

Effect of inter-impurity interaction on concentration of electroactive manganese impurity atoms in silicon

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Abstract. Influence of the concentration of electroactive manganese impurity on interatomic interactions in single-crystalline *p*-type silicon initially doped with boron was investigated. The electrophysical parameters of silicon samples subjected to diffusion doping at a given manganese vapor pressure and subsequent thermal annealing were studied. It was found that almost all of the manganese atoms incorporated in KDB-1 grade *p*-type silicon with boron concentration $N_B \geq 2 \cdot 10^{16} \text{ cm}^{-3}$ become electroactive. For theoretical calculations, the concentration value was set equal to the solubility limit of manganese atoms in silicon. At the same time, the concentration of the electroactive manganese atoms in the *n*-type silicon with phosphorus concentration $N_P \approx 10^{16} \text{ cm}^{-3}$ did not exceed $3 \cdot 10^{14} \text{ cm}^{-3}$. It was shown that the electroactivity of manganese impurity atoms is defined not only by thermodynamic parameters of diffusion but also by the type and concentration of the initial donor or acceptor impurity. It was established that boron atoms can act as nucleation centers for manganese nanoclusters, and these nanoclusters significantly affect the electrical properties of the original silicon. The developed two-stage low-temperature diffusion technology suppresses silicide formation and ensures uniform distribution of electroactive manganese atoms at maximum concentration. The obtained results are of interest for creating nanostructured silicon with tailored electrical, photovoltaic, optical, and magnetic properties, promising for use in electronics.

Keywords: silicon, manganese, boron, phosphorus, inter-impurity interaction, diffusion, concentration of electroactive impurity, nanoclusters.

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1. Introduction

Diffusion doping of silicon with impurities, particularly manganese, is of interest for creating new functional devices with controlled electrophysical, photoelectric, optical, and magnetic properties. However, obtaining silicon with the highest possible concentration of electroactive manganese atoms remains a challenge. Analysis of the literature has shown that the total manganese concentration and the concentration of electroactive manganese atoms in silicon may differ due to formation of complexes, clusters, and silicides, as well as due to interactions of manganese atoms with the atoms already present in the silicon crystal lattice, particularly boron or phosphorus [1, 2].

Silicon doped with manganese was produced in quartz ampoules with controlled diffusant vapor pressure. The charge carrier concentration was measured using the

Hall effect method. It was demonstrated for the first time that when the boron impurity concentration in the initial *p*-type silicon is $N_B \geq 2 \cdot 10^{16} \text{ cm}^{-3}$, practically all of the introduced manganese impurity atoms become electroactive. On the other hand, in *n*-type silicon initially doped with phosphorus, the portion of the electroactive manganese impurity atoms amounted to a few percent of the manganese maximum solubility [3–5].

Therefore, further research must account for how the background dopant concentration (such as boron and phosphorus) in the starting material, alongside the thermodynamic processing conditions, affects the final concentration of electroactive impurities in silicon [6–13].

Here, we investigate how the concentration of shallow-level boron dopant in the initial silicon influences the concentration of electroactive manganese impurity, which acts as deep-level centers.

2. Technology and experimental results

The study utilized KDB-grade single-crystal silicon with p -type conductivity and resistivities $\rho = 1, 4.5, 10, 100$, and $220 \text{ Ohm}\cdot\text{cm}$, as well as KEF-grade silicon with n -type conductivity and resistivities $\rho = 2, 10, 25, 70$, and $200 \text{ Ohm}\cdot\text{cm}$. The residual oxygen concentration was $N_{O_i} \approx (5\ldots 7) \cdot 10^{17} \text{ cm}^{-3}$. All the silicon samples, measuring $8 \times 4 \times 1 \text{ mm}^3$, were cut from an ingot using an STX-402 diamond wire cutting machine. After cutting, the samples were chemically etched in a solution $\text{HNO}_3\text{:HF}$ (3:1), cleaned by boiling in a peroxide-ammonia solution $\text{NH}_4\text{OH:H}_2\text{O}_2\text{:H}_2\text{O}$ (1:4:12), and further cleaned in a 10% HF solution.

Manganese impurity was incorporated by diffusion from gas phase under identical conditions for all the silicon samples at a temperature $T = 1160 \text{ }^\circ\text{C}$ and a diffusion time up to $t = 1 \text{ hour}$. Two samples of each of the p - and n -conductivity types were placed separately in each quartz ampoule.

Simultaneously, control samples containing no manganese impurity were thermally annealed under the same conditions to assess the effect of the diffusion temperature on the electrical properties of manganese-doped silicon samples.

During diffusion in quartz ampoules, the manganese vapor pressure did not exceed $P = 10^{-6} \text{ mmHg}$. The diffusion coefficient of the manganese atoms in silicon was determined by the following relationship [14]:

$$D = 0.26 \cdot \exp\left(\frac{E_a}{kT}\right), \frac{\text{cm}^2}{\text{s}}, \quad (1)$$

where k is the Boltzmann constant ($k = 1.38 \cdot 10^{-23} \text{ J/K}$), and E_a is the diffusion activation energy ($E_a = 1.3 \text{ eV}$), respectively.

The solubility of manganese in silicon was determined by the following expression:

$$N_{\text{Mn}} = 2.5 \cdot 10^{22} \cdot \exp\left(\frac{-2.1}{kT}\right), \text{cm}^{-3}, \quad (2)$$

According to the results of [15], the total solubility of manganese in silicon at a temperature $T = 1090 \text{ }^\circ\text{C}$ is $N_{\text{Mn}} \sim 10^{16} \text{ cm}^{-3}$.

Diffusion of manganese atoms into silicon was carried out in a MAGNETIC electric furnace, providing temperature control with an accuracy of $\pm 0.5 \text{ }^\circ\text{C}$.

Initially, the samples were heated from room temperature ($30 \text{ }^\circ\text{C}$) to $580\ldots 680 \text{ }^\circ\text{C}$ at $5 \text{ }^\circ\text{C/min}$ and held for $10\ldots 20 \text{ min}$. The temperature was then ramped at $15\ldots 20 \text{ }^\circ\text{C/min}$ up to the high value corresponding to diffusion regime ($980\ldots 1250 \text{ }^\circ\text{C}$) and maintained for $20\ldots 40 \text{ min}$ (Fig. 1). Finally, the quartz ampoules were extracted from the furnace and quenched in technical oil at a cooling rate of $\sim 150\ldots 200 \text{ }^\circ\text{C/s}$.

In the remainder of the text, the stated diffusion temperature corresponds to the peak final temperature of the process. The specific thermal history and this final temperature govern the proportion of electrically active manganese atoms.

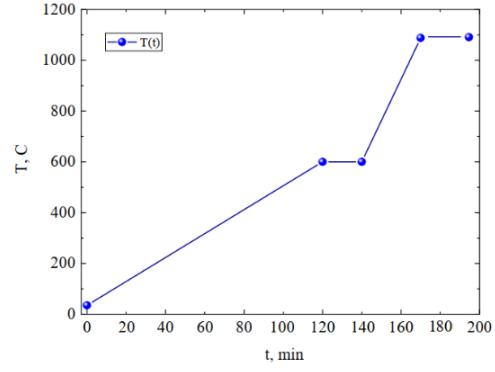


Fig. 1. Temperature regimes of diffusion of manganese impurity into silicon.

Then the samples were mechanically ground with M-14 silicon carbide abrasive (removing a $\sim 50 \text{ }\mu\text{m}$ thick layer on all sides) to remove manganese silicides and chemically etched in an HF:HNO_3 (1:8) solution for $t = 3 \text{ min}$ to remove the Si surface layer with grinding-induced defects. The described procedure enabled formation of p -type samples with the resistivity $\rho = 10^3 \ldots 10^5 \text{ Ohm}\cdot\text{cm}$.

3. Experimental methodology

Measurements of electrophysical characteristics of silicon samples after diffusion of manganese impurity were performed using an Ecopia Hall HMS-7000 van der Pauw setup. The results showed that, regardless of identical diffusion conditions (temperature, diffusion time, cooling rate), the concentration of electroactive manganese atoms significantly depended on the concentration of boron or phosphorus impurity in the original silicon.

The total concentration of electroactive manganese atoms was determined by solving the electroneutrality equation based on the obtained experimental results, taking into account the degree of filling of both energy levels of the manganese atoms ($E_1 = E_c - 0.24 \text{ eV}$, $E_2 = E_c - 0.5 \text{ eV}$) in the silicon band gap.

For the compensated p -type silicon samples ($p\text{-Si(B,Mn)}$), the concentration of manganese atoms was determined by solving the following neutrality equation:

$$N_{\text{Mn}} = \frac{1}{2} (p_0 - p - N_{TD}). \quad (3)$$

For overcompensated silicon samples ($n\text{-Si(B,Mn)}$), the concentration of manganese atoms was determined as the solution of the following equation:

$$N_{\text{Mn}} = p_0 + n_0 + f(E_1)N_{\text{Mn}} + f(E_2)N_{\text{Mn}} - N_{TD}. \quad (4)$$

The concentration of manganese atoms in $n\text{-Si(P,Mn)}$ was determined from the following equations:

$$N_{\text{Mn}} = \frac{1}{2} (n - n_0 + f(E_1)N_{\text{Mn}} + f(E_2)N_{\text{Mn}} - N_{TD}). \quad (5)$$

$$n_0 = \sqrt{N_C N_D} \exp\left(-\frac{E_C - E_D}{2kT}\right), \quad (6)$$

$$p_0 = \sqrt{N_C N_A} \exp\left(-\frac{E_A - E_V}{2kT}\right), \quad (7)$$

$$E_F = \frac{E_C + E_D}{2} + \frac{kT}{2} \ln \frac{N_D}{N_C}, \quad (8)$$

where p_0 and p are the hole concentrations before and after diffusion of manganese, n_0 and n are the electron concentrations before and after the diffusion, N_{TD} is the thermal donor concentration, $f(E_1)$ and $f(E_2)$ are the filling degrees of the 1st and 2nd energy levels of manganese atoms, E_D and E_A are the activation energies of boron and phosphorus atom levels, and E_F is the Fermi level, respectively. The hole concentrations p_0 and p as well as the concentrations of electrons n_0 and n were determined from Hall measurements of electrical parameters of silicon samples before and after the diffusion. The N_{TD} concentration was determined by measuring the charge carrier concentration before and after thermal annealing in control (undoped, annealed under the same conditions) silicon samples.

Fig. 2 shows concentrations of electroactive manganese impurity atoms in silicon. As can be seen from this figure, the concentration of the electroactive manganese atoms in the samples with the resistivity $\rho = 1 \text{ Ohm}\cdot\text{cm}$ is almost two orders of magnitude higher than in the samples with the resistivity $\rho = 220 \text{ Ohm}\cdot\text{cm}$ despite the fact that these samples were doped in the same ampoule under absolutely identical process conditions.

The concentration of the electroactive manganese impurity atoms in the initial silicon with the resistivity $\rho = 1 \text{ Ohm}\cdot\text{cm}$ reaches $N_{Mn} = 1.2 \cdot 10^{16} \text{ cm}^{-3}$, which is practically close to the manganese maximum solubility at 1200°C ($N_{Mn} = 2 \cdot 10^{16} \text{ cm}^{-3}$). Under the same diffusion conditions, the manganese concentration in the initial silicon with a resistivity $\rho = 220 \text{ Ohm}\cdot\text{cm}$ does not exceed $(3...3.5) \cdot 10^{14} \text{ cm}^{-3}$, i.e., almost two orders of magnitude less than in the KDB-1 silicon samples. It was found therefore that the concentration of the electroactive manganese impurity atoms depends on the boron concentration in the initial silicon.

It is known that the concentration of boron impurity atoms in KDB-1 silicon is $N_B = 2 \cdot 10^{16} \text{ cm}^{-3}$, while for samples with the resistivity $\rho = 220 \text{ Ohm}\cdot\text{cm}$ (KDB-220), it is $N_B = 1.5 \cdot 10^{13} \text{ cm}^{-3}$, which corresponds to the observed difference in the concentrations of the electroactive manganese impurity in both cases. These results demonstrate that under identical doping process conditions, the electroactive manganese concentration is defined by the boron concentration in the original silicon.

The resistivity of the KDB-1 silicon samples reaches $\rho \approx 3.4 \cdot 10^4 \text{ Ohm}\cdot\text{cm}$ after diffusion of manganese at a temperature $T = 1200^\circ\text{C}$. At the same time, the samples with the initial resistivity $\rho = 100 \text{ Ohm}\cdot\text{cm}$, the boron concentration in which is $N_B = 2 \cdot 10^{14} \text{ cm}^{-3}$, become n -type with the resistivity $\rho \approx 5.8 \cdot 10^2 \text{ Ohm}\cdot\text{cm}$ after diffusion of manganese at the same temperature as in the previous cases.

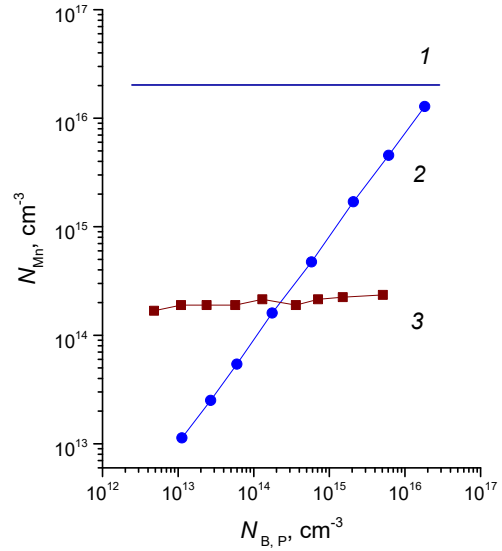


Fig. 2. Concentrations of electroactive manganese atoms versus concentration of boron or phosphorus in the original silicon. 1 – maximum solubility of manganese in silicon (1160°C); 2 – dependence of the concentration of electroactive manganese atoms on the boron concentration in the original silicon; 3 – dependence of the concentration of electroactive manganese atoms on the phosphorus concentration in the original silicon.

It was found therefore that the concentration of the electroactive manganese atoms increases at increasing the boron concentration in the original silicon. At $N_B \sim 2 \cdot 10^{16} \text{ cm}^{-3}$, almost all of the embedded manganese atoms become electroactive [16–20]. Unlike this, in the KDB-100 case, the concentration of the electroactive manganese impurity reaches only about 1% of the total dissolved manganese concentration.

The effect of the phosphorus concentration in the initial n -type (KEF) silicon on the concentration of electroactive manganese impurity was also studied. The obtained results showed that, unlike the cases of initial silicon with boron impurity (p -type conductivity), this concentration is independent of the phosphorus concentration in the initial material.

The concentration of the electroactive manganese impurity atoms obtained at the studied diffusion temperatures $T = 1000...1270^\circ\text{C}$ is approximately $N_{Mn} = (2...2.5) \cdot 10^{14} \text{ cm}^{-3}$, which is almost two orders of magnitude lower than the concentration of the manganese impurity atoms in the initial KDB-1 silicon (curve 3). These results demonstrate that the concentration of the electroactive manganese atoms significantly depends on the conductivity type and the concentration of boron and phosphorus in the initial silicon.

As can be seen from Table 1, depending on the initial concentration of boron impurity, materials with different electrophysical parameters (specific resistance and conductivity type) can be obtained at the same temperature and time of diffusion annealing.

Table 1. Resistivity of silicon samples doped with manganese impurity as a function of the initial boron concentration at various diffusion temperatures. The diffusion time at all the temperatures is $t = 60$ min.

Diffusion temperature, °C	KDB-1 $2 \cdot 10^{16} \text{ cm}^{-3}$	KDB-2 10^{16} cm^{-3}	KDB-4.5 $6 \cdot 10^{15} \text{ cm}^{-3}$	KDB-10 $2 \cdot 10^{15} \text{ cm}^{-3}$	KDB-100 $2 \cdot 10^{14} \text{ cm}^{-3}$
1050	65* <i>p</i> -type	$1 \cdot 10^2$ <i>p</i> -type	$3 \cdot 10^2$ <i>p</i> -type	$2 \cdot 10^5$ <i>p</i> -type	$1.6 \cdot 10^3$ <i>n</i> -type
1100	80 <i>p</i> -type	$6 \cdot 10^3$ <i>p</i> -type	$1.6 \cdot 10^5$ <i>p</i> -type	$9.2 \cdot 10^3$ <i>n</i> -type	$4 \cdot 10^2$ <i>n</i> -type
1150	$1.2 \cdot 10^3$ <i>p</i> -type	$8 \cdot 10^4$ <i>n</i> -type	$2.3 \cdot 10^4$ <i>n</i> -type	$1.4 \cdot 10^2$ <i>n</i> -type	60 <i>n</i> -type
1200	$6.4 \cdot 10^4$ <i>p</i> -type	$1.5 \cdot 10^4$ <i>n</i> -type	$7.5 \cdot 10^3$ <i>n</i> -type	$8.5 \cdot 10^2$ <i>n</i> -type	34 <i>n</i> -type

*Specific electrical resistivity of the samples after doping

4. Discussion

Electron paramagnetic resonance (EPR) studies of silicon samples doped with manganese impurity revealed [21, 22] that manganese atoms form nanoclusters, each consisting of four atoms located in nearby interstitial positions of the silicon crystal lattice. Broadening of the EPR spectrum to 21 lines [21] confirms close relative positions of these nanoclusters, caused by resonant interaction of the manganese atom d-shells containing unpaired electrons.

It has been found out that manganese atoms cannot form nanoclusters without additional thermal annealing. X-ray diffraction analysis [23] revealed that manganese nanoclusters contain boron atoms. Presence of Mn_xB_y

peaks indicates the possibility of manganese nanoclusters forming around a nodal boron atom [23]. Furthermore, boron atoms serve as nuclei for formation of interstitial manganese nanoclusters. Consequently, the initial concentration of boron atoms in silicon influences the electroactivity by stimulating formation of manganese atom clusters in the lattice.

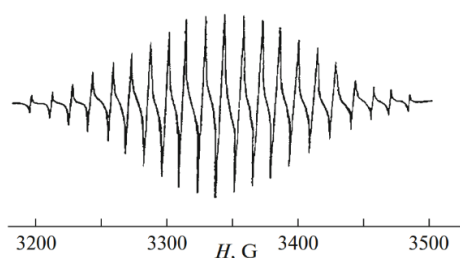
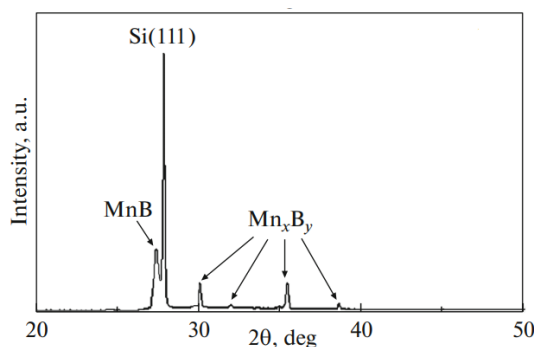
X-ray dispersive analysis of *n*-type silicon samples doped with manganese impurity showed that nanoclusters do not form in these samples. This confirms that presence of boron impurity atoms in the original silicon is a necessary condition for the formation of manganese nanoclusters.

5. Conclusions

Analysis of the obtained results reveal that the electroactive manganese impurity atoms in silicon significantly depends on the type and concentration of donor and acceptor impurities present in the starting material. At a boron concentration $N_B \geq 2 \cdot 10^{16} \text{ cm}^{-3}$, almost complete activation of the manganese atoms is achieved (up to $N_{\text{Mn}} \sim 1.2 \cdot 10^{16} \text{ cm}^{-3}$). In *n*-type silicon, the concentration of the electroactive manganese does not exceed $N_{\text{Mn}} \approx (2 \dots 3) \cdot 10^{14} \text{ cm}^{-3}$ even at high diffusion temperatures. It is shown that use of the developed low-temperature two-stage diffusion technology under controlled vapor pressure of manganese during the diffusion process significantly reduces the concentration of electroactive manganese atoms. Therefore, optimization of diffusion doping parameters including selection of initial silicon based on the concentration of impurity atoms as well as choice of temperature, diffusion time, and vapor pressure of the diffusant are key factors in obtaining silicon with specified electrophysical parameters.

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**Fig. 3.** EPR spectrum of a nanocluster consisting of four manganese atoms in silicon [21].**Fig. 4.** X-ray dispersion analysis of a silicon sample with manganese nanoclusters [23].

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Вплив міждомішкової взаємодії на концентрацію електроактивних атомів домішки марганцю в кремнії

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Анотація. Досліджено вплив концентрації електроактивної домішки марганцю на міжатомні взаємодії в монокристалічному кремнії *p*-типу, початково легуваному бором. Досліджено електрофізичні параметри зразків кремнію, що піддалися дифузійному легуванню при заданому тиску пари марганцю та подальшому термічному відпалу. Було виявлено, що майже всі атоми марганцю, що входять до складу кремнію *p*-типу марки KDB-1 з концентрацією бору $N_B \geq 2 \cdot 10^{16} \text{ cm}^{-3}$, стають електроактивними. Для теоретичних розрахунків значення концентрації було встановлено рівним межі розчинності атомів марганцю в кремнії. Водночас, концентрація електроактивних атомів марганцю в кремнії *n*-типу з концентрацією фосфору $N_P \approx 10^{16} \text{ cm}^{-3}$ не перевищувала $3 \cdot 10^{14} \text{ cm}^{-3}$. Було показано, що електроактивність атомів домішки марганцю визначається не лише термодинамічними параметрами дифузії, але й типом та концентрацією початкової донорної або акцепторної домішки. Було встановлено, що атоми бору можуть виступати центрами нуклеації для нанокластерів марганцю, і ці нанокластери суттєво впливають на електричні властивості вихідного кремнію. Розроблена двостадійна низькотемпературна дифузійна технологія пригнічує утворення силіцидів та забезпечує рівномірний розподіл електроактивних атомів марганцю при максимальній концентрації. Отримані результати становлять інтерес для створення наноструктурованого кремнію із заданими електричними, фотоелектричними, оптичними та магнітними властивостями, перспективним для використання в електроніці.

Ключові слова: кремній, марганець, бор, фосфор, міждомішкова взаємодія, дифузія, концентрація електроактивної домішки, нанокластери.