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Morphological Parameters of Impurity Clumps of Co and Ni in Si under the Influence of Pressure

Zainabidinov S.Z.[1], Akbarov Sh.K.[1], Turgunov N.A.[2]
[1]*Andijan State University, Andijan, Republic of Uzbekistan*
[2]*Research Institute of Semiconductor Physics and Microelectronics at the National University of Uzbekistan, Tashkent, Republic of Uzbekistan*
Corresponding Author: shuhratjonakbarov31@gmail.com

ABSTRACT: *This article presents the results of experimental studies of the morphological parameters of impurity accumulations of cobalt and nickel in silicon single crystals, as well as their decay under the influence of external all-round hydrostatic pressure, obtained using the electron probe microanalysis method. The sizes, geometric shapes, and structural structures of impurity micro- and nanoaccumulations in silicon have been studied. It has been revealed that the sequence of decay of impurity micro- and nano-accumulations under the influence of uniform hydrostatic pressure depends on their size and shape.*
KEYWORDS: *morphology, silicon, cobalt, impurity accumulations, morphology, pressure, electron probe microanalysis.*

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I. INTRODUCTION

As is known, during high-temperature doping of silicon single crystals with impurity elements of 3d transition metals, impurity accumulations with different morphological parameters are formed in the bulk of silicon [1-3]. Therefore, from the point of view of controlling the electrical parameters of semiconductor silicon, studying the structure and state of impurity micro- and nano-accumulations of elements of the transition 3d group in silicon single crystals, as well as their behavior under the influence of external influences, is of great scientific and practical interest.

The results of previous studies have shown that external pressure significantly affects the electrical properties of semiconductor silicon [4-6]. To improve the technology for producing semiconductor materials with high sensitivity to external pressure, it is necessary to study the physical laws of the processes occurring in the bulk of semiconductor materials with impurity accumulations.

The results of previous studies have shown that external pressure significantly affects the electrical properties of semiconductor silicon [7,8].To improve the technology for producing semiconductor materials with high sensitivity to external pressure, it is necessary to study the physical laws of the processes occurring in the bulk of semiconductor materials with impurity accumulations. Impurity accumulations formed due to the accelerated formation of iron deposits on these defects were discovered [9]. It has been established that the size of impurity precipitates reaches several hundred nanometers.

In this work, the morphology of cobalt and nickel impurity nanoclusters in n-type silicon and their disintegration under confining hydrostatic pressure (HCP) were studied.

II. MATERIALS AND METHODS

n-Si(Co) samples were obtained from single-crystal silicon (KEF brand, resistivity $\rho = 20\ \Omega\cdot$cm) and n-Si(Ni) samples (initial resistivity $\rho = 5\ \Omega\cdot$cm) grown by the Czochralski method. Cobalt and nickel diffusion was performed at 1523 K for 2 hours, followed by rapid cooling at a rate of 200 K/s. Comprehensive structural studies of the as-received and doped silicon samples were conducted using a Superprobe JXA-8800R electron probe

microanalyzer. To study the kinetics of impurity accumulation decay, the samples were subjected to uniform hydrostatic pressure in the range 0.1–1.2 GPa at room temperature.

Structural studies of cobalt-doped silicon single crystals revealed the formation of impurity nanoclusters with various geometric shapes and sizes within the material (Fig. 1). Electron probe analysis determined the morphological parameters of the cobalt nanoaccumulations. Cobalt nanoclusters were found to reach ~400 nm in size and exhibit a monolayer structure, appearing as needles, discs, lenses, or spheres.

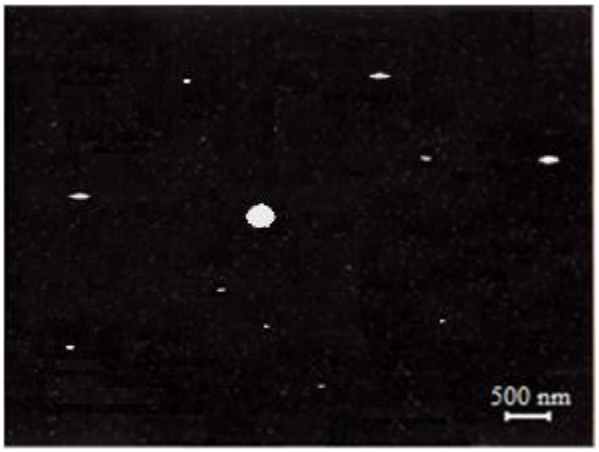

Fig. 1. General view of cobalt impurity nanoclusters in silicon.

Analysis of the chemical composition of various impurity nanoclusters formed in the volume of n-Si<Co> samples showed that the percentage of cobalt atoms throughout the entire volume of such nanoclusters is ~30% (Fig. 2). This ratio of impurity atoms and the main matrix shows that they consist of cobalt silicide $CoSi_2$ [10]. In the near-surface regions of nanoclusters, a sharp decrease in the percentage of cobalt atoms is observed.

The results of similar studies with n-Si<Ni> samples showed that impurity micro- and nano-accumulations of nickel with different morphological parameters are formed in their volume. As shown in Fig. 3, these impurity accumulations also have a needle-shaped, disk-shaped, lens-shaped or spherical shape. The sizes of impurity accumulations of nickel in silicon range from several tens of nanometers to ~2 microns. It was revealed that nickel impurity nanoclusters mainly have a monolayer structure. In contrast, impurity microclusters of nickel have a multilayer structure, i.e. such microclusters consist of two or more layers of nickel silicide and are predominantly lenticular and spherical in shape.

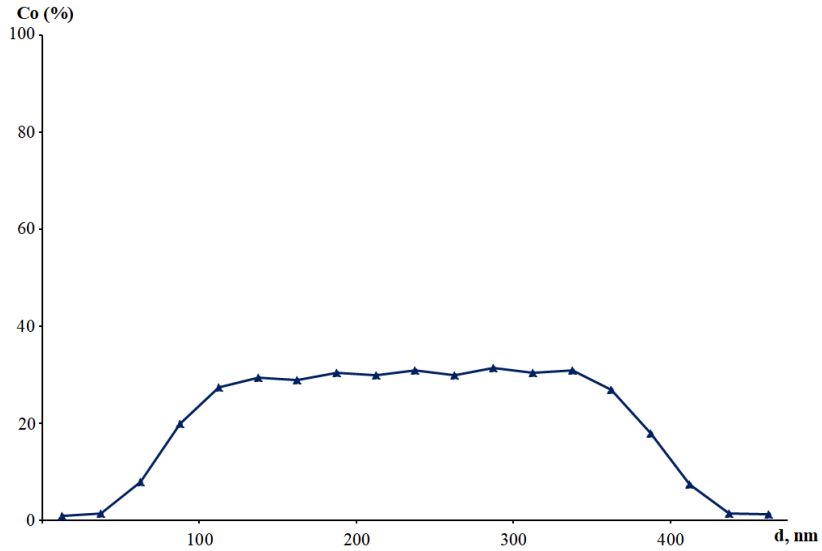

Fig. 2. Dependence of the percentage of cobalt atoms on the diameter of a spherical nanocluster with size d = 400 nm.

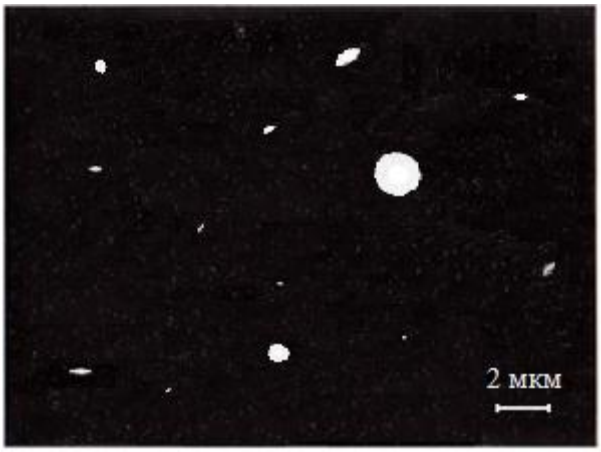

Fig. 3. General view of impurity accumulations of nickel in silicon.

Studies of the chemical composition of relatively large impurity accumulations (>1 μm) with a lens-shaped and spherical shape have shown that nickel atoms are unevenly distributed throughout their volume. In Fig. Figure 4 shows the dependence of the percentage of nickel atoms on the diameter of a spherical microcluster, the size of which is d = 1.2 μm, consisting of two layers. As can be seen from the graph, in the central layer of the impurity accumulation the percentage of nickel atoms has its maximum value and reaches up to ~30%, and in the near-surface layer this figure is 23%. Consequently, it turns out that the central layer consists of nickel silicide – Ni_2Si_5, and the near-surface layer consists of – $NiSi_3$ [10]. Depending on the shape and size of impurity accumulations, these layers can have different thicknesses.

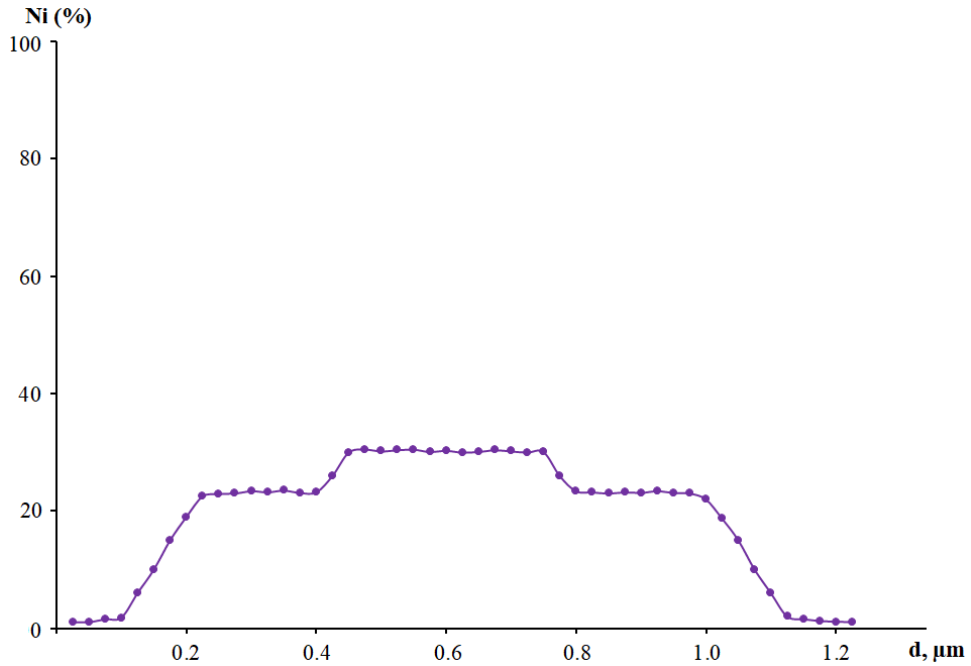

Fig. 4. Dependence of the percentage of nickel atoms on the diameter of a spherical nanoinclusion with size d = 1.2 μm.

The measured resistivity (ρ) versus hydrostatic pressure (HP) dependencies for the initial and doped samples showed significant differences. Unlike the initial samples, those with cobalt and nickel impurity clusters exhibited non-monotonic behavior. Figure 5 shows the ρ_p/ρ_0 versus HP dependencies for the initial n-Si samples (curve 1), and the doped n-Si(Co) (curve 2) and n-Si(Ni) (curve 3) samples subjected to HP in the range P = 0.1–

1.2 GPa. Resistivity measurements were performed before and after HP application. As shown, the ρ = f(P) dependence for the initial samples showed no significant changes within this pressure range (curve 1, Fig. 5).

Studies of the effect of HP on the resistivity of n-Si(Co) samples with $\rho_0 = 8 \times 10^3$ Ω·cm revealed a two-stage dependence (curve 2, Fig. 5). The first stage is observed at pressures P < 0.3 GPa, and the second at P ≥ 0.3 GPa. In the first stage, the sample resistivity decreases by ~15%. Further increase in HP leads to a second stage, characterized by a sharp increase in resistivity; at P = 0.4 GPa, it increases by almost a factor of 8. Further increasing the external pressure to P = 1.2 GPa does not significantly change the resistivity of the n-Si(Co) samples; it remains almost constant.

This behavior of resistivity change for n-Si(Co) samples is explained by an increase in the concentration of free charge carriers at P < 0.3 GPa due to the shift of energy levels caused by deformation of the silicon crystal structure, leading to a decrease in ρ_p/ρ_0. A further increase in pressure to P = 0.4 GPa causes a sharp increase in ρ_p/ρ_0, which is attributed to an increase in the concentration of electrically active nickel impurity atoms.

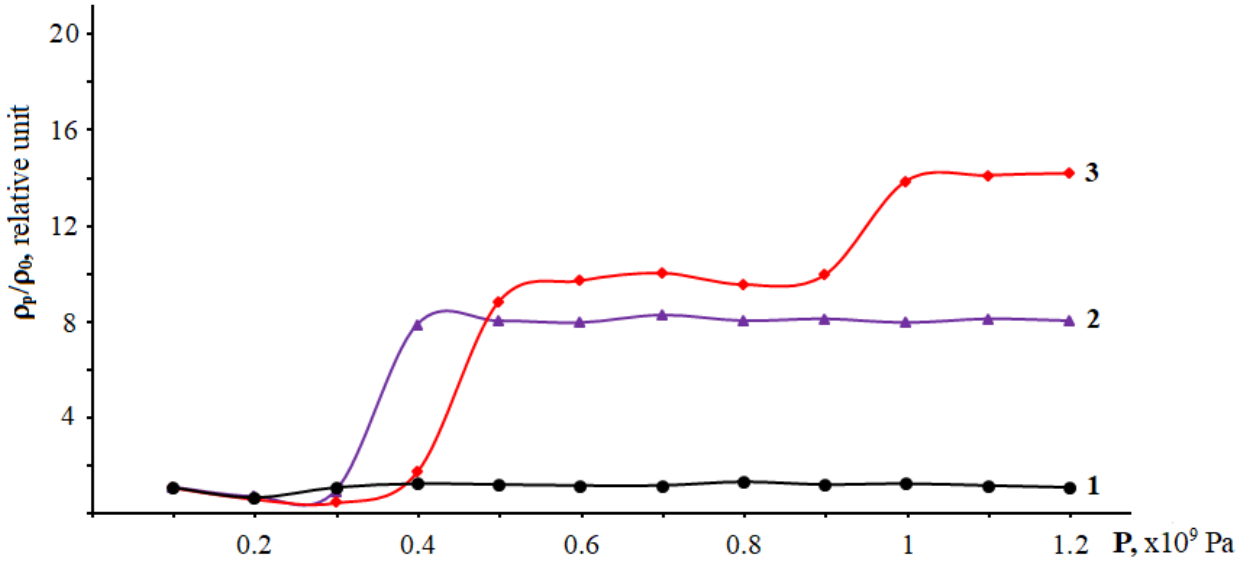

Fig 5. Fig. 5. Dependence of ρ_p/ρ_0 on HP in samples: 1 – initial n-Si; 2 – n-Si; 3 – n-Si.

Results of studying the effect of hydrostatic pressure (HP) on the resistivity of n-Si(Ni) samples showed a two-stage dependence (curve 3, Fig. 5). In the first stage, at pressures P < 0.4 GPa, a decrease in sample resistivity of ~18% was observed. In the second stage, at P ≥ 0.4 GPa, a sharp increase in resistivity occurred; at P = 0.5 GPa, resistivity increased by almost an order of magnitude. Further increasing the HP to 0.9 GPa resulted in almost no further change in resistivity. A further sharp increase in resistivity, approximately 15 times, occurred at P = 1 GPa. No significant changes were observed for higher HP values.

The observed decrease in ρ_p/ρ_0 for n-Si(Ni) samples at P < 0.4 GPa is attributed to pressure-induced crystal structure deformation, shifting energy levels, and thus increasing the concentration of free charge carriers. The subsequent sharp increase in ρ_p/ρ_0 at P ≥ 0.4 GPa is attributed to an increase in the concentration of electrically active nickel impurity atoms, associated with the decay of impurity clusters. The first significant jump in ρ_p/ρ_0 was observed between 0.4 and 0.5 GPa, and a second such jump occurred between 0.9 and 1 GPa.

To understand the nature of the resistivity changes (ρ) in n-Si(Co) samples under hydrostatic pressure (HP), comprehensive structural analyses of the impurity nanoclusters before and after pressure application were performed. Analysis revealed that the number of cobalt impurity nanoclusters (up to 400 nm in size and various shapes) within the samples drastically decreased at P = 0.4 GPa after HP application, indicating a disintegration of the cobalt nanoclusters (Fig. 6).

Comparative analysis of n-Si(Ni) samples before and after HP application showed that at P ≥ 0.5 GPa, nanoclusters disintegrated and larger nickel impurity microclusters (lens-shaped and spherical) fractured (Fig. 7). At P = 0.5 GPa, the complete disintegration of nickel nanoclusters and further decomposition of larger microclusters occurred (Fig. 7). Further HP application at P = 1 GPa resulted in the disintegration of nickel micro-inclusions. The observed irreversible nature of the ρ=f(P) dependence at P > 0.4 GPa also supports the conclusion that HP induces the disintegration of nickel impurity clusters in silicon.

Fig. 6. Images of n-Si samples after HP application at P = 0.4 GPa.

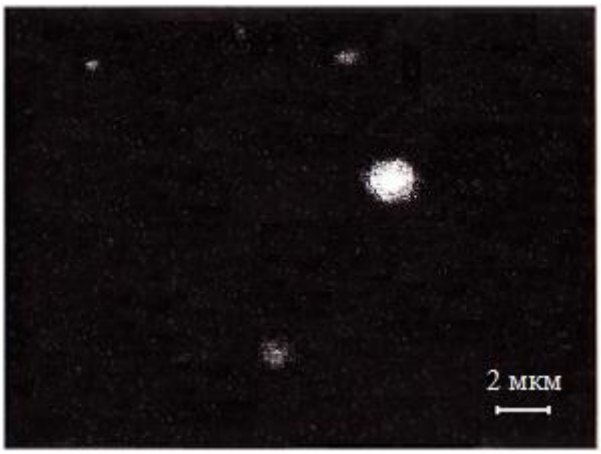

Fig. 7. Photographs of n-Si<Ni> samples after exposure to HP at P = 0.5 GPa.

Figure 8 shows the distribution diagram of cobalt atoms within a spherical impurity nanocluster (400 nm diameter) in n-Si before and after applying 0.5 GPa HP. The diagram demonstrates the disintegration of this nanocluster after the pressure application. This nanocluster, with a monolayer structure, is composed of cobalt silicide ($CoSi_2$). As shown, applying HP at 0.3 GPa (curve 1, Fig. 8) did not significantly alter the distribution of cobalt atoms within the nanocluster. A further increase in pressure to 0.4 GPa resulted in a noticeable decrease in the volumetric fraction of cobalt atoms within the nanocluster (curve 2, Fig. 8), which confirms the fracturing of the nanocluster.

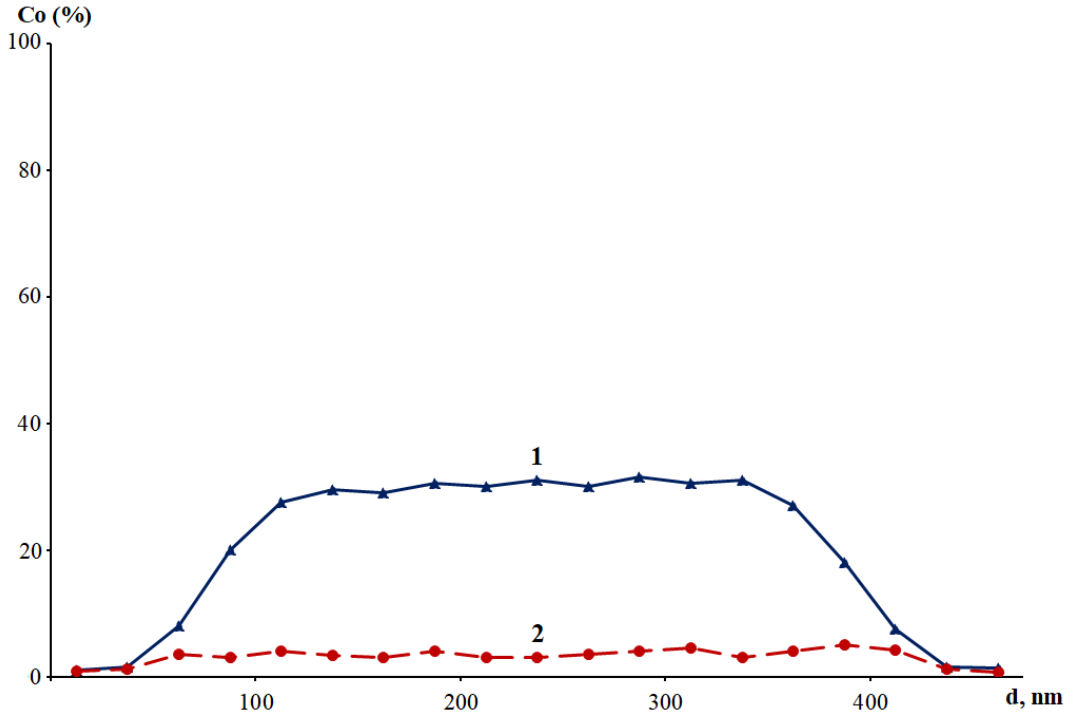

Fig. 8. Percentage of impurity atoms in the volume of cobalt nanoscale clusters in silicon under the influence of high hydrostatic pressure (HHP) at: 1 – 0.3 GPa; 2 – 0.4 GPa.

Thus, from the obtained experimental results, it has been revealed that during the process of diffusion doping of silicon with cobalt impurities at a temperature of 1523 K, impurity cobalt nanoscale clusters are formed with various geometric shapes and sizes, reaching up to ~400 nm. The percentage content of cobalt atoms throughout the volume of such impurity nanoscale clusters is approximately 30%.

III. CONCLUSION

The results of studies of the influence of HP on the resistivity of n-Si<Co> and n-Si<Ni> samples showed that these dependences consist of two stages. The first of them is observed for n-Si<Co> samples at a pressure value of P<0.3 GPa, and for n-Si<Ni> samples at P<0.4 GPa, where the ρ value of the samples decreases by ~15% and ~18%, respectively . This change in the resistivity of the samples is explained by the fact that at this stage, under the influence of pressure, the concentration of free charge carriers in them increases due to a shift in energy levels due to deformation of the silicon crystal structure, which leads to a decrease in the value of ρ_p/ρ_0 of the samples. In the second stage, for n-Si<Co> samples at a pressure value of P≥0.3 GPa, and for n-Si<Ni> samples at P≥0.4 GPa, an abrupt increase in the value of ρ_p/ρ_0 of the samples is observed. It was revealed that an increase in the value of ρ_p/ρ_0 of samples of this nature occurs due to the decay of impurity accumulations, which leads to an increase in the concentration of electroactive impurity atoms of cobalt and nickel in the volume of silicon.

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