

## STUDY OF SILICIDE FORMATION IN LARGE DIAMETER MONOCRYSTALLINE SILICON

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To study the formation of silicides, dislocation-free ingots of single-crystalline silicon with a diameter of 65÷110 mm, grown by the Czochralski method, were used. When studying such silicon samples using electron microscopy, small-angle scattering of CO<sub>2</sub> laser radiation, three types of defects were identified: swirl defects, impurity micro inclusions and impurity clouds. It has been shown that silicide inclusions with sizes of 8-20 μm are formed in the near-surface layer of doped silicon, and they decrease linearly deeper into the crystal. The electrical parameters of semiconductor chromium silicide were determined: resistivity 1800 μOhm·cm, thermopower coefficient 180 μV/k, Hall constant  $1.2 \cdot 10^{-2} \text{ cm}^3/\text{Kl}$ , hole concentration  $6 \cdot 10^{19} \text{ cm}^{-3}$ , charge carrier mobility  $18.6 \text{ cm}^2/\text{V}\cdot\text{s}$ , band gap  $(0.29 \pm 0.02) \text{ eV}$ .

**Key words:** Dislocation-Free; Etching; Defects; Swirl; Heterogeneity; Dissolution; Super-Large Circuits; Hardening; Inclusion; Extrapolation

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### INTRODUCTION

Currently, an urgent problem is the study of physical processes occurring both in the bulk and on the surface, and in the near-surface layers of a large-diameter single-crystal semiconductor, in particular, silicon in the process of diffusion doping with impurities that create deep levels (DL) [1-6]. First of all, the need for these studies is due to the fact that in the process of diffusion doping of a semiconductor material—silicon—it is possible to obtain compensated materials with specified electrophysical, photoelectric and optical properties [7-12].

In diffusion-doped silicon crystals, a near-surface layer with a thickness of about ten microns is formed with an impurity concentration that exceeds their solubility by 2÷4 orders of magnitude, and with other electrophysical parameters, namely, high specific conductivity and low mobility in relation to the parameters of the bulk part. In previous works [13-16], studies in doped samples were focused mainly on bulk parts of the crystal obtained by removing the near-surface region, since the near-surface layer was considered to be damaged and not of practical interest.

However, for an in-depth study of the process of diffusion doping of silicon with metals and atoms of rare earth elements, it is necessary to consider such important issues as the physics of the formation of a heavily doped near-surface region, the nature of the formation of metal silicides, which are very different from the metal and semiconductor, as well as the physical and chemical processes occurring in the near-surface region (mutual diffusion, solid-phase reactions, etc.).

It is known that at present, transition metal silicides are becoming the base materials for new, promising technological integrated circuits ((Very Large-Scale Integrated Circuit (VLSI)) of future generations due to their resistance to aggressive environments and high-temperature treatments [17-19].

In particular, transition element silicides can be used to create Schottky barriers and ohmic contacts in integrated circuit technology, serving as gates and interconnects.

In this regard, if we consider the formation of silicides by metallurgical interaction between a metal film and silicon, then subsequent high-temperature heat treatment, which promotes diffusion doping of silicon, leading to the formation of a distribution profile of the concentration of current carriers, requires comprehensive studies of the mechanism of impurities entering the crystal volume and their interaction both with matrix (main) atoms of the crystal, and with technological (background) impurities, as well as with structural defects.

Therefore, the study of silicides in the near-surface region in the process of diffusion doping of silicon is in itself relevant, and the formation of a carrier concentration profile in the near-surface region of silicon doped with impurities that create deep levels opens up ways to create new semiconductor devices.

### EXPERIMENTAL PART

For the study, we used dislocation-free single-crystalline silicon ingots with a diameter of 65÷110 mm, grown by the Czochralski method, with  $\rho=10^4 \text{ Ohm}\cdot\text{cm}$ , because This method was the first to study such crystals. To convincingly

prove our results, the experiment was carried out first for known crystals. The ingots were cut in cross section into washers. Samples were cut from the washers in the form of a parallelepiped with dimensions of  $(65 \div 110) \times (20 \div 25) \times (3 \div 5) \text{ mm}^3$ .

After grinding, the samples were subjected to chemical etching in a solution in the following composition (weight in %): HF -  $36 \div 30$ ,  $\text{CgO}_3$  -  $15 \div 18$ , water - the rest. Etching was carried out in a closed vessel for  $8 \div 10$  minutes. at a temperature of  $80\text{-}100 \text{ }^\circ\text{C}$ .

Studies of high-purity non-heat-treated dislocation-free silicon samples using electron microscopy, X-ray microanalysis and small-angle scattering of  $\text{CO}_2$  laser radiation revealed that three types of defects were found in them: swirl defects, impurity micro inclusions and impurity clouds.

Swirl defects are generated by a heterogeneous mechanism and are arranged in layers. The concentration of such defects is  $10^6\text{-}10^8 \text{ cm}^{-3}$ . By selecting the cooling rate when growing silicon, you can get rid of this type of defects [20].

The second type of defect is an impurity micro inclusion includes impurities of carbon, calcium, phosphorus, chlorine, aluminum, sodium and some others. Micro inclusions have sizes from fractions of a micron and higher and a concentration of  $\sim 10^7 \text{ cm}^{-3}$ . It is assumed that these impurities are captured by the growing single crystal from the melt. In a high-purity crystal that is undersaturated in impurities included in the micro inclusions, their partial dissolution can occur. In this case, slowly diffusing impurities will form clusters around the inclusions with a size of about 10 microns. Such local areas with an increased concentration of dissolved impurities are called impurity clouds [21]. Such defects were detected by small-angle scattering of  $\text{CO}_2$  laser radiation with  $\lambda = 10.6 \text{ }\mu\text{m}$ .

Thus, the proposed methods for monitoring the level of silicon homogeneity by the presence of microdefects in the structure can be used in the production of large-diameter dislocation-free silicon, from which ultra-large circuits and semiconductor devices are created.

The method of small-angle scattering of  $\text{CO}_2$  laser radiation was used to determine silicide islands in the surface layer of diffusely doped silicon with manganese, chromium and cobalt. The samples were made from p-type silicon of the KDB-10 brand at a diffusion temperature of  $1040\text{-}1100 \text{ }^\circ\text{C}$  for 20-120 minutes. The samples were hardened by cooling at a rate of  $100\text{-}120 \text{ }^\circ\text{C/s}$  with the ampoules being dropped into water. To identify silicide islands, five of the six sides of the parallelepiped were polished to a depth of  $50\text{-}100 \text{ }\mu\text{m}$ . On the remaining face, measurements were taken as a layer  $3\text{-}5 \text{ }\mu\text{m}$  thick was removed from the surface. Measurements showed that silicide inclusions with sizes of  $8\text{-}20$  microns are formed in the near-surface layer. The determination of silicide inclusions by the small-angle scattering method is based on light scattering caused by the deviation from the average value of the dielectric constant  $\Delta\epsilon$  for the crystal. At  $\lambda = 10.6 \text{ }\mu\text{m}$ , the value of  $\Delta\epsilon$  for a charge carrier accumulation is three orders of magnitude greater than for a neutral impurity accumulation with the same concentration. It is assumed that  $\Delta\epsilon$  for silicide islands is mainly due to holes. Inhomogeneity such as impurity clouds scatters light at small diffraction angles  $\theta \sim \lambda/a$ , where  $a$  is the characteristic size of the cloud. According to the Rayleigh-Gans theory [22], the scattered light flux per unit solid angle is determined by the expression:

$$I(\theta) = WCL|G|^2 \quad (1)$$

where:  $W$  is the initial light flux in the crystal,  $C$  is the concentration of scattering centers,  $L$  is the thickness of the sample,  $G(\theta)$  is the integral over the volume of an individual inhomogeneity, the form of which depends on the polarization of light.

The nature of the dependence of the intensity of small-angle scattering of laser radiation  $I$  (normalized to the power of the incident beam and to the thickness of the sample) on the scattering angle  $\theta$  makes it possible to establish the radial profile and size "a" of the silicide inclusion: the intensity  $I_0$  extrapolated to  $\theta = 0$  serves as a characteristic of the total amount of electrically active impurity in silicide clouds.

## RESULTS AND DISCUSSION

In Fig. 1. The dependence of the size of the silicide inclusion at three points of the crystal on the thickness of the layer removed from the crystal surface is shown. It can be seen that the sizes of silicide inclusions decrease linearly deeper into the crystal.

In Fig.2. The dependence of the concentration of silicide inclusions on the distance (into the crystal) is shown. From Fig. It can be seen that on the surface of a crystal with a thickness of  $\sim 3 \text{ }\mu\text{m}$ , silicide inclusions are saturated, i.e. a continuous film is formed. At depths from 3 to  $30 \text{ }\mu\text{m}$ , the concentration of inclusions decreases smoothly.

It has been established that the concentration of silicide inclusions in the near-surface region of the crystal depends on the type of substrate, the amount of diffusant, the diffusion temperature and the quenching rate.

The electrical properties of chromium disilicide were determined: band gap  $(0.29 \pm 0.02) \text{ eV}$ , resistivity  $1800 \text{ }\mu\text{Ohm}\cdot\text{cm}$ , thermoEMF coefficient  $180 \text{ }\mu\text{V/KI}$ , Hall constant  $1.2 \cdot 10^{-2} \text{ cm}^3/\text{KI}$ , hole concentration  $6 \cdot 10^{19} \text{ cm}^{-3}$ , charge carrier mobility  $18.6 \text{ cm}^2/\text{V}\cdot\text{s}$ ,  $ZT = 0.25$ .

The experimental results obtained show that Cr, Mn, Co in silicon have increased reactivity: metal phases of chromium, manganese and cobalt are not detected on the surface of the doped crystals. Mono- and di-higher metal silicides are formed on the surface in the case of diffusion doping of silicon from the gas phase. The amorphous phase was observed in the surface layer with a thickness of  $3\text{-}10 \text{ }\mu\text{m}$ . At a depth of  $10\text{-}50 \text{ }\mu\text{m}$ , various silicides were observed in different

samples: for Si<Mn>, predominantly higher manganese silicide was observed, for Si<Cr> - chromium mono and disilicides, and for Si<Co> - predominantly cobalt disilicide. When removing a layer from the surface of samples with a thickness of more than 50 microns, silicides of the above metals were not detected.

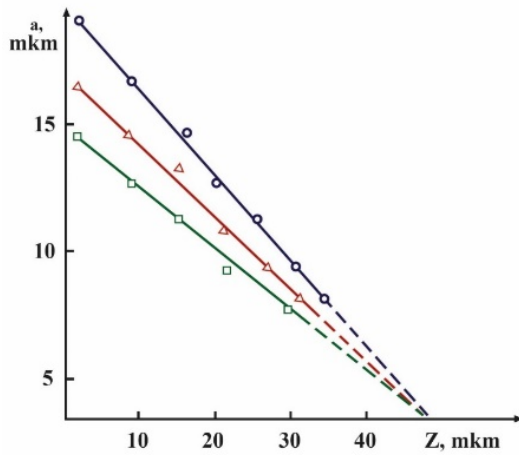


Figure 1. Dependence of the size of various silicide inclusions on the crystal depth

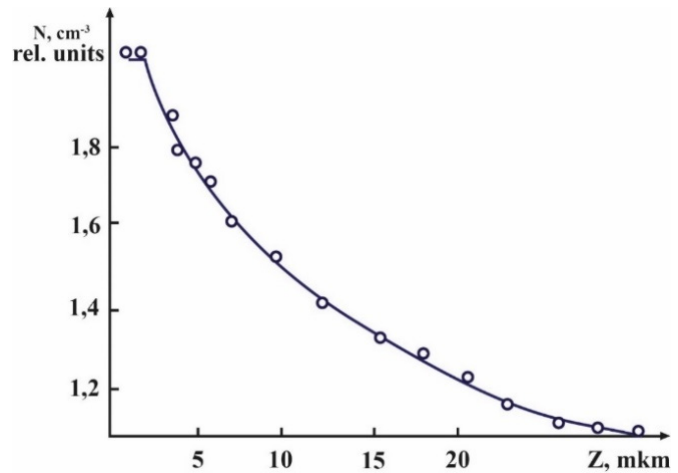


Figure 2. Dependence of the surface concentration of silicide inclusions on the crystal depth

The formation of cobalt silicides on polycrystalline silicon by direct implantation of metals was studied in the work of M. Kozicki [23]. Co ions with an energy of 150-350 keV (at a dose of  $7 \cdot 10^{16} - 7 \cdot 10^{17} \text{ cm}^{-2}$ ) were implanted into single-crystal silicon wafers. After implantation, the sample was annealed at a temperature of 950 °C for 10-20 s. The properties of Si silicide layers were studied using Rutherford inverse scattering, X-ray diffractometry, and other methods.

A study of the kinetics of growth of the silicide phase along a cross section of samples in the case of annealing of deposited manganese on silicon showed that the dependence of the thickness of the silicide layer ( $W$ ) on the annealing time ( $t$ ) is parabolic (Fig. 3.). At lower values of  $t$ , this dependence is violated (Fig. 3, section AB), which is apparently associated with limiting the reaction rate of silicide formation. The layers are formed in thickness proportional to the square root of time (section BC). The thickness of the silicide layer  $W$  formed during time  $t$  is determined by the expression

$$W = \{[B \cdot \exp(-E_a/kT)]t^{1/2}\} \quad (2)$$

where  $B$  is a constant,  $E_a$  is the average activation energy of the growth process,  $k$  is Boltzmann's constant,  $T$  is the absolute temperature.

In the formation of the  $\text{CoSi}$  and  $\text{Co}_2\text{Si}$  layer, the same  $t^{1/2}$  dependence of phase growth was also established. And in the case of  $\text{CrSi}_2$ , a linear dependence on time was discovered, suggesting interaction with a rate determined by the state of the interface. The value of the diffusion coefficient  $D$  can be calculated from the slope of the time dependence lines  $W$ .

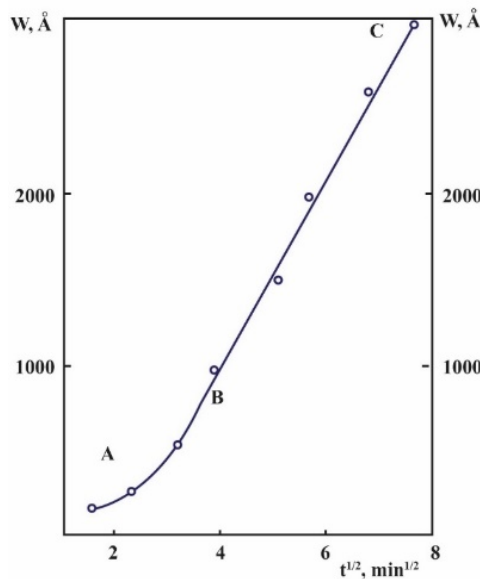


Figure 3. Dependence of the HCM layer thickness on annealing time.

The activation energy of the phase formation process can be determined from the dependence of the diffusion coefficient on the inverse temperature, i.e. according to Arrhenius plots [24]. The activation energies are 1.5 eV for CrSi<sub>2</sub>, 1.5 eV for Co<sub>2</sub>Si, 1.9 eV for CoSi, and 1.4 eV for MnSi<sub>1.75</sub>. Analysis of the results obtained shows that during the formation of silicides, silicides rich in metal atoms are first formed, and then metal monosilicides, in the case of diffusion doping of silicon from the gas phase. And in the case of metal-sputtered silicon, a metal silicide enriched with the latter is formed at the interface between silicon and metal, after thermal annealing.

It should be noted that oxygen lines are observed in the spectrum during thermal annealing, which is the Me<sub>2</sub>O<sub>3</sub> compound. Analysis of the results showed that the surface of the deposited metal during temperature annealing is covered with a thin layer of Me<sub>2</sub>O<sub>3</sub>, which plays the role of a diffusion barrier for oxygen from the annealing atmosphere.

The formation of the annealing phase of silicides during thermal annealing obeys the rule of the formation of nuclei at the metal-semiconductor interface. This rule is based on the postulate that initially the interface is a metal-glass with a concentration close to the low-temperature eutectic. However, the experimental setup used is not able to show the existence of such an intermediate layer in reality. There is no exact mechanism for the nucleation of metal silicides yet, but it can be represented by the growth of metal grains and the diffusion of silicon atoms into a polycrystalline film of chromium (or manganese, cobalt) mainly along the grain boundaries. This behavior of silicon was observed in the case of iron-silicon interaction. Ryan et al. suggest that the growth of silicide when depositing pure metal on silicon creates equilibrium conditions by lowering their interfacial energy.

Botha and Kritzinger [25] studied the diffusion mechanism in the formation of nickel, cobalt and platinum silicides using traces of radioactive silicon. They created thin-film structures on Si(100) substrates using electron-beam heating. Radioactive <sup>31</sup>Si (half-life 2.62 hours), obtained by irradiating pure natural Si in a nuclear reactor, was washed in organic solvents, etched in a 10% solution of hydrogen fluoride, after which it was deposited on Si by evaporating the source. Between layers of radioactive Si with thicknesses of 500 Å and 700 Å, layers of metal (Pt, Ni, Co) with thicknesses of 1000 Å and 1600 Å were deposited. After this, annealing was carried out at 10<sup>-6</sup> Pa to form silicides. The profiles were studied using layer-by-layer HF sputtering using Rutherford backscattering and Geiger-Muller methods. During the growth of NiSi, diffusion of the metal was detected, while during the growth of CoSi and PtSi, predominantly Si diffuses. The Co<sub>2</sub>Si transition occurs predominantly during Si diffusion along grain boundaries, while PtSi is formed from Pt<sub>2</sub>Si both during Si diffusion at grain boundaries and through the vacancy diffusion mechanism. After nucleation, a reaction of the form Me + 2Si → MeSi<sub>2</sub> occurs, occurring at the MeSi<sub>2</sub> – Me boundary.

The growth of silicides is limited by three factors:

1. The presence of surface silicon atoms;
2. Transfer of silicon atoms through an already formed MeSi<sub>2</sub> layer;
3. Reaction at the interface.

In the temperature range studied, an increase in the thickness of the MeSi<sub>2</sub> layer either leads to a decrease in the growth rate or the second factor is limiting.

The lack of formation of metal silicides at temperatures below ~400 °C is explained by the increasing difficulty of releasing silicon atoms from its covalent bond. At temperatures above ~400 °C, phonons can provide the necessary energy to liberate a silicon atom.

Finally, when the growth temperature of the silicide decreases, the Si-MeSi<sub>2</sub> interface becomes rougher and rougher, so that the exchange across the surface increases, which can explain the observed time dependence of t<sup>1/2</sup> in the case of the growth of manganese and cobalt silicides.

## CONCLUSION

As a result of the scientific research, we came to the following important scientific conclusions:

1. Based on the study of surface morphology by electron and IR microscopy, the formation of impurity accumulations, which represent the second phase on the surface of diffusion doped silicon with atoms of manganese, chromium and cobalt, has been established. The sizes of the clusters were determined, reaching up to 30-40 μm on the surface and decreasing deeper into the crystal. Qualitative and quantitative analysis showed that atoms of the doped atom and silicon are present on the surface of the crystals under study. Some crystals exhibit a granular structure, which is explained by the diffusion condition.
2. By studying the kinetics of growth of the silicide phase along a cross section of samples, it was shown that during the formation of silicides CoSi, Co<sub>2</sub>Si, CoSi<sub>2</sub>, MnSi, MnSi<sub>1.75</sub>, MnSi<sub>2</sub>, there is a quadratic dependence of the layer thickness on the annealing time, and for silicides CrSi and CrSi<sub>2</sub> there is a linear dependence. The activation energies of phase formation for the studied silicides were determined and are 1.4-1.9 eV.
3. It has been shown that the formation of silicide nuclei at the silicon-metal interface begins with the decomposition of silicon atoms at the grain boundaries of the deposited metal layer. In this case, metal disilicides are formed. It has also been shown that the formation of Cr, Mn, Co silicides occurs at temperatures above 400°C.
4. Using the method of small-angle CO<sub>2</sub> laser scattering, the formation of silicide inclusions with dimensions of 8÷20 μm in the near-surface region with a thickness of 3÷40 μm in samples of diffusion-doped silicon with manganese, chromium and cobalt was established. The determination of such inclusions is based on the deviation of their dielectric constant from the average value for the crystal.

5. The dependence of the size of silicide inclusions and their concentration on the thickness of the removed layer (depth profile) was studied. A decrease in the size of silicide inclusions with depth of the crystal has been established, which makes it possible to study the dynamics of growth of the silicide layer.

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**ДОСЛІДЖЕННЯ СИЛІЦИДОУТВОРЕННЯ В МОНОКРИСТАЛІЧНОМУ КРЕМНІІ ВЕЛИКОГО ДІАМЕТРА****Абдугафур Т. Мамадалімов<sup>a</sup>, Махмудходжа Ш. Ісаєв<sup>b</sup>, Мухаммадсодік Н. Маматкулов<sup>c</sup>,****Сардор Р. Кодиров<sup>d</sup>, Джамшидjon Т. Абдуразоков<sup>e</sup>**<sup>a</sup>*Інститут фізики напівпровідників та мікроелектроніки Національного університету Узбекистану, Ташкент, Узбекистан*<sup>b</sup>*Національний університет Узбекистану, Ташкент, Узбекистан*<sup>c</sup>*Ташкентський хіміко-технологічний інститут, Ташкент, Узбекистан*<sup>d</sup>*Ургенцький державний університет, Ургенч, Узбекистан*<sup>e</sup>*Ташкентська медична академія, Ташкент, Узбекистан*

Для дослідження утворення силіцидів використовували бездислокаційні злитки монокристалічного кремнію діаметром 65÷110 мм, вирощені методом Чохральського. При дослідженні таких зразків кремнію методом електронної мікроскопії, малокутового розсіювання випромінювання CO<sub>2</sub>-лазера виявлено три типи дефектів: завихрені дефекти, домішкові мікрровключення та домішкові хмари. Показано, що в приповерхневому шарі легованого кремнію утворюються силіцидні вклучення розміром 8-20 мкм, які лінійно зменшуються вглиб кристала. Визначено електричні параметри напівпровідникового силіциду хрому: питомий опір 1800 мкОм·см, коефіцієнт термоЕРС 180 мкВ/к, стала Холла  $1,2 \cdot 10^{-2}$  см<sup>3</sup>/Кл, концентрація дірок  $6 \cdot 10^{19}$  см<sup>-3</sup>, рухливість носіїв заряду 18,6 см<sup>2</sup>/В·с, ширина забороненої зони (0,29±0,02) еВ.

**Ключові слова:** бездислокаційний; травлення; дефекти; завихрення; гетерогенність; розчинення; надвеликі схеми; зміцнення; вклучення; екстраполяція