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Investigation of Temperature Sensors Based on Si <P, Ni>

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The paper presents the investigation results of the characteristics of a new type of temperature sensor based on silicon nanoclusters of nickel atoms in extreme conditions. It is shown that such sensors have high sensitivity and speed; they can successfully operate at elevated radiation and vibration.

Keywords: Thermal sensitivity, Speed, Nanostructure, Nanoclusters, Microprobe analysis.

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1. INTRODUCTION

Modern electronic temperature sensors of different types based on semiconductor structures and alloys do not allow to perform a continuous temperature control of the objects in conditions of increased radiation and vibration. In order to improve automated systems and obtain more reliable and timely information on the state of the objects, which are in various extreme conditions, it is necessary to develop more sensitive, high-speed temperature sensors with stable parameters allowing temperature control. Currently, devices with sufficiently complex electronic amplifiers, which are not suitable for use in conditions of increased radiation and vibration, are used to control and measure temperatures of the objects. High rated resistance, low energy consumption, when utilized in various environments, high operation speed and sensitivity and also long service life with stable parameters are the key technical requirements to temperature sensors. A continuous temperature control of the objects in corrosive media without the use of additional electronic amplifiers is one of the urgent problems of modern electronics.

From this point of view, utilization of semiconductor nanomaterials, whose parameters should possess high sensitivity to temperature, is of great interest. It is also necessary to pay attention to the fact that the use of modern technologies of formation of the temperature sensor materials by the high-temperature diffusion method does not allow to obtain the material nanostructured over the whole volume.

2. EXPERIMENTAL TECHNIQUE

We have developed a new method of doping of silicon with impurities – the so-called low-temperature diffusion, allowing to form nanoclusters of impurity atoms with necessary parameters. The essence of this method lies in implementation of step-by-step diffusion with a certain heating rate and exposure at strictly fixed time. Nickel was chosen as a doping impurity atom, since its solubility and diffusion coefficient are more optimal for the formation of nanoclusters with the required parameters. The structure, composition and distribution of nanoclusters both on the surface and in the bulk of the crystal are studied by the method of infrared microscope MIC-5 and microprobe analysis (JOIL). Concentration of nanoclusters was controlled by the temperature of each diffusion stage. We have obtained the silicon samples with concentration of nanoclusters of $N \sim 10^{15}$ cm^{-3} and resistivity of $\rho \sim 10^4 \div 10^5$ Ohm·cm at $T = 300$°K. These values of the sample resistivity provided not only high sensitivity of the temperature sensor, but also the possibilities of their use for remote temperature monitoring of the objects (up to 1 km) in extreme conditions. Analyzing the results obtained in the conducted studies, one can conclude that parameters of the developed temperature sensors are significantly larger than those of existing analogues.

Since the main aim of this work was to create a new class of heat-sensitive structures, which contain nickel nanocrystals in silicon with reproducible and stable with respect to different external conditions parameters, we have chosen the simplest way of chemical deposition of Ni film on the Si surface from the solution and subsequent low-temperature diffusion of Ni atoms. Chemical deposition of Ni was carried out in order to create the ohmic contacts. To improve adhesion, the plates before nickel plating were polished in the micropowder M-9 and treated in the hydrofluoric acid solution for removing oxide ($HF : H_2O$ in the ratio of 1 : 1). Electrolyte for nickel plating was prepared by the following formulation per 100 ml of the solution: nickel chloride ($NiCl_2$) – 2.1 g; sodium hypophosphite (NaH_2PO_2) – 2.4 g; tri-sodium citrate – 4.5 g; ammonia chloride (NH_4Cl) – 3 g; aqueous ammonia (25 %) – 5 g.

At that, multistage diffusion of impurity atoms provides for the formation of inherent clusters as well as clusters with participation of structural defects and atoms of the lattice matrix. Based on the developed low-temperature doping technique, we succeeded to create not only favorable conditions for the formation of the clusters of impurity Ni atoms in Si, but also provide for the possibility of self-organized ordering of arrangements in the crystal bulk (Fig. 1). As seen from Fig. 1, ordering of the clusters of impurity Ni atoms in the Si lattice occurs over the whole crystal volume.

It has been created an entirely new class of the universal sensors of physical quantities with improved degradation properties and parameter stability, low power consumption and diminutiveness, absence of additional gain circuits providing ease of operation, which are superior in the threshold sensitivity and operation speed the similar existing sensors that is associated with their functioning on the basis of the principally new physical phenomena quite sensitive to external influence.

To create the temperature sensors, silicon doped with nickel was cut in the form of a crystal with geometry of 1.5 × 1.5 × 1.5 mm. Based on a special technology, we have

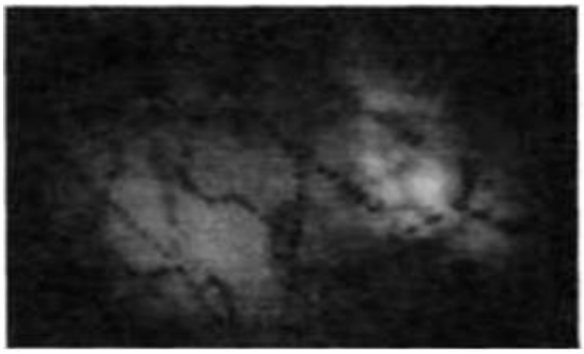

Fig. 1 – Self-organized ordered clusters of Ni atoms in the Si crystal lattice (obtained on the IR microscope)

created the ohmic contacts on the crystal, which provide a current ramp in a wide range of the temperature sensor electric field. When selecting a sealant, the main attention is focused on its strength, watertightness and resistance to radiation, vibration and influence of gaseous media. According to requirements to sealants, we have developed the optimal formulations of caulking compounds, as the basis of which resins ED-20 with maleic anhydride and triethanolamine were taken as hardeners. The choice of maleic anhydride and triethanolamine is conditioned by the fact that maleic anhydride allows to obtain compounds with a low initial viscosity and a great vitality. The choice of triethanolamine is explained by its low toxicity. The formulation of the epoxy caulking compound D-2 is represented in Table 1.

From the point of view of prevention of mechanical stresses, compound shrinkage in polymerization is of a great importance. Therefore, we have conducted an exothermal heating at different temperatures during polymerization: at $T = 80$ °C during 4 hours, at $T = 120$ °C during 12 hours, at $T = 140$ °C during 24 hours. This allowed to minimize the formation of mechanical stresses during hardening of epoxy resins. Red lead (RL) paint was used as filler.

Power consumption of temperature sensors is equal to 0.01 W.

Table 1 – Composition of the epoxy caulking compound D-2

Composition, weight parts					
Resin		Hardener		Filler	
ED-20	100	ma	2.28K	RL	1.5-1.8 g

3. EXPERIMENTAL

3.1 Investigation of the diffusion characteristics of nickel in *n*- and *p*-type silicon

The diffusion coefficient of Ni in Si in the temperature range of 450-800 °C can be described by the function $D = 0.1\exp(1.9/kT)$. The value of the diffusion coefficient of Ni is equal to 10^{-4}-10^{-5} cm^{2}s^{-1} [1] and at the temperature of 1350 °C it makes $9\cdot10^{17}$ cm^{-3} [2]. In Fig. 2 we show the distribution of Ni throughout the Si depth.

It is seen from Fig. 2 that the profile consists of two regions: the first region – near-surface with 25 μm in length – is characterized by a sharp 2-3-fold decrease in concentration; the second one – bulk – is characterized by a uniform distribution of the impurity in the sample volume. We should note that uniform level of Ni concen-

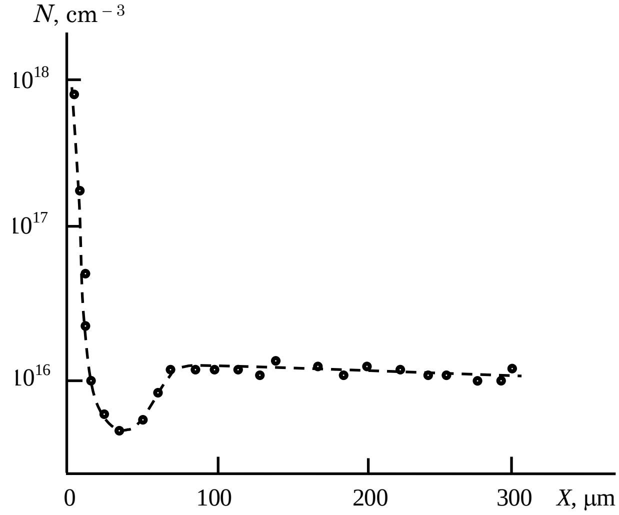

Fig. 2 – Distribution of Ni concentration in Si over depth

tration in the volume does not depend on the annealing time (15 min-90 hours). It is also established that change in the concentration of the initial small impurities within the range of 10^{13}-10^{19} cm^{-3} in Si of both *n*- and *p*-types does not significantly influence on the value of the diffusion coefficient, which weakly depends on temperature. Diffusion activation energy of Ni is equal to 0.47 eV. Temperature dependence of the diffusion coefficient of Ni (Fig. 3) can be described by the expression

$$D_{Ni} = 2.1\cdot10^{-3}\exp\left(-\frac{0.47}{kT}\right). \qquad (3.1)$$

Temperature dependence of Ni solubility (Fig. 4) has a retrograde character, at that the maximum value of its solubility at $T = 1310$ °C is equal to $7\cdot10^{6}$ cm^{-3}. This dependence up to the maximum value can be described by the expression

$$D_{Ni} = 1.4\cdot10^{25}\exp\left(-\frac{2.3}{kT}\right). \qquad (3.2)$$

The values of the diffusion coefficients and activation energies indicate the interstitial diffusion of Ni atoms in Si. In Table 2 we present the electrical parameters of Si obtained before and after doping with nickel at different temperatures.

A substantial change in the conductivity of the samples occurs during introduction of Ni into *n*-Si with the resistivity of $\rho \sim 10$ Ohm·cm. In order to obtain the sam-

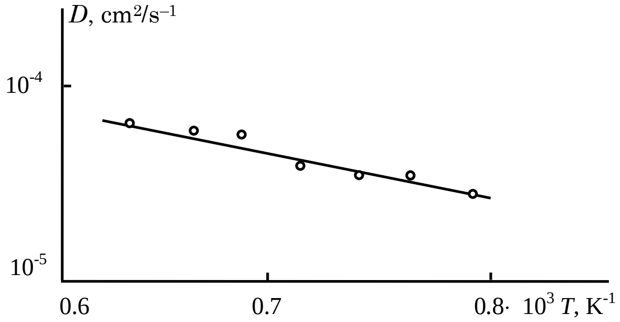

Fig. 3 – Temperature dependence of the diffusion coefficient of Ni in Si

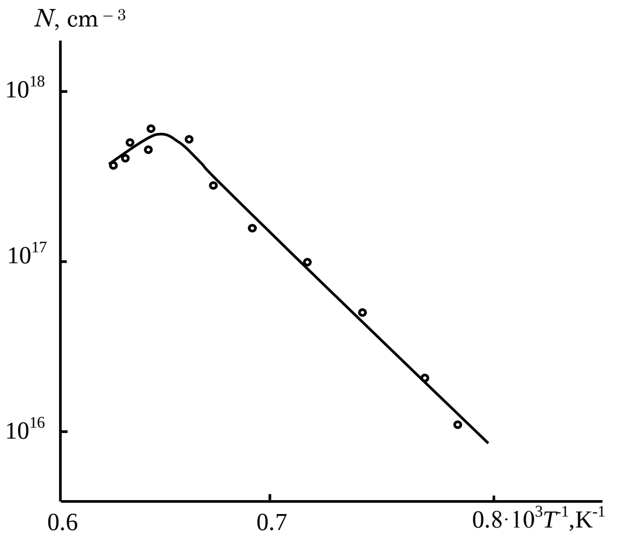

Fig. 4 – Temperature dependence of Ni solubility in Si

Table 2 – Electrical parameters of silicon

Before diffusion		Diffusion mode		After diffusion (the type is not changed)		
type	ρ, Ohm·cm	T, K	t, time	ρ, Ohm·cm	n, p, cm^{-3}	μ, cm^{2}V^{-1}s^{-1}
n	10	1523	1	65.0	$7.5\cdot10^{13}$	1252
n	10	1473	1	19.7	$3.6\cdot10^{14}$	1372
n	10	1423	1.5	9.4	$4.5\cdot10^{14}$	1228
n	10	1373	2	8.6	$6.4\cdot10^{14}$	1209
n	10	1323	2	9.1	$5.6\cdot10^{14}$	1233
n	40	1523	1.5	$1\cdot10^5$	$4.8\cdot10^{14}$	2010
n	40	1423	1.5	65.0	$6.0\cdot10^{13}$	1400
n	200	1523	1	$6\cdot10^4$	$9.0\cdot10^{11}$	261
n	200	1473	1.5	10^5	$6.0\cdot10^{10}$	2017
n	200	1373	1.5	$3.5\cdot10^2$	$1.98\cdot10^{13}$	1292
n	200	1273	2	220.0	$1.93\cdot10^{13}$	1468
p	10	1523	1.5	11.8	$2.15\cdot10^{15}$	267
p	10	1373	1.5	10.9	$1.95\cdot10^{15}$	288
p	30	1373	1.5	31.0	$5.0\cdot10^{14}$	376

ples with different resistivity, it is necessary to use n-Si with resistivity of $\rho \sim 40 \div 60$ Ohm·cm. Analysis of these results shows that nickel in silicon forms acceptor levels with maximum concentration of 10^{14} cm^{-3}.

Based on the experimental data on investigation of the influence of annealing conditions and parameters of the initial material on the properties of compensated silicon, we have developed the production technology of the n- and p-type samples with the specified parameters, which consists in the following:

– nickel diffusion occurs from the nickel layer deposited on the chemically etched silicon surface in an atmosphere of an inert gas or in air;

– cooling rate after diffusion should be not more than 100-120 deg/s;

– the optimal temperature of nickel diffusion is equal to 1100 °C, and the diffusion annealing time makes 1.5-2 hours;

– n-type Si should be used as the initial material;

– the above listed diffusion conditions are quite applicable for the samples of thickness up to 1 mm, area of $S = 2$ cm^2; after diffusion it is necessary to remove from the surface a layer of thickness of ~ 40-50 μm;

– these technological conditions are also suitable for the production of compensated samples with the studied impurities by ion implantation. At that, the ion energy should be not more than 40-60 keV.

3.2 Investigation of the influence of γ-radiation on the operating parameters and sealing states of temperature sensors

Temperature sensors underwent γ-radiation with doze of $D = 3000$ R/s (1 R = $2.57976\cdot10^{-4}$ C/kg) at $T = 70$ °C. Parameters of temperature sensors, states of the ohmic contacts as well as the sealing state were studied after each radiation stage under the same conditions. Operation speeds of temperature sensors were determined by their dumping in the temperature range from 20 °C to 100 °C (setting time) and from 100 °C to 20 °C (recovery time). Table 3 shows the influence of γ-radiation (Co^{60}) on the operating parameters of temperature sensors based on Si<P, Ni>.

It is established that before irradiation sensitivity of temperature sensors (B) and their operation speeds are sufficiently high and almost constant up to the radiation dose of $D = 10^8$ R for temperature sensors with the rated resistance of $R = (1 \div 5)10^5$ Ohm. At higher radiation doses $D > 10^8$ R, parameters become gradually worse, and for $D \sim 10^9$ R they decrease by 25÷30 %. For temperature sensors with higher rated resistance of $R \sim 10$ Ohm·cm, deterioration of their parameters starts at $D = 5\cdot10^8$ R. It is established that the higher concentrations of impurity atoms, the larger resistance of the temperature sensor and its radiation resistance.

Table 4 displays the influence of γ-radiation (Co^{60}) on the sealing state of temperature sensors on the basis of Si<P, Ni>. Resistance of the developed temperature sensor is equal to $R = 10^4 \div 10^6$ Ohm. Temperature sensors possess two reliable contacts and sealing, which completely protects them from moisture, dust and provides resistance to different gaseous media.

It is established that state of the electric contact (reliability and ohmicity) are almost constant in the studied ranges of radiation doses.

Thus, temperature sensors based on silicon with nanoclusters of nickel atoms possess a sufficiently high sensitivity, operation speed and radiation resistance. It was studied that γ-radiation (Co^{60}) does not influence the operating parameters and sealing state. Up to the radiation

Table 3 – Operating parameters of temperature sensors after irradiation

Radiation dose							
10^7		$5\cdot10^7$		10^8		$5\cdot10^8$	
B, K	τ, s	B, K	τ, s	B, K	τ, s	B, K	τ, s
6750	13	6700	13	6650	13	6600	13
6950	13	6900	13	6850	13	6800	13
7150	13	7100	13	7050	13	7000	13

Table 4 – Sealing state of temperature sensors under the influence of γ-radiation

Radiation dose			
10^7	$5\cdot10^7$	10^8	$5\cdot10^8$
No changes	Light is changed	Began to crumble	Is crumbled

dose of $D = 10^8$ R parameters of temperature sensors are virtually unchanged irrespectively of their rated resistance. A relative change in the resistance (R / R_0) of the temperature sensor is observed for high radiation doses (see Fig. 5).

In Fig. 6 we show the relative variation of the heat sensitivity on the radiation dose of temperature sensors with different rated resistances. As seen from the figure, the critical radiation dose (D_{cr}), at which an appreciable decrease in the heat sensitivity starts, is shifted to higher doses with increasing rated resistance of temperature sensors.

These results show that temperature sensors based on Si<P, Ni> can be used in conditions with a high level of radiation. Operation speed of temperature sensors under irradiation is changed slightly. Temperature sensors operate in the temperature range of $T = -60$ °C÷120 °C and provide temperature control of the objects located at distance up to 1 km. Coefficient of their heat sensitivity is almost 1.5-2 times larger than that of existing temperature sensors.

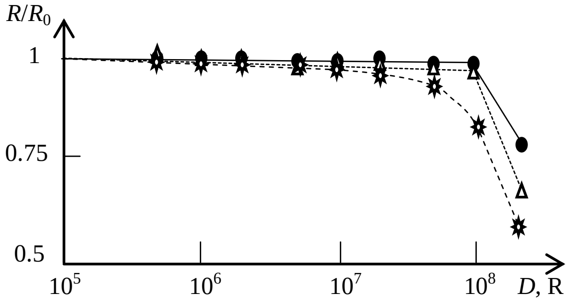

Fig. 5 – Relative variation of the temperature sensor resistance on the X-ray irradiation dose

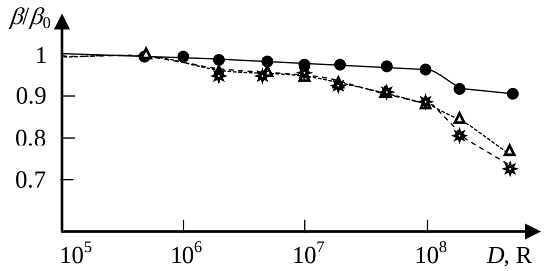

Fig. 6 – Relative variation of the heat sensitivity of temperature sensors β/β_0 with different rated resistances on the X-ray irradiation dose

3.3 Investigation of the influence of vibration on the operating parameters of temperature sensors

Parameter reliability tests in conditions of increased vibration are of practical interest. In this connection, the next stage of the study was carried out in conditions of high vibration.

In order to reproduce vibrations in practice in industrial tests and scientific research, they use the vibration machines allowing to conduct the tests on vibration stability of the objects, investigate the design elements under the specified parameters, as well as calibrate, control and assess them.

In order to estimate the availability of the developed temperature sensors under vibration, we have performed the study of its influence on the state and physical properties of the temperature sensor. Mechanical vibration plant VU-15 with operating frequency in the range from 1 to 200-300 Hz was used. It is established that under conditions of vibration during 12 hours the key parameters of temperature sensors are almost unchanged; however, we should note that sealing quality remains well enough up to the frequency of 350 Hz. With increasing frequency more than 450 Hz, sealing is almost destroyed. In Table 5 we present the influence of vibration on the operating parameters and sealing state of temperature sensors based on Si<P, Ni>. Table 5 shows that the developed temperature sensors can be used in conditions of increased radiation and mechanical vibration.

Table 5 – Influence of vibration on the operating parameters and sealing state of temperature sensors

Frequency, Hz							
300		400		500		600	
B, K	τ, s	B, K	τ, s	B, K	τ, s	B, K	τ, s
6750	14	6700	14	6650	14	6600	14
6950	14	6900	14	6850	14	6800	14
7150	14	7100	14	7050	14	7000	14
No changes		Light is changed		Began to crumble		Is crumbled	

4. RESULTS AND DISCUSSION

The tests of finished temperature sensors during a long time (more than 3 years) at different temperatures ($T = -60$°÷100 °C) showed a sufficient reliability of the sealing and ohmic contacts and also the parameter stability of the produced temperature sensors.

It is experimentally established that 2 acceptor levels with energies of $E_v + 0.2$ eV and $E_c - 0.4$ eV are formed during diffusion of Ni in Si. The maximum concentration of electrically active atoms is equal to $-4 \cdot 10^{14}$ cm^{-3}. The optimal modes of low-temperature doping of Ni in Si are established for the formation of temperature sensors with the maximum sensitivity and stability of the characteristics. It is shown that thermal annealing of Si samples doped with Ni leads to the formation of electrically neutral complexes NiO_x and increase in the stability of the temperature sensor parameters.

Implementation of a new low-temperature nickel doping method developed in this work allows to obtain nanoclusters of nickel atoms in the doped volume with self-organization effect of impurity clusters. According to the performed studies, temperature sensors based on silicon with nickel nanoclusters are developed for the first time.

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