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STRUCTURAL FEATURES AND MORPHOLOGY OF THIN FILMS OF MANGANESE SILICIDES OBTAINED BY ION IMPLANTATION AND THERMAL ANNEALING

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ABSTRACT

The paper investigates the formation processes, structural features and morphology of thin films of manganese silicides obtained by ion implantation of manganese into single-crystal silicon followed by thermal annealing.

It has been shown that at low implantation doses and annealing temperatures up to 600 °C, silicidal phases are predominantly in an amorphous or nanocrystalline state. Increasing the implantation dose to and the annealing temperature to 700–800 °C results in the formation of crystalline phases of MnSi and Mn_5Si_3. The size of crystallites, estimated from X-ray diffraction analysis, increases from 20–30 nm to 50–60 nm with increasing annealing temperature.10^16 〖см〗^(-2)

Atomic force microscopy has established that an increase in the degree of crystallinity is accompanied by a change in the morphology of the surface: the RMS roughness increases from ~0.8 nm at 600 °C to 4–5 nm at 800 °C, while a granular and insular structure is formed. Measurements of the electrical properties showed a decrease in the resistivity of the films to values of the order of Ω10^(-4)·cm under optimal annealing regimes, which correlates with an improvement in the crystal order.

KEYWORDS

Manganese silicides, ion implantation, thermal annealing, thin films, structure, morphology

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. In devices of the submicron and nanometer scale, it is the properties of the

contact and interconnecting layers that largely determine the speed and reliability of devices, which causes a steady interest in the study of new silicidal materials and methods of their formation.

A special place among the silicides of transition metals is occupied by manganese silicides - MnSi, Mn_5Si_3 and $MnSi_{1.75}$. These compounds are characterized by a complex electronic structure and exhibit a wide range of physical properties, including metallic conductivity, magnetic ordering, and pronounced dimensional effects. In particular, MnSi silicide is a model object for the study of chiral magnetism and non-collinear spin structures. The transition to the thin-film state leads to a significant modification of its properties, which is associated with the influence of grain boundaries, defects, mechanical stresses, and surface morphology.

Traditional methods of forming thin films of manganese silicides (magnetron sputtering, vacuum sputtering, molecular beam epitaxy) require strict control of stoichiometry and temperature regimes, and are often accompanied by problems of phase heterogeneity and diffusion interaction with the substrate. In addition, these methods are limited in their ability to control the deep distribution of manganese in silicon.

In this context, ion implantation followed by thermal annealing represents a technologically attractive approach. Ion implantation makes it possible to form highly non-equilibrium systems with a high concentration of impurities exceeding the limit of its solubility in silicon. Subsequent annealing initiates the processes of diffusion, recrystallization and phase formation, which leads to the formation of silicidal phases directly in the near-surface layer of the substrate.

It should be emphasized that silicide formation in the implanted layers takes place under conditions far from thermodynamic equilibrium. Silicon amorphization, high density of radiation defects, and the presence of residual mechanical stresses significantly affect the kinetics of phase transformations, the choice of the forming phase, and the morphology of the resulting films. In this regard, the task of a comprehensive study of the structural and morphological characteristics of manganese silicides formed by ion implantation and thermal annealing is urgent.

INVESTIGATIONS

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

Implantation was carried out at an ion energy of 100 keV and doses in the range of 1×–1×.Thermal annealing was carried out in vacuum at temperatures of 600–800 °C for 30 minutes.$10^{15}10^{16}см^{-2}$

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 a doped near-surface layer, 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. $\mathbf{10^{15}}$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.$\mathbf{10^{15}}$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.$\mathbf{10^{16}}$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 revealed a distinct evolution of surface morphology with increasing annealing temperature. At 600 °C, the surface remains relatively smooth, the RMS roughness does not exceed 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 the enlargement of grains to 80–120 nm and an increase in roughness to 4–5 nm. This effect is due to the coalescence of silicidal crystallites and the relaxation of internal stresses.

Measurements of resistivity have shown a systematic decrease with increasing annealing temperature. For samples annealed at 600 °C, the resistivity is on the order of $8\times10^{-4}\Omega\cdot$cm, while at 800 °C it decreases to 1-2× $10^{-4}\Omega\cdot$cm.

The decrease in resistance is associated with an increase in the degree of crystallinity, an increase in the size of grains, and a decrease in the scattering of charge carriers at defects and grain boundaries.

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	1×10^{15}	600	-	<10
2	5×10^{15}	700	MnSi	20–30
3	1×10^{16}	700	MnSi	30–45
4	1×10^{16}	800	Mn_5Si_3	40–60

Table 1 presents the results of the study of the phase composition and size of crystallites in samples depending on the implantation dose and the temperature of subsequent thermal annealing. The data obtained make it possible to trace the patterns of phase formation and structural evolution of the material when varying technological parameters.

It was found that at a minimum implantation dose of 1×10^{15} cm^{-2} and an annealing temperature of 600 °C (sample No1), crystalline phases were not fixed. The size of the structural formations does not exceed 10 nm, which indicates a predominantly amorphous state or the formation of an ultrafine structure with a low degree of crystallinity. These results indicate the insufficiency of thermal energy to activate the processes of directed phase formation and crystallite growth.

Increasing the dose to 5×10^{15} cm^{-2} at 700 °C (sample No2) results in the formation of the MnSi phase. The size of crystallites is 20–30 nm, which indicates the development of diffusion-controlled processes and the beginning of the structural orderliness of the system. Thus, an increase in the annealing temperature to 700 °C provides conditions for the formation of a silicidal phase.

With a further increase in the dose to 1×10^{16} cm^{-2} at the same temperature (sample No3), the phase composition is retained (MnSi), but an increase in the size of crystallites to 30–45 nm is observed. The absence of a change in the phase composition with the simultaneous growth of crystallites indicates the predominance of the processes of coalescence and enlargement of grains over the processes of nucleation of new phases.

The maximum annealing temperature of 800 °C at a dose of 1×10^{16} cm^{-2} (sample No4) is followed by a phase transformation to form Mn_5Si_3. At the same time, an increase in the size of crystallites to 40–60 nm is recorded. Probably, at this temperature, the transition to a more thermodynamically stable phase takes place, which is due to the intensification of atomic diffusion and the redistribution of the components of the system.

In general, the results demonstrate a consistent evolution of the structure of the material: from an amorphous or nanocrystalline state to the formation of the MnSi phase and its subsequent enlargement, and at a higher temperature to the formation of the Mn_5Si_3 phase with further growth of crystallites. The revealed regularities confirm the decisive role of annealing temperature and implantation dose in the processes of phase formation and structural transformation of the material.

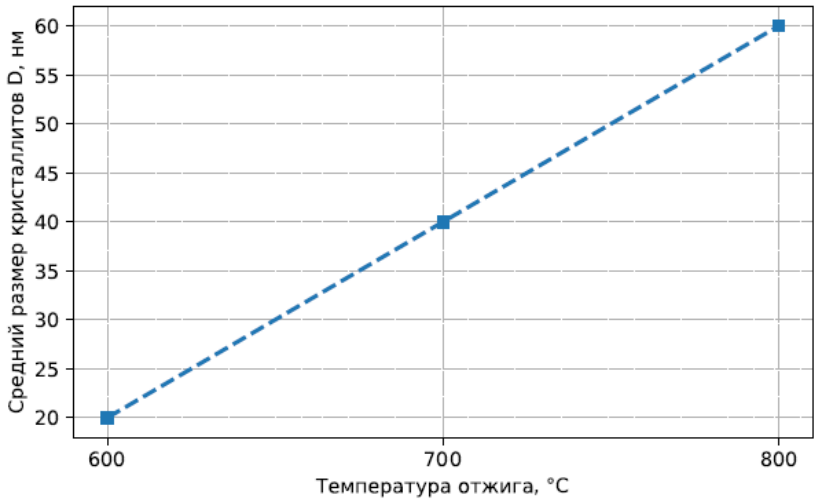

Fig. 1. **X-**ray diffraction spectra of samples after annealing at 600, 700 and 800 °C.(description: at 700 °C there are MnSi peaks, at 800 °C there are intense Mn_5Si_3 peaks)

Figure 1 shows the X-ray diffraction (XRD) spectra of samples after annealing at 600, 700, and 800 °C. The abscissa axis shows the diffraction angle of 2θ (degrees), and the ordinate axis shows the relative intensity of the diffraction signal. Comparison of the spectra makes it possible to trace the phase evolution of the material as the annealing temperature increases.

Annealed at 600 °C. The diffraction spectrum is characterized by weakly pronounced and relatively wide peaks or a predominantly diffuse background. This indicates a low degree of crystallinity or the formation of a finely dispersed, partially amorphous structure. Clearly identifiable reflexes of silicidal phases are practically absent or have a very low intensity.

Annealed at 700 °C. Distinct diffraction peaks corresponding to the phase of MnSi appear on the spectrum. Their appearance indicates the solid-phase reaction between the components and the crystallization of manganese silicide. The peaks become narrower and more intense compared to a sample annealed at 600 °C, which indicates an increase in the size of crystallites and an increase in the degree of structural ordering.

Annealing at 800 °C. Their high intensity and smaller width indicate the formation of a coarse-crystalline and well-ordered structure. At the same time, the intensity of MnSi peaks can decrease or change, which indicates a phase transformation and redistribution of the composition towards a thermodynamically more stable phase of Mn_5Si_3 at a given temperature. maximum crystallinity and a well-defined phase structure.

Thus, X-ray diffraction data reflect the consistent phase evolution of the material with an increase in the annealing temperature: from a weakly crystalline state (600 °C) to the formation of the MnSi phase (700 °C) and then to the predominance of the Mn_5Si_3 phase at 800 °C.

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

Surface morphology parameters Table 2.

№	Annealing, °C	Grain size, nm	RMS-roughness, nm
1	600	20–30	0,8
2	700	40–60	1,9
3	800	80–120	4,5

Table 2 presents the results of the study of the morphological characteristics of the surface of samples after annealing at different temperatures. The average grain size and the root mean square surface roughness (RMS) were used as analysis parameters.

It has been established that an increase in the annealing temperature is accompanied by a monotonous increase in the size of the grains. At a temperature of 600 °C (sample No1), the grain size is 20–30 nm, which corresponds to the formation of a fine-grained structure. An increase in temperature to 700 °C (sample No2) leads to the enlargement of grains to 40–60 nm, which indicates the activation of surface and volume diffusion processes.

At a maximum temperature of 800 °C (sample No3), further grain growth up to 80–120 nm is observed. A significant increase in their size indicates the development of coalescence and recrystallization processes, leading to the formation of a more pronounced grain structure.

A similar trend can be seen for the RMS roughness parameter. At 600 °C, the RMS value is 0.8 nm, which characterizes a comparatively smoothed surface. An increase in temperature to 700 °C is accompanied by an increase in roughness to 1.9 nm, and at 800 °C to 4.5 nm. The growth of roughness correlates with an increase in grain size and may be due to the development of the surface relief due to the enlargement of crystallites and the redistribution of the material during heat treatment.

Thus, a direct dependence of morphological parameters on the annealing temperature has been established: its increase leads to a consistent enlargement of grains and an increase in the RMS surface roughness. The results obtained confirm the decisive role of thermal activation of diffusion processes in the formation of surface morphology and are consistent with the ideas about temperature-induced grain growth in thin-film and implanted systems.

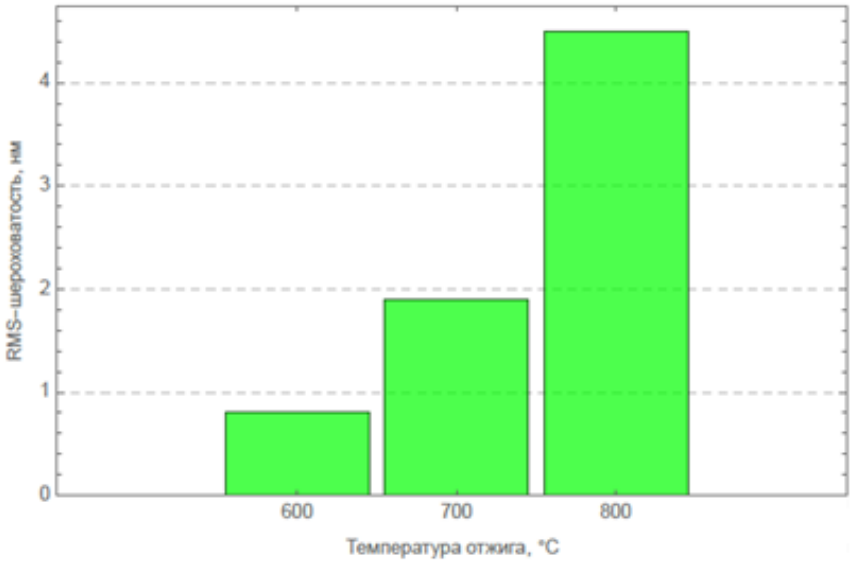

Fig. 2. AFM images of the surface after annealing at 600, 700 and 800 °C. (Description: Transition from smooth surface to granular and insular structure)

Figure 2 shows AFM images of the film surface after annealing at 600, 700, and 800 °C. The images clearly demonstrate the evolution of the surface morphology with an increase in the temperature of heat treatment, from relatively smooth to pronounced granular and further to an insular structure.
Annealed at 600 °C. The surface is characterized by a comparatively low roughness and a uniform texture. The relief is weakly pronounced, height differences are insignificant. Small evenly distributed grains or weakly pronounced structural inhomogeneities are observed. The surface looks solid, without breaks and large agglomerates, which indicates the preservation of the continuity of the film and the initial stage of crystallization.
Annealing at 700 °C. the surface becomes more developed and grainy. The grain structure is clearly manifested: the size of the grains increases, the boundaries between them become more distinguishable. The root mean square roughness increases, and the height differences increase. The morphology indicates the growth of grains and the recrystallization of the material. The film remains generally solid, but its surface is already noticeably heterogeneous.
Annealing at 800 °C. A pronounced islet structure is formed. The surface consists of separate elevated areas (islands) separated by low areas. The size of structural elements increases significantly compared to previous temperatures. Elevation differences are maximum, the roughness increases significantly. Such morphology may be associated with the processes of agglomeration, coalescence of grains, and partial film discontinuity due to thermal instability or redistribution of matter.

Thus, a series of AFM images demonstrates a consistent morphological transformation of the surface as the annealing temperature increases: from a smooth and relatively homogeneous structure (600 °C) to a granular structure (700 °C) and further to a developed insula structure (800 °C). This indicates the activation of diffusion processes, the growth of grains and a change in the mechanism of film formation during high-temperature treatment.
An increase in the degree of crystallinity leads to a decrease in the resistivity of films.

Resistivity Table 3.

№	Annealing, °C	ρ, Ω·cm
1	600	8×10^{-4}
2	700	3×10^{-4}
3	800	1.5×10^{-4}

Table 3 shows the values of the electrical resistivity of the samples as a function of the annealing temperature. The results obtained demonstrate a pronounced dependence of the electrophysical characteristics of the material on the conditions of heat treatment.
It has been established that at an annealing temperature of 600 °C, the resistivity is 8×10^{-4} Ω·cm.
An increase in the annealing temperature to 700 °C is accompanied by a significant decrease in resistivity to 3×10^{-4} Ω·cm.
At a maximum temperature of 800 °C, there is a further decrease in resistivity to 1.5×10^{-4} Ω·cm. The total reduction in resistance compared to a sample annealed at 600 °C is more than five times. This effect may be due to the enlargement of crystallites, a decrease in the density of grain boundaries, and the formation of a more thermodynamically stable phase with better conductive properties.

Thus, a steady trend of decreasing electrical resistivity with an increase in annealing temperature was revealed. The results obtained are consistent with the ideas about the effect of thermal activation on the processes of structural ordering, grain growth and phase transformations, which leads to a decrease in the dissipation of charge carriers and an improvement in the conductivity of the material.

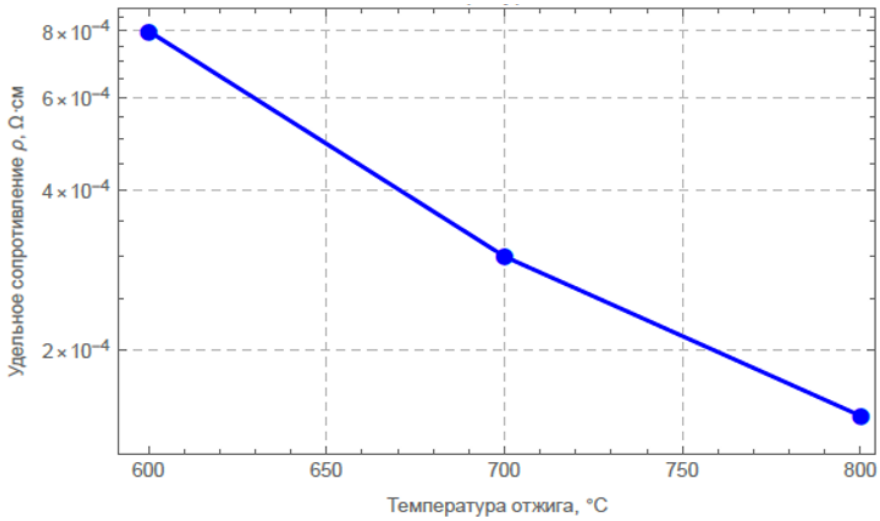

Fig. 3. Dependence of resistivity of thin films MnSi/Mn_5Si_3 on annealing temperature. (description: monotonic decrease in resistance with a rise in T)

Figure 3 shows the dependence of the electrical resistivity of thin films of the MnSi/Mn_5Si_3 system on the annealing temperature.

Along the x-axis, the annealing temperature T (°C or K) is plotted, along the ordinate axis (Y) is the resistivity ρ (for example, in μΩ·cm). The graph shows a monotonic decrease in resistance with an increase in annealing temperature.

In the area of low annealing temperatures, resistance is most important. This may be due to a high degree of structural disorder of the film, the presence of defects, grain boundaries, vacancies, as well as a possible amorphous or partially crystalline structure. In this range, charge transfer is limited by the intensive scattering of carriers on defects.

As the annealing temperature increases, there is a smooth and continuous decrease in resistivity. This indicates an improvement in the crystal structure of MnSi/Mn_5Si_3 films, a decrease in defect density, and a decrease in electron scattering. Annealing promotes recrystallization, grain growth, and the formation of a more orderly phase structure of manganese silicides.

In the region of higher annealing temperatures, the decrease in resistance may slow down, approaching a certain minimum value. This indicates that the structure of the film becomes more stable and close to the equilibrium state, and a further increase in temperature no longer leads to significant changes in the microstructure.

Thus, the figure illustrates a relationship characteristic of thin-film silicide systems: an improvement in structural perfection during heat treatment leads to a decrease in resistivity. The monotonic nature of the curve indicates the absence of abrupt phase transitions in the temperature range under consideration and the gradual formation of a more conductive phase.

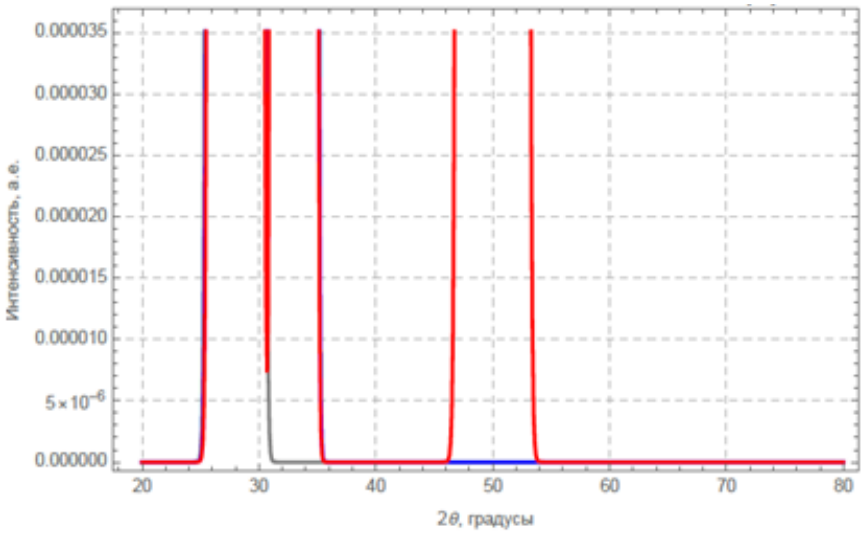

Fig. 4. Dependence of the average size of manganese silicide crystallites on the temperature of thermal annealing.

Fig. 4 shows the dependence of the average size of manganese silicide crystallites on the thermal annealing temperature. The graph shows a pronounced positive correlation between the processing temperature and the degree of structural enlargement of the formed phases.

At a temperature of 600 °C, silicide phases are not fixed, which indicates an insufficiency of thermal energy to initiate intensive diffusion processes and crystallization of manganese-silicon compounds. Consequently, the low temperature region is characterized either by an amorphous state or by the presence of ultrafine nuclei whose sizes do not exceed the nanometer range.

When the temperature rises to 700 °C, the formation of the MnSi phase is observed, accompanied by the growth of crystallites to 20–45 nm. The presence of a spread in values is due to the difference in implantation doses, but in general, a transition to a stable crystalline state is characteristic of this temperature. The formation of MnSi silicide indicates the activation of atomic diffusion and the completion of the nucleation stage with the transition to the crystallite growth stage.

A further increase in temperature to 800 °C leads to a change in the phase composition with the formation of Mn_5Si_3 and is accompanied by a significant enlargement of crystallites to 40–60 nm. The growth of grain sizes at this temperature is intensive, which may be due to the intensification of coalescence processes, a decrease in the density of grain boundaries, and a decrease in the internal defect of the structure. In addition, the formation of a new phase indicates a redistribution of the components of the system and a transition to a more thermodynamically stable state.

Thus, the dependence presented in Fig. 4 is monotonically increasing and reflects the typical temperature-induced evolution of the structure: the nucleation of the silicidal phase, its growth and subsequent phase transformation with further enlargement of crystallites. The results obtained confirm the decisive role of the annealing temperature in controlling the size of crystallites and the phase composition of manganese silicide compounds.

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 the crystalline phases of MnSi and Mn_5Si_3, which

is accompanied by an improvement in electrical characteristics. At the same time, the increase in surface roughness is a consequence of the coalescence of silicidal grains.

Conclusion

In this work, a comprehensive study of the processes of formation, structural features and morphology of thin films of manganese silicides obtained by ion implantation of manganese into single-crystal silicon with subsequent thermal annealing is carried out. The main conclusions arising from the experiments can be formulated as follows:

- **Phase formation:** at low implantation doses (1×10^{15} cm^{-2}) and annealing temperatures up to 600 °C, silicidal phases are predominantly in an amorphous or nanocrystalline state, which is confirmed by the absence of pronounced X-ray diffraction peaks. An increase in the implantation dose to 1×10^{16} cm^{-2} and the annealing temperature to 700–800 °C leads to the formation of stable crystalline phases of MnSi and Mn_5Si_3, which indicates the predominance of the diffusion mechanism of phase formation under conditions of high local concentration of manganese;

- Crystal structure and dimensions of crystallites: X-ray diffraction analysis showed an increase in the size of crystallites from 20–30 nm at 700 °C to 50–60 nm at 800 °C, which indicates an increase in the degree of crystallinity and a decrease in the concentration of defects with an increase in the annealing temperature. The formation of crystalline phases is accompanied by relaxation of internal mechanical stresses and a decrease in the amorphous component of the layer, which has a positive effect on the structural homogeneity of the films;

-Surface morphology: atomic force microscopy methods have shown that crystallite growth is accompanied by a significant change in surface morphology: with an increase in the annealing temperature, the RMS roughness increases from ~0.8 nm at 600 °C to 4–5 nm at 800 °C. Surface morphology control by changing annealing temperatures and implantation doses allow for the targeted formation of functional layers with specified topographic characteristics;

-Electrical properties: resistivity measurements showed a decrease in resistivity from 8×10^{-4} Ω·cm at 600 °C to ~$1–2\times10^{-4}$ Ω·cm at 800 °C, which correlates with an increase in the size of crystallites and an improvement in crystal order. A decrease in resistance indicates a decrease in the scattering of charge carriers at defects and grain boundaries, which is important for the use of these films in silicon-compatible electronic devices;

- Technological significance: the results obtained demonstrate the possibility of controlled formation of thin films of manganese silicides with specified structural, morphological and electrical characteristics by precise selection of ion implantation and thermal annealing parameters. This approach can be used to create contact and functional layers in micro- and nanoelectronics, including silicon devices with low contact resistance and improved thermal stability;

Thus, the study confirms the effectiveness of the ion implantation method followed by thermal annealing for the synthesis of thin films of MnSi and Mn_5Si_3 with controlled properties. Dose and annealing temperature control optimizes phase composition, morphology, and electrical parameters,

which opens up prospects for the use of these materials in modern silicon-compatible electronic and spintronic devices.

The results obtained demonstrate the possibility of controlled formation of thin films of manganese silicides with specified structural and morphological characteristics by varying the parameters of ion implantation and thermal annealing, which is of interest for the development of silicon-compatible materials for micro- and nanoelectronics.

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