровне 750 K. Кроме того, первоначальная аморфная или мелкозернистая кристаллическая структура изменилась, превратившись в более крупные кристаллические домены, что свидетельствует о начале формирования фазы $NiSi_2$. Энергодисперсионная рентгеновская спектроскопия (EDX) показала практически полное отсутствие кислорода, углерода и других примесных пиков, что подтверждает высокую чистоту образца. Результаты исследования указывают на то, что на границе Ni/Si образовались силицидные фазы, наиболее вероятно $NiSi_2$, в результате ионно-плазменного осаждения с последующей термической диффузией.

Ключевые слова: металл, силицид, тонкие плёнки, наноразмерные структуры, контактное сопротивление, ионно-плазменное осаждение, термическая диффузия, энергодисперсионная рентгеновская спектроскопия.

ION-PLAZMA CHANGLATISH USULI ORQALI OLINADIGAN NIKEL SILITSID YUPQA QATLAMLARINING SIRT MORFOLOGIYASI VA TUZILMA XUSUSIYATLARI

Annotatsiya

Aniqlanishicha, 800 K da ortiqcha qizdirish aglomeratsiya jarayonlariga olib kelishi mumkin; shu sababli optimal tovlanish harorati 750 K etib belgilandi. Bundan tashqari, dastlabki amorf yoki mayda donali kristall tuzilma kattaroq kristall domenlarga o‘zgarib, $NiSi_2$ fazasi hosil bo‘lish jarayonining boshlanishini ko‘rsatdi. Energiya-dispersion rentgen spektroskopiyasi (EDX) namunada kislorod, uglerod va boshqa qo‘shimcha cho‘qqilarning deyarli yo‘qligini aniqladi, bu esa namunadagi yuqori tozalik darajasini tasdiqlaydi. Tadqiqot natijalari shuni ko‘rsatadiki, Ni/Si chegarasida ion-plazma changlatish va keyingi issiqlik diffuziyasi natijasida, ehtimol, $NiSi_2$ bo‘lgan silitsid fazalari hosil bo‘lgan.

Kalit so‘zlar: metall, silitsid, yupqa qavatlar, nanoo‘lcham, kontakt qarshiligi, ion-plazma changlatish, issiqlik diffuziyasi, energiya-dispersion rentgen spektroskopiyasi.

Introduction. Nickel silicide thin films and their metallic counterparts have attracted significant interest in nanoelectronics as submicron-level materials. Nickel silicides are widely utilized as contacts for field-effect transistors, in solar-thermal energy converters, as ohmic contacts, as protective coatings against electromagnetic radiation, and as one of the key electronic materials for various nanoelectronic devices [1–5].

Due to their advantages, such as low-temperature processing, low silicon consumption, and low contact resistance [6], nickel silicide thin films have gained increasing attention as promising candidates for next-generation nanoscale materials capable of replacing various types of silicide thin films currently in use.

The formation of nickel silicide thin films primarily occurs through a solid-phase reaction between the deposited nickel layer and the silicon substrate. Nickel is deposited onto a pretreated silicon substrate via physical vapor deposition (PVD) [7–9] or chemical vapor deposition (CVD) techniques. The phase formation of nickel silicides strongly depends on several factors, including the thickness of the Ni layer, the deposition technique, the orientation of the substrate, annealing conditions, and the morphology of the silicon surface. Therefore, controlling these parameters is critically important but remains challenging when aiming to obtain a single-phase NiSi structure [10].

The temperature-dependent silicidation reaction of Ni, which is governed by diffusion mechanisms, mainly proceeds through the diffusion of Ni atoms into the silicon region. In this process, the resulting Ni vacancies are predominantly localized within the nickel layer rather than in the silicon layer [11].

Furthermore, during thermal annealing, the sequential formation of nickel silicide phases Ni_2Si, NiSi, and $NiSi_2$ occurs [12]. These silicides differ in their formation temperatures and specific resistivity, which significantly affect the electrical properties of the surface [13]. The Ni_2Si phase is typically formed within the temperature range of 475 K to 600 K [14]. According to [15], the transition from Ni_2Si to NiSi takes place at 575–650 K, while the $NiSi_2$ phase forms at higher temperatures of approximately 700–750 K [16,17].

In this work, post-deposition annealing regimes were employed to control the diffusion and redistribution of low-energy Ni atoms on the Si(111) surface during the solid-phase ion-plasma deposition process and to improve the interface quality of nickel silicide layers. This study provides an experimental foundation for optimizing the structural and electronic properties of metallic silicides in integrated circuits, aiming to enhance the overall performance and efficiency of nanoelectronic devices [18].

Methods and discussion of results. Silicon substrates with a (111) crystallographic orientation were used in this study. The substrates were cleaned in three successive stages to ensure high surface quality. Nickel with a purity of 99.95% was deposited onto the heated Si(111) surface by the ion-plasma deposition method using a radio-frequency (RF) magnetron sputtering system under the following conditions: I = 296 mA, U = 337 V, and P = 100 W.

Initially, an amorphous Ni/Si layer was formed, which subsequently transformed into crystalline structures with face-centered cubic (FCC) and body-centered cubic (BCC) lattice configurations. These crystalline structures define the spatial arrangement of atoms within the metallic layer.

Upon annealing at a temperature range of 700–750 K for 150 minutes, significant grain growth and crystallization were observed, indicating improved structural ordering within the nickel silicide films.

Figure 1 shows a scanning electron microscopy (SEM) image of the Ni/Si thin film obtained by the ion-plasma deposition method. The image reveals that the top layer (lighter contrast) has an approximate thickness of 0.39 µm, as determined from the scale bar. A well-defined and smooth interface is observed between the thin film and the substrate (darker lower region), indicating strong adhesion and minimal interdiffusion.

The top layer exhibits a polycrystalline structure, where grains of various sizes and their boundaries are clearly visible. The surface of the film is relatively uniform, although minor variations are noticeable in certain areas. Due to the use of a backscattered electron (BSE) detector, a compositional contrast is observed: regions containing heavier atoms appear brighter, whereas areas with lighter atoms appear darker [19].

At a magnification of 44,600×, the microstructural details of the film are clearly resolved. The results indicate that the crystallization process is essentially complete, with minimal agglomeration and preserved film integrity. These findings are critical for evaluating the film quality, predicting its electrical properties, and optimizing the technological deposition process.

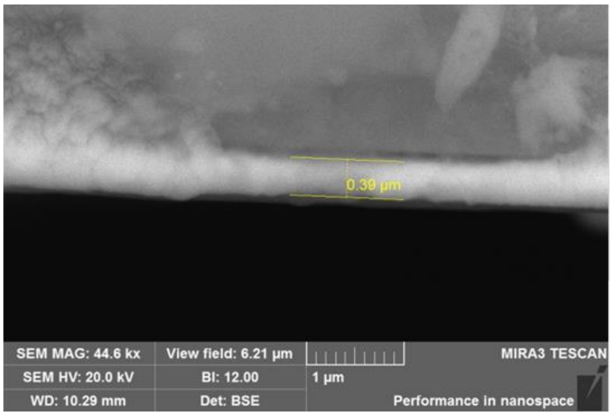

Figure 1. Scanning electron microscopy (SEM) image of the Ni/Si thin film with an approximate thickness of 390 nm, obtained by the ion-plasma deposition method.

At a temperature of 300 K, a nickel thin film with a thickness of 390 nm was deposited onto the silicon surface using the ion-plasma deposition method. At this stage, small surface spots were observed on the as-deposited film. The samples were then subjected to thermal annealing in a vacuum furnace at 750 K for 1.5 hours. After heating, due to thermal diffusion of atoms and their high mobility, the surface reached a more energetically favorable state, which corresponds to the classical Ostwald ripening process. The Ni/Si system tended to minimize its surface free energy, resulting in the formation of a smooth and well-defined interface.

These morphological changes indicate a reduction in electrical resistivity and an improvement in contact quality, as a smoother surface leads to lower electron scattering and better electrical conductivity. However, at excessive annealing temperatures of about 800 K, agglomeration effects may occur; therefore, the optimal processing temperature was determined to be 750 K [20]. Furthermore, the initially amorphous or fine-grained crystalline structure transformed into larger crystalline domains, which may indicate the early stage of $NiSi_2$ phase formation.

In Figure 2 given 2D and 3D surface morphology images of the nickel thin film obtained by the ion-plasma deposition method. The surface images were captured using a confocal laser scanning microscope (CLSM). As seen from the results, the surface roughness is extremely low, indicating a highly uniform and homogeneous film surface. This smoothness is attributed to the effect of subsequent thermal annealing, which significantly improves the microstructural quality of the film.

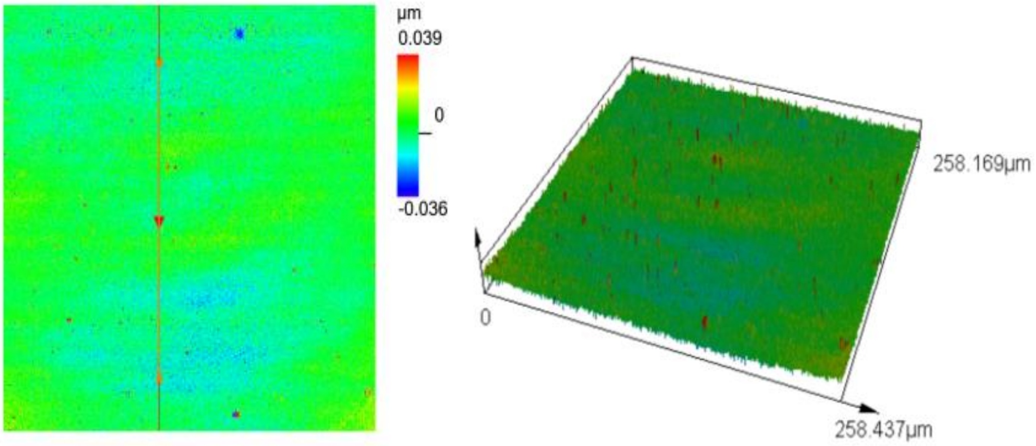

Figure 2. 2D and 3D atomic force microscopy (AFM) images of the Ni/Si thin films deposited by the ion-plasma method, showing the surface morphology and uniformity of the film.

Measurements were carried out at a depth of 390 nm, and the average surface roughness was determined to be 2.6 nm.

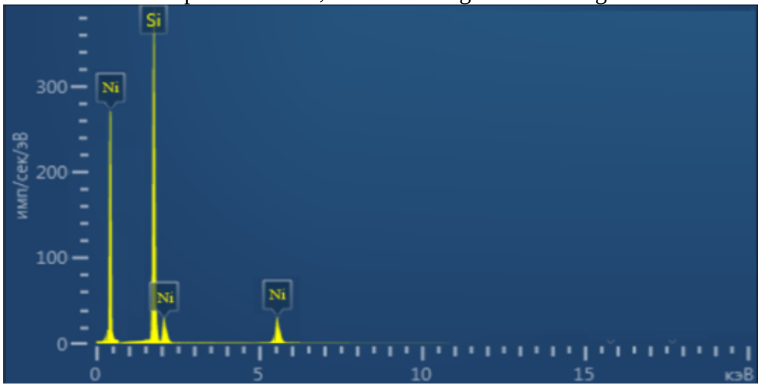

Figure 3. Energy-dispersive X-ray spectroscopy (EDS) spectrum of the Ni/Si thin film, showing distinct Si and Ni peaks, confirming the formation of nickel silicide phases.

Figure 3 shows the energy-dispersive X-ray spectroscopy (EDS) spectrum, where distinct peaks corresponding to silicon (Si) and nickel (Ni) are clearly observed. The Si peak is located at approximately ~1.0 keV, whereas the Ni peak appears near ~8.0 keV. The intensity of the Si peak is about 380 counts/eV, while that of the Ni peak is approximately 285 counts/eV.

These results indicate that the silicon concentration is roughly twice that of nickel, suggesting the formation of the $NiSi_2$ silicide phase within the thin film.

In the EDS spectrum, peaks corresponding to oxygen (O), carbon (C), or other impurities are practically absent, which confirms the high purity of the sample. The obtained results indicate that, at the Ni/Si interface, silicide phases - most likely $NiSi_2$ - were formed as a result of the ion-plasma deposition process. The relatively low intensity of the Ni peak suggests that a significant portion of nickel atoms reacted with silicon, leading to the formation of nickel silicide compounds.

The EDS results, combined with the surface morphology obtained via scanning electron microscopy (SEM), provide a comprehensive assessment of the film quality and its phase composition.

Table 1. Elemental composition of the Ni/Si thin film obtained by energy-dispersive X-ray spectroscopy (EDS).

Element	Weight %	Atomic %
Si	43.70	66.22
Ni	56.30	33.78

Element	Weight %	Atomic %
Total	100.00	100.00

Table 1 shows that, in terms of atomic fraction, silicon (Si) accounts for 66.22 at.%, which is significantly higher than the nickel (Ni) content of 33.78 at.%. This indicates the presence of a silicon-rich phase in the investigated material. In terms of weight fraction, nickel (56.30 wt.%) dominates, which can be explained by its higher atomic mass (Ni ≈ 58.7 a.m.u., Si ≈ 28.1 a.m.u.). The atomic ratio of Ni:Si ≈ 1:2 is close to the stoichiometry of $NiSi_2$, suggesting that a stable $NiSi_2$ silicide phase was formed in the film during ion-plasma deposition followed by thermal annealing.

REFERENCES

1. Lee, S., Mangelinck, D., Pey, K. L., Ding, J., Chi, D. Z., Osipowicz, T., Dai, J. Y., & See, A. (2002). Enhanced stability of Ni monosilicide on MOSFETs poly-Si gate stack. *Microelectronic Engineering*, 60, 171–181. https://doi.org/10.1016/S0167-9317(02)00407-1
2. Lavoie, C., d’Heurle, F. M., Detavernier, C., & Cabral, C. (2003). Towards implementation of a nickel silicide process for CMOS technologies. *Microelectronic Engineering*, 70, 144–157. https://doi.org/10.1016/S0167-9317(03)00320-1
3. Nurbobo Ugli, E. S. & Karshievich, T. A. (2025). Study of the process
4. of formation of heterostructural nanofilms Me_xSi_y/Si and Ga_xMe_{1-x}As/GaAs by ion implantation. J. Phys. Sci., 36(1), 17–25. https://doi.org/10.21315/jps2025.36.1.2
5. Liu, C. M., Liu, W. L., Hsieh, S. H., Tsai, T. K., & Chen, W. J. (2005). Interfacial reactions of electroless nickel thin films on silicon. *Applied Surface Science*, 243, 259–264. https://doi.org/10.1016/j.apsusc.2004.09.002
6. Zhao, F. F., Zheng, J. Z., Shen, Z. X., Osipowicz, T., Gao, W. Z., & Chan, L. H. (2004). Thermal stability study of NiSi and $NiSi_2$ thin films. *Microelectronic Engineering*, 71, 104–111. https://doi.org/10.1016/j.mee.2003.09.018
7. Pilipenko, V. A., Solovjov, J. A., & Gaiduk, P. I. (2021). Nickel silicide formation with rapid thermal treatment in the heat balance mode. *Doklady of the National Academy of Sciences of Belarus*, 65(1), 111–118. https://doi.org/10.29235/1561-8323-2021-65-1-111-118
8. Peter, A. P., Meersschaut, J., Richard, O., Moussa, A., Steenbergen, J., Schaekers, M., & Adelmann, C. (2015). Phase formation and morphology of nickel silicide thin films synthesized by catalyzed chemical vapor reaction of nickel with silane. *Chemistry of Materials*, 27, 245–254. https://doi.org/10.1021/cm504198g
9. Dovranov, K.T., Vinnichenko, M., Korablev, V., Normuradov, M., Eshboboyev, S., Egamberdiyeva. O. Raman and IR Spectrum Analysis of $CrSi_2$ Thin Films Formed in Direct Current and Variable Frequency Modes of a Magnetron Sputtering Device. International Conference on Electrical Engineering and Photonics, EExPolytech, 2024, pp. 304–307. DOI:10.1109/EExPolytech62224.2024.10755629
10. Nakahigashi, K., & Shimomura, Y. (1975). Electron microscope observations on nickel-oxide whiskers. *Journal of Crystal Growth*, 28(3), 367–371. https://doi.org/10.1016/0022-0248(75)90131-3
11. Wagner, R. S., & Ellis, W. C. (1964). Vapor–liquid–solid mechanism of single crystal growth. *Applied Physics Letters*, 4(5), 89–90. https://doi.org/10.1063/1.1753975
12. Detavernier, C., Lavoie, C., & d’Heurle, F. (2003). *Journal of Applied Physics*, 93, 2510. https://doi.org/10.1063/1.1540749
13. Bhaskaran, M., Sriram, S., Holland, A. S., & Evans, P. J. (2008). Characterisation of nickel silicide thin films by spectroscopy and microscopy techniques. *Micron*, 40(1), 99–103. https://doi.org/10.1016/j.micron.2008.07.001
14. Kamins, T. I., Williams, R. S., Chen, Y., Chang, Y., & Chang, Y. A. (2000). Chemical vapor deposition of Si nanowires nucleated by $TiSi_2$ islands on Si. *Applied Physics Letters*, 76(5), 562–564. https://doi.org/10.1063/1.125763
15. Tiwari, S., Rana, R., Chan, K., Shi, L., & Hanafi, H. (1996). Single charge and confinement effects in nanocrystal memories. *Applied Physics Letters*, 69(9), 1232–1234. https://doi.org/10.1063/1.117380
16. Hayes, W., & Loudon, R. (1978). *Scattering of Light by Crystals*. New York: Wiley.
17. Kamins, T. I., Williams, R. S., Basile, D. P., Hesjedal, T., & Harris, J. S. (2001). Ti-catalyzed Si nanowires by chemical vapor deposition: Microscopy and growth mechanisms. *Journal of Applied Physics*, 89(2), 1008–1016. https://doi.org/10.1063/1.1334614
18. Iwai, H., Ohguro, T., & Ohmi, S.-I. (2002). NiSi salicide technology for scaled CMOS. *Microelectronic Engineering*, 60, 157–169. https://doi.org/10.1016/S0167-9317(02)00394-5
19. Kang, T. J., Lee, H.-Y., & Kim, Y. H. (2007). Reduction of sheet resistance and low-thermal budget relaxation of stress gradients in polysilicon microcantilever beams using nickel-silicides. *Journal of Microelectromechanical Systems*, 16(2), 279–288. https://doi.org/10.1109/JMEMS.2007.892232
20. Lee, P. S., Mangelinck, D., Pey, K. L., Shen, Z. X., Ding, J., Osipowicz, T., & See, A. (2000). Micro-Raman spectroscopy investigation of nickel silicides and nickel (platinum) silicides. *Electrochemical and Solid-State Letters*, 3(3), 153–155. https://doi.org/10.1149/1.1390951
21. Qin, M., Poon, M. C., & Yuen, C. Y. (2000). A study of nickel silicide film as a mechanical material. *Sensors and Actuators A: Physical*, 87, 90–95. https://doi.org/10.1016/S0924-4247(00)00472-1