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INVESTIGATION OF THE INFLUENCE OF NICKEL ON THE BEHAVIOR OF THERMAL DONORS IN SILICON

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It has been established that doping silicon with nickel in the temperature range T = 1000÷1250°C makes it possible to almost completely suppress the generation of thermal donors during thermal annealing in the temperature range T = 100÷700°C. It has been established that impurity nickel atoms form clusters and precipitates in silicon that absorb oxygen atoms. The most effective technological method for producing silicon with stable electrophysical parameters has been proposed. The proposed method for gettering uncontrolled impurity atoms can be used in the production of various electronic devices, especially in the development of efficient silicon-based solar cells.
Keywords: *Diffusion; Cluster; Nickel; Oxygen; Thermal donor; Silicon*
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INTRODUCTION

Impurity nickel atoms, unlike other elements of transition groups, have not only the highest diffusion coefficient in silicon, but also high solubility in the crystal lattice ($N_{Ni} \sim 10^{18}$ cm^{-3}) [1,2]. However, the maximum concentration of electroactive impurity nickel atoms is less than ~ 0.1% of the total solubility of atoms at a given temperature, which means that the bulk of impurity nickel atoms in silicon are in an electrically neutral state.

The authors in [3-8] showed that some of the nickel atoms located in the interstices of the silicon crystal lattice can form impurity clusters and precipitates. The structure, size and distribution of the formed clusters are mainly determined by the defectiveness of the original silicon, the conditions of diffusion doping, the cooling rate after diffusion annealing, as well as the temperature and time of additional thermal annealing.

This work is devoted to the study of the electrophysical parameters of silicon doped with nickel impurity atoms at various temperatures, as well as its behavior during additional heat treatments in a wide range of temperatures (in which intensive generation of thermal donors occurs) and time.

MATERIALS AND METHODS

Single-crystalline silicon, grade KDB-1, grown by the Czochralski method of p - type conductivity with a boron concentration of $N_B \sim 2\cdot10^{15}$ cm^{-3} was chosen as the starting material. The size of the samples was 1x5x10 mm^3. The concentration of residual oxygen in the studied silicon samples was $N_{O2} \sim 6\cdot10^{17}$ cm^{-3}, the dislocation density S ~ 10^3 cm^{-2}.

Ni diffusion was carried out from a layer of pure metallic nickel deposited on the surface of a silicon sample, both in open air and in evacuated quartz ampoules up to P ~ 10^{-6} atm. in the temperature range T = 1000÷1200°C. The diffusion time was chosen in such a way as to ensure a uniform distribution of nickel atoms in the volume of silicon. In separate ampoules, control silicon samples, without nickel admixture, were annealed under similar conditions in order to evaluate the effect of diffusion annealing on the electrical parameters of the original silicon samples. After diffusion annealing, the silicon samples were ground off on all sides by 5÷10 μm to remove the surface layer enriched in nickel, and then etched by 5÷7 μm with an alkaline etchant. Mechanical and chemical treatment of all samples was carried out under identical conditions. The electrical parameters of the samples were measured by the Van der Pauw method using an ECOPIA HALL HMS-3000 installation.

Table 1 shows the electrical parameters of silicon samples before and after diffusion of nickel impurity atoms at various temperatures and a duration of 120 minutes. As can be seen from the experimental results, samples doped with impurity nickel atoms at T = 1220 °C practically retain their original electrical parameters, that is, no significant changes in parameters are observed in them, and in control samples annealed under the same conditions, but without impurity nickel atoms, resistivity increases 30 times

These experimental results show that at this thermal annealing temperature T = 1220 °C the generation of thermal donors occurs, bound by oxygen atoms in the silicon lattice, and the concentration of thermal donors reaches ($N_d > 2\cdot10^{15}$ cm^{-3}), which is in good agreement with the results of the work [9-12].

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As can be seen from Table 1, with a decrease in the diffusion annealing temperature, the resistivity of the control samples increases significantly (by 3 and 4 orders of magnitude) and reaches ρ = 4·10^4 and ρ = 2·10^5 Ω·cm at annealing temperatures T = 1170 and 1120°C, respectively. At the same time, in samples doped with impurity nickel atoms, the electrical parameters practically do not change. These experimental results make it possible to determine the optimal conditions for doping silicon with nickel, which almost completely suppress the generation of thermal donors.

Table 1. Electrical parameters of control and nickel-doped silicon samples at different diffusion temperatures

no.	Before diffusion			Diffusion mode			After diffusion		
	Type conductivity	ρ, Ω·cm Resistivity	μ, cm^2/V·s Mobility	T,°C Temperature	t, min Time	Impurity / Control	Type conductivity	ρ, Ω·cm Resistivity	μ, cm^2/V·s Mobility
1	*p*	9.8	317	1220	120	*Ni*	*p*	9.2	354
	p	10.3	319		120	*Ni*	*p*	8.3	325
	p	10.1	285		120	*Control*	*n*	295	1075
2	*p*	9.5	355	1170	120	*Ni*	*p*	9.1	370
	p	10.4	327		120	*Ni*	*p*	9.75	335
	p	10.2	332		120	*Control*	*n*	4·10^4	1052
3	*p*	10.8	335	1120	120	*Ni*	*p*	9.2	279
	p	9.6	322		120	*Ni*	*p*	9.9	314
	p	9.3	325		120	*Control*	*i*	2·10^5	505
4	*p*	10.2	270	1070	120	*Ni*	*p*	9.75	315
	p	10.5	290		120	*Ni*	*p*	9.42	306
	p	9.7	297		120	*Control*	*p*	11.5	295
5	*p*	40.3	338	1170	120	*Ni*	*p*	45.5	340
	p	39.6	345		120	*Control*	*n*	1.5·10^3	1130

Ni – nickel doped silicon samples, C – silicon control samples

The electrical parameters of the samples doped with nickel impurity atoms and the control samples annealed at the annealing temperature T = 1070°C do not differ significantly. This may be due to the fact that the concentration of thermal donors at this temperature and annealing time will be noticeably lower than the concentration of holes in the original samples. The results showed that in this case, the control silicon samples change their type of conductivity, that is, they become n - type with a resistivity of ρ ~ 1.5·10^3 Ω·cm.

From an analysis of the research results, it was established that the influence of impurity nickel atoms on the generation of thermal donors does not depend on the conditions of nickel diffusion (in air or evacuated quartz ampoules).

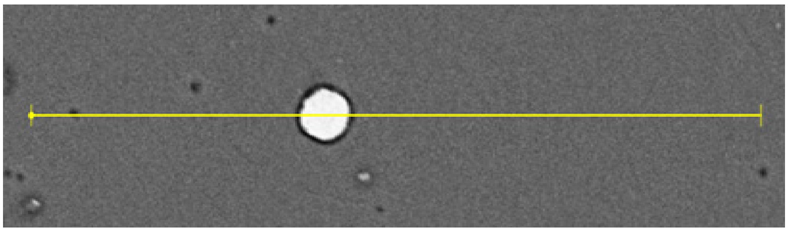
In this regard, at the next stage of work, higher-resistance silicon samples of *p* and *n* type conductivity with different resistivity values were used as the starting material (Table 2).

Table 2. Technological parameters of the nickel alloying process.

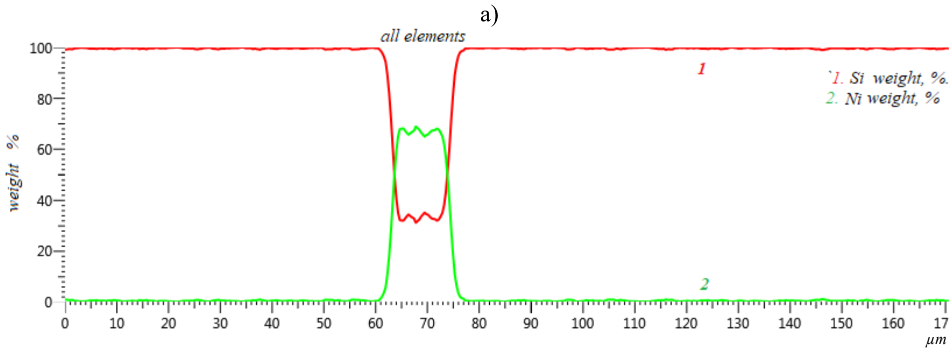
Parameters of the original silicon		Ni diffusion temperature	Diffusion time	Heat treatment parameters after diffusion		Changing parameters after heat treatment	
Type conductivity	ρ, Ω·cm	T, °C	t, min	T, °C	t, hour	ρ, Ω·cm	τ, %
	Resistivity	Temperature	Time	Temperature	Time	Resistivity	Life time change
n	≤ 10	1100÷1150	15÷20	100÷700	t=1÷20	11÷13	does not change
n	≤ 40	1060÷1100	25	100÷600	t=1÷20	41÷46	+ 0 ÷ + 12
n	≤ 100	1050	30÷35	100÷500	t=1÷20	97÷105	+ 0 ÷ + 16
p	≤ 10	1150÷1100	15÷20	100÷700	t=1÷20	8÷12	little changes
p	≤ 40	1100÷1050	20÷25	100÷700	t=1÷20	43÷48	+16 ÷ +19
p	≤ 100	1050÷1000	30÷35	100÷500	t=1÷20	97÷106	9÷16

Table 2 shows the main technological conditions for doping with nickel impurities, under which the original electrical and recombination parameters of silicon are preserved after the diffusion process and after subsequent thermal annealing.

It has been established that the electrical and recombination parameters of silicon samples with nickel clusters remain unchanged during additional thermal annealing for a time t = 1÷20 hours in a wide range of heat treatment temperatures (T = 100÷700°C).



a)

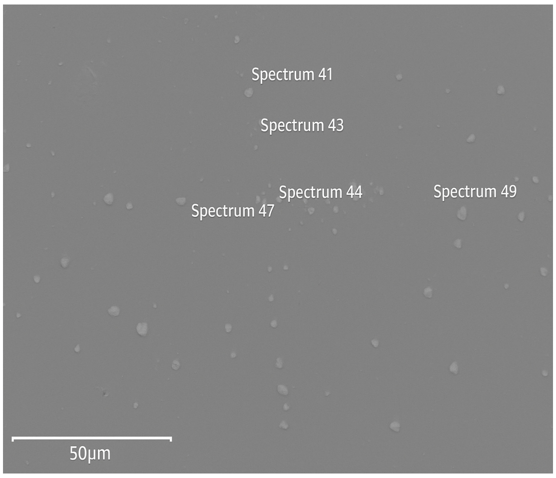


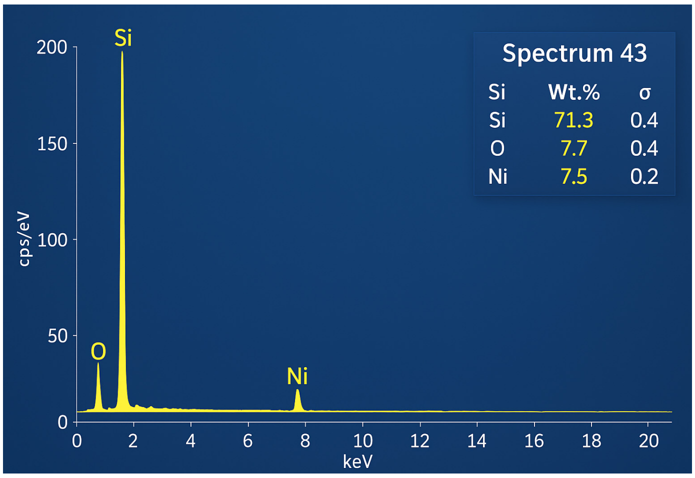
b)

Figure 1. Distribution of nickel atoms on the surface of silicon samples
a) clusters of nickel atoms on the silicon surface, b) elemental composition of silicon doped with impurity nickel atoms

Analysis of the research results showed that electrically neutral nickel atoms in silicon are involved in the formation of clusters (Fig. 1), the size of which is on average 1÷10 µm and depends on the diffusion temperature and the cooling rate after diffusion annealing.

The composition of such clusters was studied using energy-dispersive X-ray spectroscopy (EDS). In Fig. 2 presents the results of these studies.





a) b)

Figure 2. a) image of the surface, and b) elemental analysis of the surface of silicon doped with impurity nickel atoms at the "Spectrum 43" point

As can be seen from Table 3, the clusters consist of silicon, nickel and oxygen atoms. On average, the content of elements in them is: silicon atoms - 60%, nickel atoms - 3%, oxygen atoms - 11%. Based on these research results, it can be argued that impurity nickel atoms getter oxygen atoms when forming clusters. This means that clusters are a region of silicon enriched in nickel and oxygen atoms.

As is known from the literature, oxygen atoms in silicon are a source of thermal donors and also act as recombination centers [13]. This behavior is clearly manifested in control silicon samples (without nickel impurities). Thus, it can be assumed that electrically neutral nickel atoms form clusters that capture and retain a significant concentration of oxygen atoms and other uncontrolled impurities, and suppress the generation of thermal donors, which in turn ensures the stability of electrical parameters and recombination properties during various heat treatment processes [14-18].

Table 3. Result of elemental analysis of a sample of silicon doped with nickel atoms!

Element	Type line	Weight, %	σ, %	Atomic, %
Si	K series	71,3	0,4	59,9
O	K series	7,7	0,4	11,3
Ni	K series	7,5	0,2	3,03

CONCLUSIONS

From the analysis of the research results, it can be stated:

1. Impurity nickel atoms in the silicon lattice are mainly in electrically neutral states, easily forming clusters and precipitates.
2. Clusters of impurity nickel atoms can act as active getter centers for oxygen atoms and other impurities in silicon, thereby significantly suppressing the generation of thermal donors and other recombination centers in the silicon lattice.
3. The experimental results obtained show that the presence of impurity nickel atoms in silicon leads to stabilization of the initial electrical parameters of the samples, regardless of the type of conductivity and the concentration of the initial impurity boron and phosphorus atoms.
4. The optimal thermodynamic conditions for obtaining silicon samples with clusters of impurity nickel atoms and electrical parameters that are stable during heat treatment have been determined.

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ДОСЛІДЖЕННЯ ВПЛИВУ НІКЕЛЮ НА ПОВЕДІНКУ ТЕПЛОВИХ ДОНОРІВ У КРЕМНІЇ

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Встановлено, що легування кремнію нікелем в інтервалі температур *T* = 1000÷1250 °C дозволяє практично повністю придушити генерацію термодонорів під час термічного відпалу в інтервалі температур *T* = 100÷700 °C. Встановлено, що домішкові атоми нікелю утворюють у кремнії кластери та осади, які поглинають атоми кисню. Запропоновано найбільш ефективний технологічний спосіб отримання кремнію зі стабільними електрофізичними параметрами. Запропонований метод гетерування неконтрольованих домішкових атомів може бути використаний у виробництві різних електронних пристроїв, особливо при розробці ефективних сонячних елементів на основі кремнію.

Ключові слова: *дифузія; кластер; нікель; кисень; термодонор; кремній*