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FORMATION OF $Cu_{15}Si_4$/Si NANOPHASE FILMS ON SILICON SURFACES

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We report on the formation of copper silicide nanofilms using different magnetron sputtering modes. Copper silicide thin films were formed by sputtering Cu onto a Si(111) surface heated to 467 °C in high vacuum using the mid-RFMS method at a frequency of 100 kHz and a D=70% efficiency. The thickness of the resulting heteroepitaxial Cu/$Cu_{15}Si_4$/Si film was measured using SEM. Also, a $Cu_{15}Si_4$ film was formed by thermally annealing Cu/Si(111) nanofilms in a vacuum at 800 K for 1.5 hours using the DCMS method. The thickness and surface morphology of the obtained samples were studied using SEM. The formation of silicide films is confirmed by the results of energy-dispersive spectra. The formation of a copper (Cu) silicide film depends on the copper crystal size and substrate temperature, and at 467°C, a 75 nm-thick $Cu_{15}Si_4$ film was formed under a 130 nm-thick copper layer. These findings provide new insights into the mechanisms governing copper-silicon interface reactions and highlight the potential of copper silicide nanofilms to improve the performance of metal-oxide-semiconductor transistors and high-speed integrated circuits.

Keywords: *$Cu_{15}Si_4$; Ion-plasma; Silicide films; RFMS; Magnetron sputtering*
PACS: 68.55.-a; 81.15.Cd; 73.40.Ns

INTRODUCTION

In recent years, the copper-silicide (Cu-Si) system has been widely studied as a promising material for micro- and nanoelectronics, solar cell contacts [1-3], metal-oxide-semiconductor transistors [4], and high-speed integrated circuits (ICs) [5,6]. There are several stable and metastable phases in the Cu-Si system, the formation of which depends on the diffusion of copper and silicon atoms, the crystal structure of the substrate, and the temperature conditions. Cu and its silicide films are commonly produced by various methods, including direct current magnetron sputtering (DCMS) [7-9], mid-radio frequency magnetron sputtering (mid-RFMS) [10-12], high-power pulsed magnetron sputtering (HiPIMS), molecular beam epitaxy (MNE) [13], and reactive pulsed laser deposition (RPLD) [14]. Cu and its silicide films have been reported in many articles. However, most of them used glass substrates to form contact films, and several papers have processed silicon substrates [15]. Many studies have investigated the solid-state reaction sequence in Cu/α-Si systems based on amorphous silicon. It has been found that the reaction between copper and amorphous silicon initially forms the η″-Cu_3Si phase, which then proceeds to the γ-Cu_5Si phase [16-19]. Although the $Cu_{15}Si_4$ phase is thermodynamically stable, its formation is kinetically limited. This means that there is insufficient information to fully elucidate the formation mechanism of the $Cu_{15}Si_4$ phase during direct deposition, especially on single-crystal Si substrates.

Thus, although the existing literature provides important scientific results on the Cu–Si system, the direct formation of the $Cu_{15}Si_4$ phase on single-crystal Si substrates by ion-plasma magnetron deposition, its structural and phase properties have not yet been fully studied. Also, comparative studies of the effects of magnetron deposition modes, such as mid-RFMS and DCMS, on the formation of the Cu–Si phase are very few. This study aims to fill this scientific gap and open up the possibilities for the controlled synthesis of $Cu_{15}Si_4$/Si nanophase films.

MATERIALS AND METHODS

In this study, we simultaneously deposited films of different thicknesses in an argon atmosphere using a magnetron sputtering device in two different modes. Copper (Cu) with a purity of 99.998% was sputtered onto a single-crystal silicon surface by a solid-state ion-plasma method using variable reactive mid-frequency magnetron sputtering (mid-RFMS) and constant current magnetron sputtering (DCMS). We used a pulsed mid-RF mode of 100 kHz and a duty cycle of 70% to improve the crystallization and crystal size of copper (Cu) thin films grown at room temperature and 467°C. Si(111) was used as the substrate. The substrate temperature was monitored and controlled using a temperature regulator module (TRM, Epos, Russia) in combination with a K-type (TXA) thermocouple. The C and O on the film surface were removed by bombarding the substrate surface with low-energy ionized argon atoms using an "ion gun" source in a vacuum. Prior to deposition, the Si(111) substrates were cleaned by low-energy Ar^+ ion bombardment using an ion gun. The ion energy was set to 300 eV, and the cleaning process was carried out for 8–10 minutes. The base pressure before introducing argon was 4.1×10^{-6} Torr, while the chamber pressure during ion bombardment was maintained at 1.8×10^{-3} Torr. This procedure

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effectively removes native oxides and contaminants, ensuring good adhesion and reproducibility of thin-film growth. The maximum deposition rate and sputtering efficiency of the DCMS source under a power density of 1.15 W/cm² for the Cu target were 17 Å/s, D=70%, respectively. Furthermore, the formation of a $Cu_{15}Si_4$ film was confirmed by thermally annealing Cu/Si(111) nanofilms, deposited via the DCMS method, in a vacuum at 800 K for 1.5 hours. The surface morphology of the obtained silicide films was examined using an Olympus "LEXT™ OLS5100" laser confocal microscope and SEM. The elemental composition of the obtained samples was determined using EDS, and the phase structure of the Cu-Si system.

RESULTS AND DISCUSSION

In this work, two different ion-plasma magnetron sputtering approaches for forming $Cu_{15}Si_4$ silicide films on silicon surfaces were studied. We describe two methods using a magnetron sputtering device. Although the final product is the same in both methods, the mechanisms of phase formation are fundamentally different. The formation process of copper silicide films is shown in Figure 1.

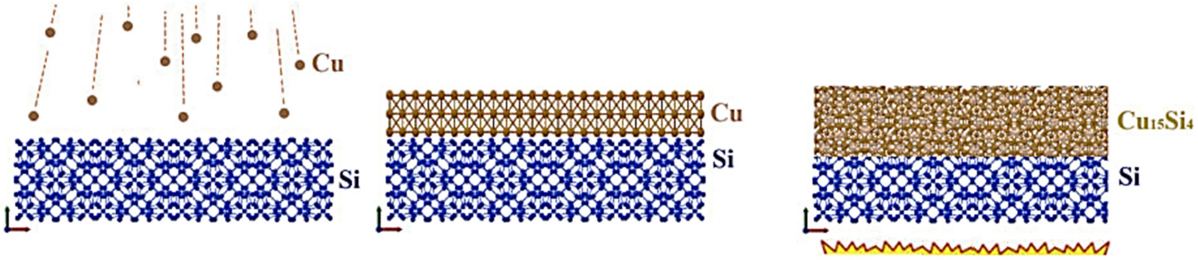


Figure 1. Schematic illustration of the formation mechanism of copper silicide films on Si(111) surfaces during magnetron sputtering using the DCMS deposition mode

Figure 1 demonstrates Cu atom deposition, diffusion into the heated silicon substrate, and the subsequent in situ formation of the $Cu_{15}Si_4$ silicide phase. In the first approach, copper (Cu) was sputtered onto the surface of a Si substrate at room temperature using the DCMS (Direct Current Magnetron Sputtering) method. Under these conditions, the diffusion of Cu atoms along the surface and into the bulk was limited due to the low substrate temperature. As a result, an amorphous or poorly crystallized Cu film was formed on the Si surface. The initial Cu film deposited by DCMS was structurally uneven, with grains and islands observed. This is explained by the limited mobility of Cu atoms along the surface and the lack of immediate chemical reaction with silicon. In the next step, this sample was heated in a vacuum at 800 K for 1.5 hours. During the heating process, the diffusion coefficient of Cu atoms increased sharply, and a solid-state reaction began at the Cu/Si interface. Copper atoms penetrated the silicon lattice, forming a thermodynamically stable $Cu_{15}Si_4$ silicide phase. According to the SEM results, the islands in the initial Cu film interacted with silicon during heating, forming a more homogeneous, continuous $Cu_{15}Si_4$ silicide film. This process represents a classical annealing-induced silicide formation mechanism controlled by diffusion.

Figure 2(a) shows the SEM image of the copper film deposited on the Si(111) substrate by the DCMS method before annealing. The surface exhibits a granular and island-like morphology, indicating limited surface diffusion of Cu atoms during room-temperature deposition. Such morphology is typical for as-deposited copper films on silicon, where insufficient thermal energy restricts atomic rearrangement and promotes the formation of isolated clusters.

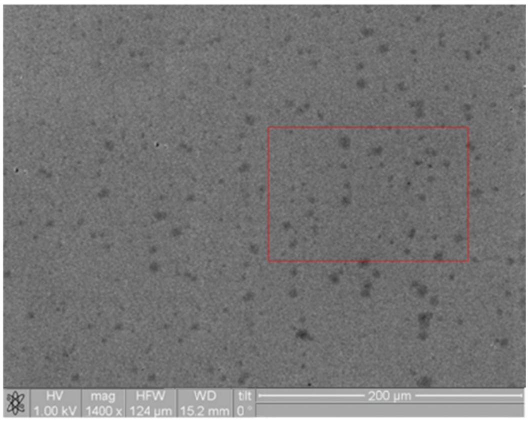


Figure 2(a). SEM image of a copper film formed by DCMS on a Si(111) surface (before annealing).

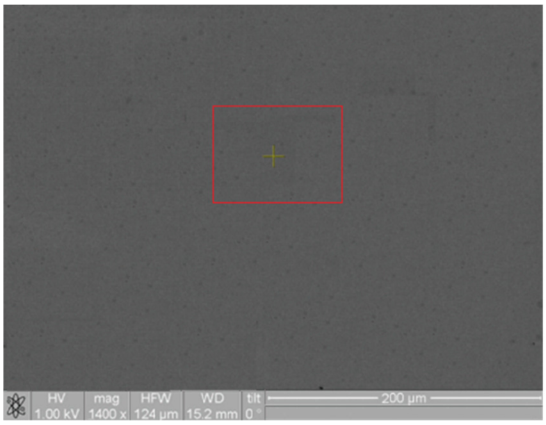


Figure 2(b). SEM image of a copper silicide film formed by the DCMS method (after annealing).

After thermal annealing, the surface morphology changes significantly, as shown in Figure 2(b). The film becomes more uniform and compact, which indicates enhanced atomic diffusion and the onset of a solid-state reaction between Cu and Si. The disappearance of isolated islands and the formation of a continuous layer suggest the transformation of the initial copper film into a copper silicide phase. This morphological evolution confirms that annealing activates Cu diffusion into the silicon substrate and promotes the formation of a stable $Cu_{15}Si_4$ silicide layer.

In the second approach, the Cu film was sputtered onto the surface of a Si(111) substrate heated to 467°C using the mid-RFMS (mid-Radio Frequency Magnetron Sputtering) method at a frequency of 100 kHz and a duty cycle of D = 70%. The main difference in this method is that silicide formation occurs without further heating, that is, directly during the deposition process itself. Due to the high temperature of the substrate, the sputtered Cu atoms have high mobility upon reaching the Si surface. Due to the high ionization degree of the plasma in the mid-RFMS mode, the Cu atoms receive additional energy and are able to diffuse deeper into the Si lattice. As a result, an in-situ solid-state reaction occurs at the Cu/Si interface, and the $Cu_{15}Si_4$ silicide phase begins to form at the time of deposition. This process is schematically shown in Figure 3, which depicts the diffusion of Cu atoms onto the heated Si substrate and the formation of a continuous silicide layer. In this approach, the optimal substrate temperature, ion-plasma energy, and pulsed mid-RFMS mode were controlled to form the $Cu_{15}Si_4$ phase. As a result, a more homogeneous, structurally stable, and well-crystallized silicide film was formed. The thickness of the resulting heteroepitaxial $Cu/Cu_{15}Si_4/Si$ film was measured using SEM (Figure 4). The film thickness was determined from the deposition rate and deposition time, with an uncertainty of approximately ±8%. The film thickness was also re-determined from the SEM images, with an estimated uncertainty of ±5%. The formation of the copper silicide film depends on the copper crystal size and substrate temperature, and at 467°C, a 75 nm-thick $Cu_{15}Si_4$ film was formed under a 130 nm-thick copper layer.

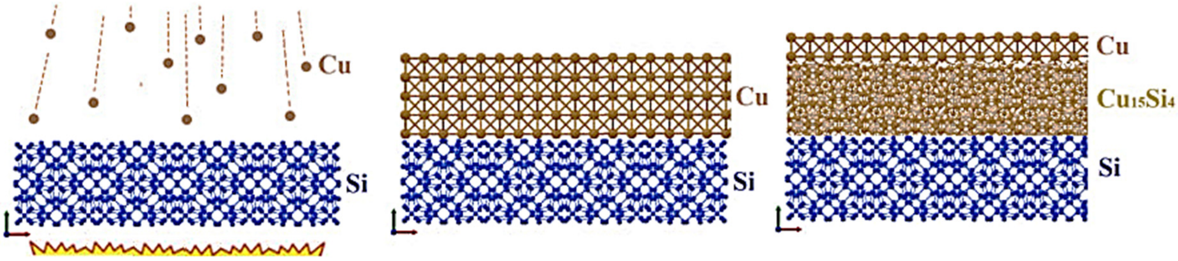


Figure 3. Formation mechanism of copper silicide films by the mid-RFMS method.

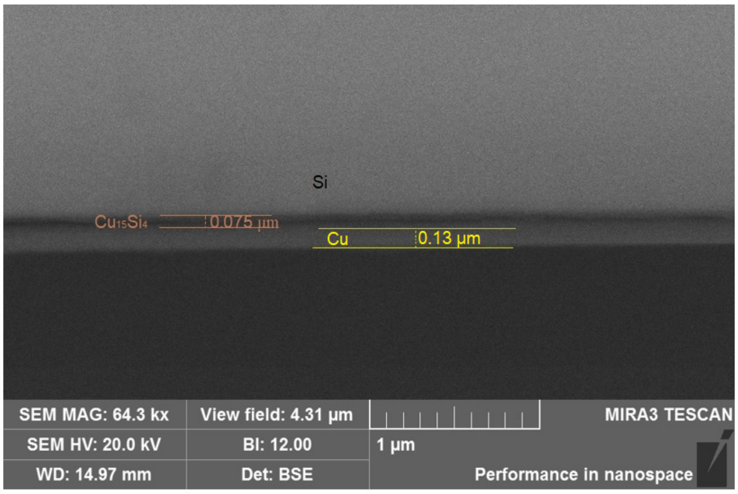


Figure 4. SEM image of $Cu/Cu_{15}Si_4/Si$ layer by the mid-RFMS method.

Thus, while in the "DCMS and heating" method the silicide phase is formed in two steps (deposition and annealing), in the "mid-RFMS and heated substrate" approach, the $Cu_{15}Si_4$ phase is formed in a single-step in-situ process. The second method, while being technologically simpler, allows for more precise control of phase formation. Figures 1 and 3, along with the experimental results, show that the formation of the $Cu_{15}Si_4$ silicide phase proceeds via a diffusion- and ion-plasma-controlled mechanism. A comparative analysis of the DCMS and mid-RFMS approaches clearly shows that the substrate temperature and plasma regime play a decisive role in silicide formation.

These results indicate that the mid-RFMS method is more effective for forming silicide phases in the Cu–Si system and is a promising technology for application in microelectronic devices. The elemental composition and mass fraction of the thin film were determined using energy dispersive spectroscopy (EDS) Figure 5. Based on the measurement results, the mass fraction of the sample, as a result of mid-radiofrequency pulsed magnetron sputtering (mid-RFMS) on the surface of monocrystalline silicon using a solid-phase ion plasma method, was 10.61% Si, 89.39% Cu, and the atomic fraction was 21.13% Si, 78.87% Cu (Table 1).

According to the results of the study, the formation of Cu and its silicide nanofilms from a copper target using the DCMS and mid-RFMS methods in the magnetron device depends on the initial substrate temperature and the subsequent heating temperatures of the films. The optimal temperature for crystallization of the films was found to be 467°C. The surface morphology of the formed copper silicide nanofilms was measured and analyzed using an atomic force microscope and an Olympus "LEXT™ OLS5100" laser confocal microscope (Figure 6-7, Table 2). Thanks to the advanced optical components, high-quality 3D measurements were obtained in these microscopes, and the surface roughness was found to be 6 nm.

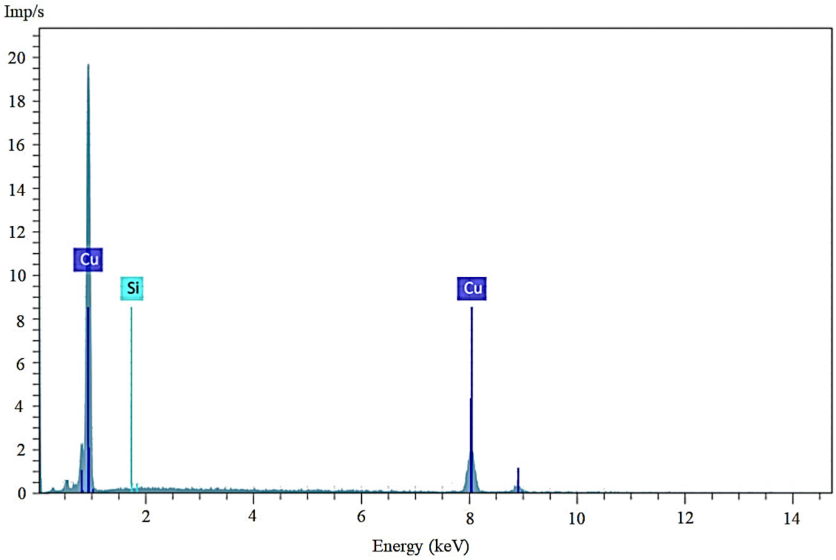


Figure 5. Energy-dispersive spectrum of a copper silicide film.

Table 1. Elemental composition of the copper silicide thin film

Element	mass. %	atom. %
Si	10.61	21.13
Cu	89.39	78.87
	100.00	100.00

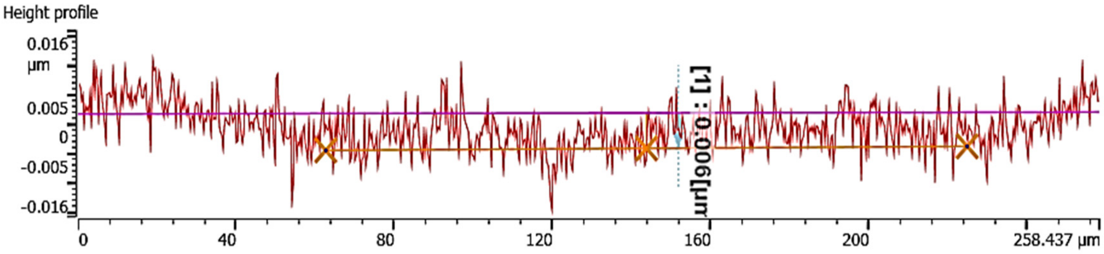
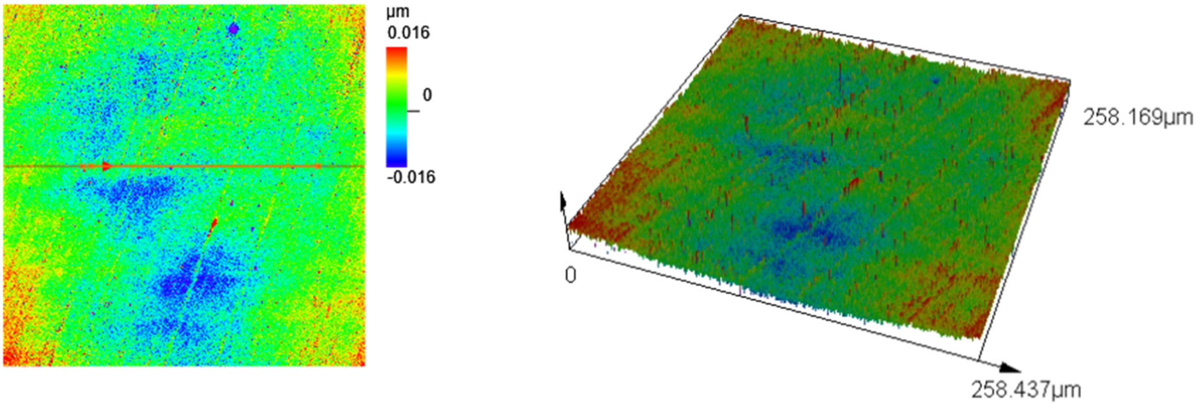


Figure 6. Surface roughness image of $Cu_{15}Si_4$ thin film formed by the DCMS method.



a) b)

Figure 7. Surface morphology of $Cu_{15}Si_4$ thin film formed by the DCMS method.
a) 2D image b) 3D image

Table 2. Surface roughness of the copper silicide thin film

no.	**Distance[μm]**	**Width[μm]**	**Height[μm]**	**Angle [°]**
1	0.006	168.216	0.006	179.991

The temperature-dependent electrical conductivity of the Cu–Si thin films was measured using a NETZSCH SBA 458 Nemesis® system, which employs the four-point probe technique for precise conductivity determination. Electrical contacts were established using the built-in current pins of the system, ensuring stable and reproducible ohmic contact. The sample was mounted in a horizontal configuration and measured in the temperature range from room temperature up to 380°C under controlled atmospheric conditions.

The SBA 458 Nemesis® allows automatic averaging of more than 100 readings per measurement point, significantly improving accuracy. According to the instrument specifications, the electrical conductivity measurement range is 0.05–150 000 S/cm, with an accuracy of ±5% and repeatability of ±3%. The system uses Inconel®-sheathed K-type thermocouples with fixed positions, providing reliable temperature control and eliminating uncertainties related to probe spacing. During measurements, the temperature was increased stepwise with sufficient stabilization time at each point to ensure thermal equilibrium before data acquisition.

Figure 8 shows the temperature dependence of the electrical conductivity of a Cu-Si thin film grown on a silicon surface. The graph shows that in the low-temperature range (from room temperature to about 120°C), the electrical conductivity is very low, which is explained by the film's initial structural state and the diffusion barriers at the Cu/Si interface. At this stage, the movement of electric carriers is limited by the influence of intergrain boundaries, defects, and the silicon substrate. When the temperature was increased to 150-250 °C, a sharp increase in electrical conductivity was observed. This phenomenon is associated with the active diffusion of Cu atoms into the silicon lattice and the onset of a solid-state reaction in the Cu–Si system. It is in this temperature range that the formation of a continuous metal silicide phase occurs, creating paths for carrying electric current (percolation network). As a result, the electrical conductivity increases by several orders of magnitude, which is a functional sign of the phase transition process. At temperatures above 250–350°C, the increase in electrical conductivity slows down and almost stabilizes.

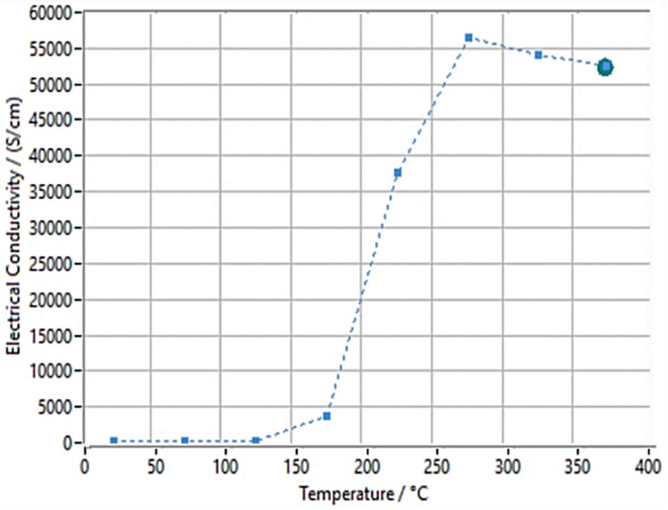


Figure 8. Temperature-dependent electrical conductivity of Cu–Si films

This saturation state indicates the complete formation of the copper silicide phase and the transition of the structure to a thermodynamically stable state. The electrical conductivity values observed at this stage (~5×10^4 S/cm) are lower than those of pure metallic copper but are in the range of values typical of metal silicides, in particular the $Cu_{15}Si_4$ phase. The temperature-dependent behavior of the electrical conductivity clearly indicates that phase changes are occurring in the film. While at low temperatures the interface and structural constraints dominate, at high temperatures the Cu–Si solid-state reaction leads to the formation of a metal silicide phase, dramatically improving the electrical transport properties of the film. These results further functionally confirm the presence of the $Cu_{15}Si_4$ phase, as detected by SEM and EDS analyses.

CONCLUSIONS

In this work, the formation mechanisms of copper silicide nanofilms on single-crystal silicon surfaces were systematically investigated using two different magnetron sputtering approaches. It was demonstrated that copper silicide formation is strongly governed by substrate temperature and the deposition process's kinetic conditions. Elevated substrate temperatures promote copper diffusion into the silicon lattice, enabling solid-state reactions and the formation of thermodynamically stable copper silicide phases. The comparative analysis revealed that copper silicide can be formed either through post-deposition thermal treatment or directly during deposition under ion-assisted conditions. In particular, ion-plasma-assisted sputtering on heated silicon substrates enables in situ formation of the $Cu_{15}Si_4$ phase, offering improved control over phase composition and interfacial structure. The resulting heterostructures exhibit well-defined Cu/$Cu_{15}Si_4$/Si configurations, confirming that copper crystallite size and substrate temperature play a decisive role in silicide phase evolution. DCMS and mid-RFMS modes were used under the same conditions to systematically study their effects on the formation of the $Cu_{15}Si_4$ phase. The $Cu_{15}Si_4$ phase is reliably confirmed by EDS, with a Cu/Si atomic ratio close to the theoretical value, providing a solid scientific basis for its existence. The obtained results provide clear insight into the diffusion-driven mechanisms responsible for copper silicide formation and highlight the advantages of ion-assisted magnetron sputtering for controlled silicide synthesis. These findings contribute to a deeper understanding of Cu–Si interfacial reactions and demonstrate the potential of $Cu_{15}Si_4$ silicide layers for applications in silicon-based microelectronic devices, including metal–oxide–semiconductor transistors and high-speed integrated circuits.

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ФОРМУВАННЯ НАНОФАЗНИХ ПЛІВОК $Cu_{15}Si_4/Si$ НА КРЕМНІЄВИХ ПОВЕРХНЯХ

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Ми повідомляємо про формування наноплівок силіциду міді з використанням різних режимів магнетронного розпилення. Тонкі плівки силіциду міді були сформовані шляхом розпилення Cu на поверхню Si(111), нагріту до 467°C у високому вакуумі, використовуючи метод середньої RFMS на частоті 100 кГц та з ефективністю D = 70%. Товщину отриманої гетероепітаксіальної плівки $Cu/Cu_{15}Si_4/Si$ було виміряно за допомогою скануючої електронної мікроскопії (СЕМ). Також плівку $Cu_{15}Si_4$ було сформовано шляхом термічного відпалу наноплівок Cu/Si(111) у вакуумі при 800 K протягом 1,5 годин за допомогою методу DCMS. Товщину та морфологію поверхні отриманих зразків було досліджено за допомогою СЕМ. Формування силіцидних плівок підтверджено результатами енергодисперсійних спектрів. Формування плівки силіциду міді (Cu) залежить від розміру кристалів міді та температури підкладки, і при 467°C під шаром міді товщиною 130 нм було сформовано плівку $Cu_{15}Si_4$ товщиною 75 нм. Ці результати дають нове розуміння механізмів, що регулюють реакції на межі розділу мідь-кремній, та підкреслюють потенціал наноплівок силіциду міді для покращення продуктивності транзисторів метал-оксид-напівпровідник та високошвидкісних інтегральних схем.

Ключові слова: *$Cu_{15}Si_4$; іонна плазма; силіцидні плівки; RFMS; магнетронне розпилення*