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# INFLUENCE OF ANNEALING CONDITIONS ON THE SURFACE TOPOGRAPHY OF THIN FILMS OF MANGANESE SILICIDES

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**ABSTRACT**

This work examines the influence of thermal annealing conditions on the surface topography of thin manganese silicide films fabricated by ion implantation. Implantation was carried out at an energy of 100 keV with doses of $1\times10^{15}$, $5\times10^{15}$, and $1\times10^{16}$ $cm^{-2}$, resulting in an average ion penetration depth of 90–110 nm and a distribution width of approximately 40 nm. Thermal annealing was performed in vacuum at temperatures ranging from 600 °C to 800 °C for 30 minutes.

Atomic force microscopy (AFM) analysis revealed that increasing the annealing temperature leads to a rise in RMS surface roughness from 0.8 nm to 4–5 nm, and an increase in the average grain size from 20–30 nm to 80–120 nm. Additional parameters, including surface area, protrusion volume, height distribution, and grain statistics, were also evaluated. The results demonstrate that the surface topography of thin manganese silicide films can be effectively controlled through thermal annealing, a factor of critical importance for the development of micro- and nanoelectronic devices.

**KEYWORDS**

Manganese silicides, thin films, surface topography, atomic force microscopy, thermal annealing, ion implantation, surface morphology, RMS roughness, height distribution.

## Introduction

Transition metal silicides are an integral part of modern silicon micro- and nanoelectronics, as they provide effective reduction of contact resistance, high thermal stability and compatibility with basic technological processes. Significant successes achieved in the field of epitaxial-planar technology and ion implantation using modern methods of submicron lithography, ion-beam doping, ion-beam etching, local epitaxy, have now made it possible to industrially produce ultra-large integrated circuits based on silicon. However, the great interest of researchers in the purposeful change and study of the

physicochemical properties of the surface of solids, first of all, is due to the great practical significance of the results obtained for various fields of modern science and technology.

Manganese thin-film silicides (MnSi, $Mn_5Si_3$) are promising materials for silicon-compatible electronic and spintronic devices due to their metallic conductivity, thermal stability, and magnetic properties.

Control of the surface morphology of such films is critical to reduce charge carrier scattering, improve adhesion of subsequent layers, and increase the stability of contacts and functional layers.

One of the most radical ways of influencing the condition and surface properties of solids is ion implantation. If the introduction of foreign atoms into silicon with medium energies (tens and hundreds of keV) and low energies (up to 1013 $cm^{-2}$) doses allow you to control individual properties of Si, then the implantation of high-energy ions (up to 100 keV) with a large dose (1015 ÷ 1017 $cm^{-2}$) makes it possible to create surface layers with a concentration of impurity atoms of the same order as the concentration of Si atoms, and in some cases even more. In other words, low-energy ion implantation can serve as a powerful tool for the synthesis of new thin film materials with modified properties. However, the successful use of this method depends on an understanding of the diverse processes that occur in a solid state at the electronic and atomic levels during ion implantation.

In this regard, the influence of annealing conditions on the surface topography of thin films of manganese silicides formed by ion implantation and thermal annealing is relevant.

**INVESTIGATIONS**

Monocrystalline n-type silicon wafers of Si(100) orientation with a resistivity of 5–10 Ohm·cm were used as substrates.

The method makes it possible to form non-equilibrium systems with an impurity concentration above the solubility limit, to control the deep distribution of manganese and to obtain crystal phases with controlled crystallite sizes and surface topography.

According to Monte Carlo calculations (SRIM), at a given energy, the average depth of the ion path is 90–110 nm, and the width of the distribution is about 40 nm. This ensures the formation of an alloyed near-surface layer that is optimal for silicide formation.

The implantation doses were 1×, 5× and 1×, which makes it possible to study the transition from low manganese concentrations to modes at which the local concentration of the impurity significantly exceeds the limit of manganese solubility in silicon (~$10^{15}10^{15}10^{16}см^{-2}10^{15}см^{-3}3$). The ion current density did not exceed 1 μA/cm², the temperature of the substrates during implantation did not exceed 100 °C, which excluded the effect of dynamic annealing.

After implantation, the specimens were thermally annealed in vacuum at a residual pressure of the order of Pa. The annealing temperature ranged from 600 to 800 °C, with a holding time of 30 minutes. These modes were chosen as a compromise between activating the diffusion of manganese and preventing its deep diffusion into the volume of the substrate.$10^{-5}$

Structural studies were carried out by X-ray diffraction (CuKα, geometry θ–2θ). The surface morphology was analyzed by atomic force microscopy in the scanning range of 1×1 and 5×5 μm². Electrical properties were evaluated by four-probe resistivity measurement at room temperature.

Results and discussion

X-ray diffraction studies have shown that the phase composition of the implanted layers is determined by the combined effect of the implantation dose and the thermal annealing temperature. At a dose of 1×, and an annealing temperature of 600 °C, there are no pronounced peaks of silicidal phases in the diffraction spectra, which indicates an amorphous or highly dispersed state of manganese in silicon.$10^{15}$см$^{-2}$

With an increase in the implantation dose to 5×, and the annealing temperature to 700 °C, the appearance of diffraction peaks corresponding to the MnSi phase is observed. This indicates the beginning of active silicide formation, due to the excess of the local concentration of manganese over the solubility limit and the activation of diffusion processes.$10^{15}$см$^{-2}$

At a maximum dose of 1×, and an annealing temperature of 800 °C, a more thermodynamically stable $Mn_5Si_3$ phase is formed. The transition from MnSi to $Mn_5Si_3$ is associated with an increase in the concentration of manganese and an increase in the mobility of atoms at high temperatures.$10^{16}$см$^{-2}$

Estimation of the size of the coherent scattering regions showed that the average size of crystallites increases from 20–30 nm at 700 °C to 50–60 nm at 800 °C, indicating an increase in the degree of crystallinity and a decrease in defects.

The nanocrystalline structure of silicidal films is due to non-equilibrium conditions of formation. The high density of defects that occur during implantation plays the role of centers of silicidal phase nucleation. In the annealing process, partial annihilation of defects and crystallite growth occurs, accompanied by relaxation of mechanical stresses. However, at temperatures above 800 °C, the development of undesirable processes is possible, such as diffusion of manganese deep into the substrate and aggregation of silicidal phases, which impairs the localization of the functional layer.

AFM studies have revealed a distinct evolution of the surface morphology with increasing annealing temperature. At 600 °C, the surface remains relatively smooth, with an RMS roughness of no more than 1 nm. At 700 °C, a granular structure is formed with a characteristic grain size of 40–60 nm. Annealing at 800 °C leads to the formation of insula morphology, characterized by grain enlargement to 80–120 nm and an increase in roughness to 4–5 nm. relaxation of internal tensions. Measurements of resistivity have shown a systematic decrease with increasing annealing temperature. For samples annealed at 600 °C, the resistivity is about $8\times10^{-4}\Omega\cdot$cm, while at 800 °C it decreases to 1-2× $10^{-4}\Omega\cdot$cm.

X-ray diffraction spectra show that at low doses and annealing temperatures up to 600 °C, silicide phases are in an amorphous or highly dispersed state. With an increase in temperature and implantation dose, crystalline phases of MnSi and $Mn_5Si_3$ are formed.

**Phase composition and structural parameters Table 1.**

| | Dose, cm$^{-2}$ | Annealing, °C | Phase | Crystallite size, nm |
|---|---|---|---|---|
| 1. | $1\times10^{15}$ | 600 | - | <10 |
| 2. | $5\times10^{15}$ | 700 | MnSi | 20–30 |
| 3. | $1\times10^{16}$ | 700 | MnSi | 30–45 |
| 4 | $1\times10^{16}$ | 800 | $Mn_5Si_3$ | 40–60 |

Table 1 presents the results of X-ray phase analysis of films after implantation with different doses and subsequent thermal annealing at 600–800 °C. At a dose of $1\times10^{15}$ $cm^{-2}$ and annealing at 600 °C, the crystalline phases are not detectable, and the crystallites do not exceed 10 nm, indicating a predominantly amorphous or nanocrystalline layer state. Increasing the dose to $5\times10^{15}$ $cm^{-2}$ and annealing at 700 °C results in the formation of a MnSi phase with a crystallite size of 20–30 nm. A further increase in the dose to $1\times10^{16}$ $cm^{-2}$ at the same temperature is accompanied by an increase in crystallites to 30–45 nm, which indicates the intensification of the processes of silicide formation and structural ordering. At a dose of $1\times10^{16}$ $cm^{-2}$ and annealing at 800 °C, the $Mn_5Si_3$ phase is formed with a crystallite size of 40–60 nm. The transition to this phase and the increase in crystallite size reflect the development of solid-phase reactions and the formation of a more thermodynamically stable compound at elevated temperatures.

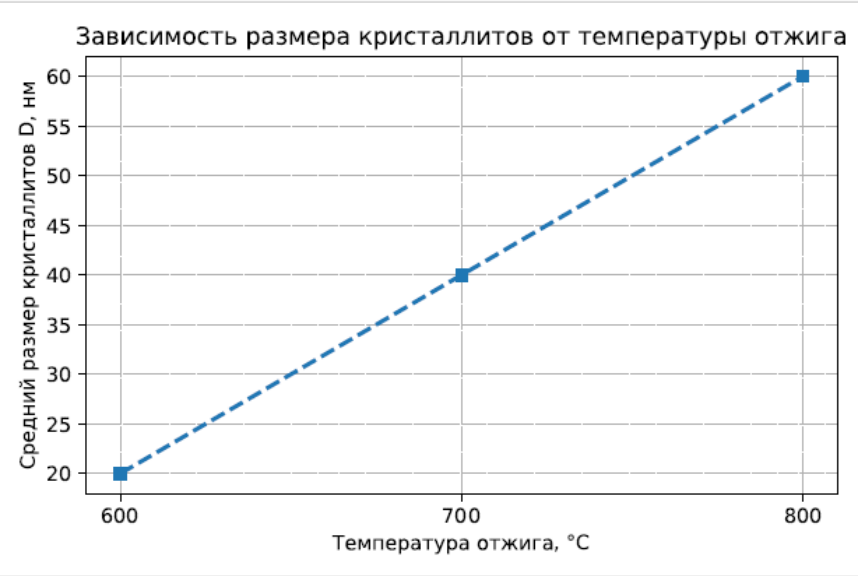


**Fig. 1.** X-ray diffraction spectra of samples after annealing at 600, 700 and 800 °C. (description: at 700 °C there are MnSi peaks, at 800 °C there are intense $Mn_5Si_3$ peaks)

Figure 1 shows the X-ray diffraction spectra of samples after thermal annealing at 600, 700 and 800 °C. After annealing at 600 °C, there are no pronounced peaks of silicidal phases on the diffractogram; either weak and expanded maxima corresponding to the nanocrystalline state or a predominantly diffuse background are observed, indicating an amorphous or weakly crystallized structure. When the temperature rises to 700 °C, distinct diffraction peaks corresponding to the phase of MnSi are recorded on the spectra. The appearance of these reflexes indicates the beginning of active silicide formation and the formation of a crystal structure. The intensity of the peaks and their narrowing compared to the sample annealed at 600 °C indicate the growth of crystallites and an increase in the degree of structural order. After annealing at 800 °C, intense and well-resolved $Mn_5Si_3$ phase peaks are observed on the diffractogram. A significant increase in their intensity and a decrease in the half-width indicate the formation of a more thermodynamically stable phase and the further growth of crystallites. At the same time, a decrease in the relative intensity of MnSi peaks is possible, indicating a phase transformation during high-temperature treatment.

Thus, X-ray phase analysis demonstrates a consistent evolution of phase composition as the annealing temperature increases: from an amorphous or nanocrystalline state (600 °C) to the formation of MnSi (700 °C) and the subsequent formation of $Mn_5Si_3$ at 800 °C.

AFM studies revealed a significant change in the morphology of the surface with an increase in the annealing temperature.

Surface Topography Parameters of Manganese Silicide Thin Films Table 2.

| | Annealing, °C | RMS roughness, nm | Average grain size nm | Min. Height, nm | Standard deviation, nm |
|---|---|---|---|---|---|
| 1. | 600 | 0,8 | 20-30 | 1,01 | 0,005 |
| 2. | 700 | 1,9 | 40-60 | 1,05 | 0,012 |
| 3. | 800 | 4,5 | 80-120 | 1,12 | 0,028 |

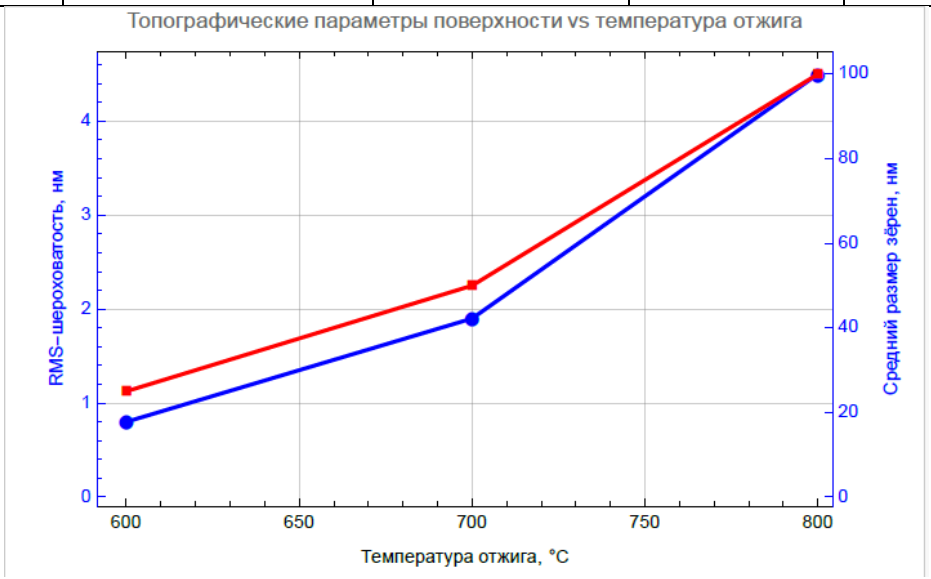


**Fig.2** Dependence of RMS roughness and average grain size of silicidal films on thermal annealing temperature

Figure 2 shows the dependence of RMS roughness and average grain size on annealing temperature in the range of 600–800 °C. An increase in temperature from 600 to 700 °C leads to an increase in RMS roughness by more than 2 times (from 0.8 to 1.9 nm), and a further increase in temperature to 800 °C is accompanied by a sharp increase in roughness to 4.5 nm. A similar trend is observed for the average grain size: at 700 °C, the size increases to 40–60 nm, and at 800 °C it reaches 80–120 nm.

The nonlinear nature of the growth of parameters indicates the intensification of thermally activated structural restructuring processes at temperatures above 700 °C. A significant increase in grain size correlates with an increase in roughness, which indicates the formation of a more pronounced grain morphology of the surface.

Thus, the presented dependencies confirm the decisive influence of the annealing temperature on the evolution of the microstructure and surface topography of thin films of manganese silicides. Conclusion: As the annealing temperature increases, the surface becomes rougher, grainier and more insulated. Coalescence of crystallites reduces the density of grains, increases their volume and the area of protrusions.

**Resistivity Table 4.**

| | Annealing, °C | ρ, Ω·cm |
|---|---|---|
| 1. | 600 | $8\times10^{-4}$ |
| 2. | 700 | $3\times10^{-4}$ |
| 3. | 800 | $1.5\times10^{-4}$ |

Table 4 shows the values of electrical resistivity (ρ) of films after thermal annealing at 600, 700 and 800 °C. With an increase in the annealing temperature, a consistent decrease in resistivity is observed: from $8\times10^{-4}$ Ω·cm at 600 °C to $1.5\times10^{-4}$ Ω·cm at 800 °C. Thus, with an increase in temperature in the studied range, the resistance decreases by more than 5 times. A decrease in ρ at 700 °C ($3\times10^{-4}$ Ω·cm) compared to 600 °C indicates an improvement in the crystal structure and the formation of a conductive silicidal phase. A further decrease in resistance at 800 °C is associated with the completion of phase transformations and the growth of crystallites, which leads to a decrease in the scattering of charge carriers at grain boundaries and structural defects.

The data obtained confirm the significant effect of annealing temperature on the electrical properties of thin films of manganese silicides.

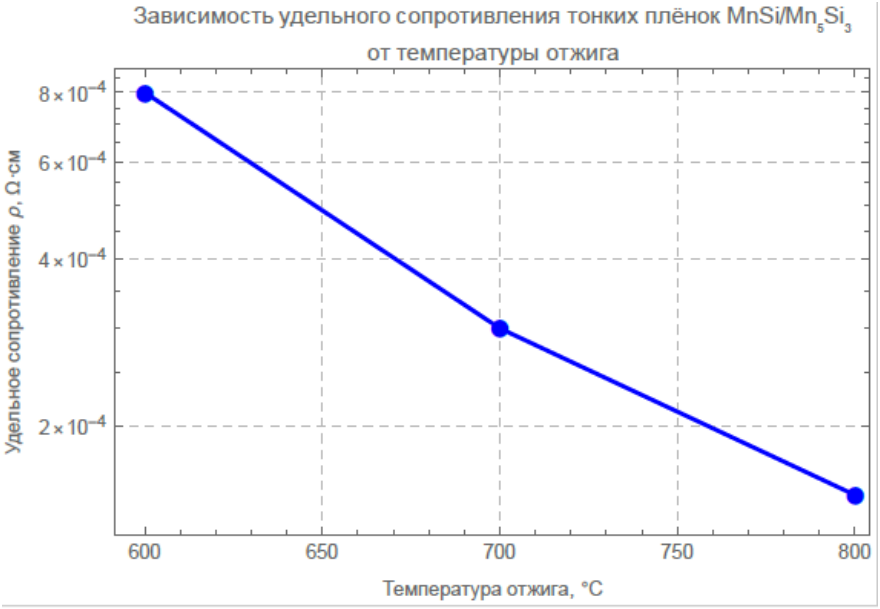


**Fig. 4.** Dependence of resistivity on annealing temperature.
(description: monotonic decrease in resistance with a rise in T)

Figure 4 shows the dependence of electrical resistivity on the temperature of thermal annealing in the range of 600–800 °C. The most significant decrease in ρ occurs during the transition from 600 to 700 °C (a decrease of more than 2.5 times), which correlates with the formation of a crystalline silicide phase. With a further increase in temperature to 800 °C, the resistance decreases to $1.5\times10^{-4}$ Ohm·cm, which is associated with an increase in the size of crystallites and a decrease in the concentration of structural defects. Thus, temperature treatment is a key factor in determining the level of electrical conductivity of films, and improving their structural condition leads to a decrease in resistivity.

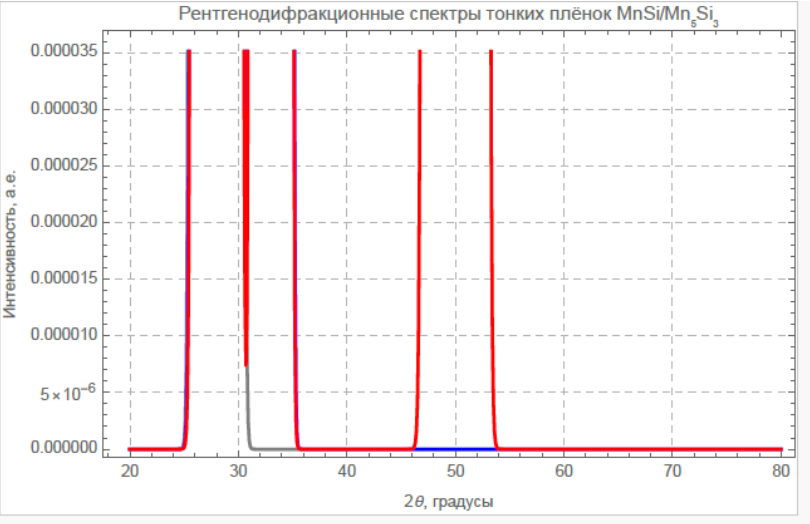


Fig. 5. Dependence of the average size of manganese silicide crystallites on the temperature of thermal annealing.

The figure shows the dependence of the average crystallite size of thin films of manganese silicides on the thermal annealing temperature in the range of 600–800 °C. Annealing at 600 °C does not form crystalline phases, and the size of crystallites does not exceed 10 nm, which indicates a predominantly amorphous or nanocrystalline layer state. An increase in temperature to 700 °C leads to the appearance of a crystalline phase of MnSi. The average crystallite size is 25–37.5 nm. depending on the dose of implantation. The increase in the size of crystallites at this stage is associated with the activation of diffusion processes and the beginning of grain coalescence. With a further increase in temperature to 800 °C, a thermodynamically more stable phase of $Mn_5Si_3$ is formed. The average size of crystallites reaches 50 nm, which reflects the completion of granular migration and the growth of crystallites. Thus, the dependence of the average size of crystallites on temperature is positive: an increase in the temperature of thermal annealing contributes to an increase in the size of crystallites and the formation of an ordered crystal structure of manganese silicide films. The results obtained indicate that the formation of silicidal phases of manganese is determined by the competition of diffusion, recrystallization and relaxation of stresses. At temperatures of 700–800 °C, optimal conditions are realized for the formation of crystalline phases MnSi and $Mn_5Si_3$, which is accompanied by an improvement in electrical characteristics.

**Conclusion**

In this work, the effect of thermal annealing conditions on the surface topography of thin films of manganese silicides formed by ion implantation, a comprehensive study of the formation processes, structural features and morphology of thin films of manganese silicides obtained by ion implantation and subsequent thermal annealing are investigated. It has been established that the phase composition, crystal structure and morphology of the surface significantly depend on the implantation dose and annealing temperature. It is shown that the optimal conditions for the formation of crystalline silicide phases are realized at implantation doses of the order of 1×, and annealing temperatures of 700–800 °C. The results obtained demonstrate the possibility of controlled synthesis of manganese silicides with

specified structural and morphological characteristics, which makes this approach promising for the creation of silicon-compatible functional materials for micro- and nanoelectronics.$10^{16}$см$^{-2}$ Coalescence of crystallites leads to the formation of insula morphology and a decrease in the density of grains per unit area. The electrical properties of the films improve with increasing crystallinity: the resistivity decreases to ~1.5×10 $^{-4}$ Ω·cm at 800 °C. Control of annealing parameters allows you to control the morphology of the surface and create functional layers with specified characteristics for micro- and nanoelectronics. The ion implantation method followed by thermal annealing provides controlled surface formation with controlled topographic and electrical properties.

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