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Formation of cobalt impurity precipitates in silicon

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Abstract**:** This article presents the results of studies of the structure and chemical composition of Co impurity precipitates in Si single crystals formed during high-temperature diffusion doping. The studies were carried out using the electron probe microanalysis method. As revealed, the sizes and shapes of cobalt impurity precipitates in silicon depend on the cooling rate of the samples after diffusion annealing. It has been established that impurity cobalt precipitates, depending on their size, can have a single-layer or multilayer structure. It was also revealed that in terms of the volume of impurity precipitates, the maximum proportion of dopant atoms, as well as technological impurities, is located in their central part.

Keywords: silicon, cobalt, precipitate, impurity, diffusion, cooling rate, cobalt silicide.

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INTRODUCTION

Studying the structure and chemical composition of accumulations of impurity atoms of 3d transition metals formed during high-temperature diffusion doping of silicon is of particular importance in controlling the electrical properties of semiconductor silicon. As is known, during diffusion doping of silicon with elements of the 3d group, depending on the diffusion parameters, various accumulations of impurity atoms with different morphological parameters are formed [1-5]. Data on the geometry and chemical composition of impurity precipitates are usually obtained from electron microscopic observations. This paper presents the results of studies of the structure and chemical composition of cobalt impurity precipitates in silicon.

MATERIALS AND METHODS

As the object of research, n- and p-type Si<Co> samples were chosen, obtained on the basis of the starting materials - monocrystalline silicon of the KEF brand, with resistivities ρ=20÷40 Ohm·cm, grown by the Czochralski method. The samples had dimensions of 10×5×2 mm in the form of a parallelepiped. Diffusion of cobalt in silicon was carried out in electric resistance furnaces of the SUOL-4M type at a temperature of 1250 ℃ for 4 hours. After diffusion annealing, the samples were cooled at different rates. For rapid cooling, the samples were quenched by dropping the ampoules into water. In this case, the cooling rate of the samples reached υ_{cool} = 200 °C/s. To obtain slower cooling rates υ_{cool} = 0.05 °C/s, the ampoules were cooled with the furnace turned off.

The study of the morphological parameters of cobalt impurity precipitates formed in the bulk of silicon was carried out by electron probe microanalysis using a JEOL electron microscope. The samples under study were attached to a brass substrate using conductive paint specially prepared on the basis of black coal. The substrate is placed in the vacuum chamber of the installation. Having received the required vacuum ($10^{-4} \div 10^{-5}$ bar), the electron source is connected. An electron beam created by a tungsten filament with an accelerating voltage of 20 kV is incident on the sample under study, and the probe current is 10 nA. When interacting with the surface of the sample, the electron beam causes secondary X-ray radiation through the diaphragm to be applied to the analyzed crystal, the reflection from which is recorded on a proportional counter. The results are processed by an electronic system.

RESULTS AND DISCUSSION

The results of experimental studies have shown that impurity precipitates are formed in the bulk of silicon single crystals doped with cobalt. They have different geometric shapes and sizes. In Figure 1 (a) shows a micrograph of impurity precipitates formed in n-Si<Co> samples with a cooling rate of υ_{cool} = 200 °C/s after diffusion annealing. In samples with rapid cooling, impurity precipitates are formed that have needle-shaped, lens-shaped, disk-shaped or spherical shapes, the maximum dimensions of which reach several hundred nanometers. The density of such precipitates by volume of the samples is ~10^3 mm^{-3}.

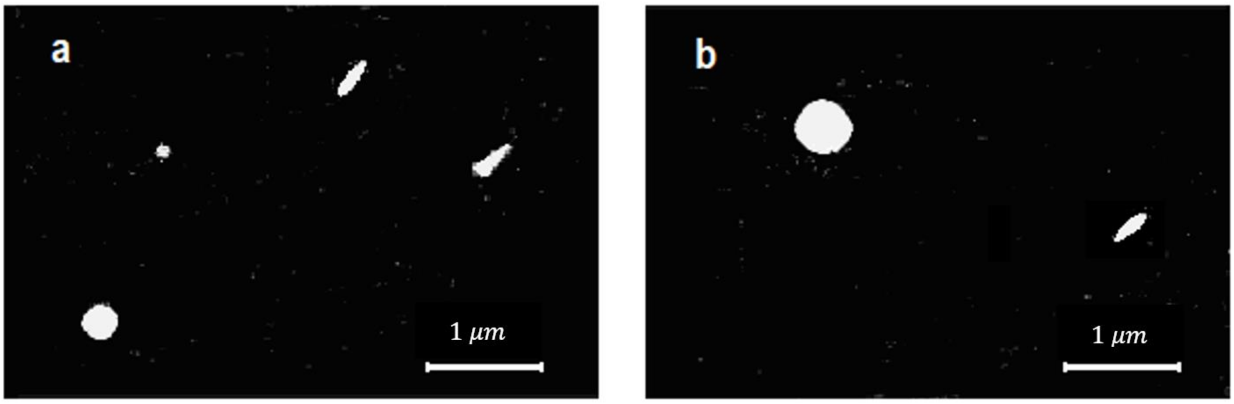

Figure 1. Photographs of impurity precipitates in n-Si<Co> samples: a – with a cooling rate υ_{cool}=200 °C/s, b – with a cooling rate υ_{cool}=0.05 °C/s.

As shown by the results of similar studies with n-Si<Co> samples obtained with a cooling rate υ_{cool} = 0.05 °C/s, after diffusion annealing, larger precipitates are formed in their volume, mainly with a lens-shaped and spherical shape. The sizes of these precipitates reach up to ~700 nm (Figure 2 (b)). The density of such precipitates over the volume of the samples is ~10^2 mm^{-3}.

The obtained images of impurity precipitates in p-Si<Co> samples with rapid cooling υ_{cool} = 200 °C/s are shown in Figure 2(a). As can be seen, the impurity precipitates formed in their volume have different shapes and sizes. The main part of these precipitates have a single-layer structure, as well as needle-, disk- and lens-shaped shapes. Their dimensions reach up to ~500 nm, and their density over the volume of the samples is ~10^3 mm^{-3}. In the volume of samples obtained with slow cooling υcool = 0.05 °C/s, precipitates with a spherical and lens-shaped shape, the sizes of which reach up to ~1.2 μm, are mainly observed. Some of these precipitates, with relatively large sizes (d≥700 nm), consist of several layers (Figure 2 (b)).

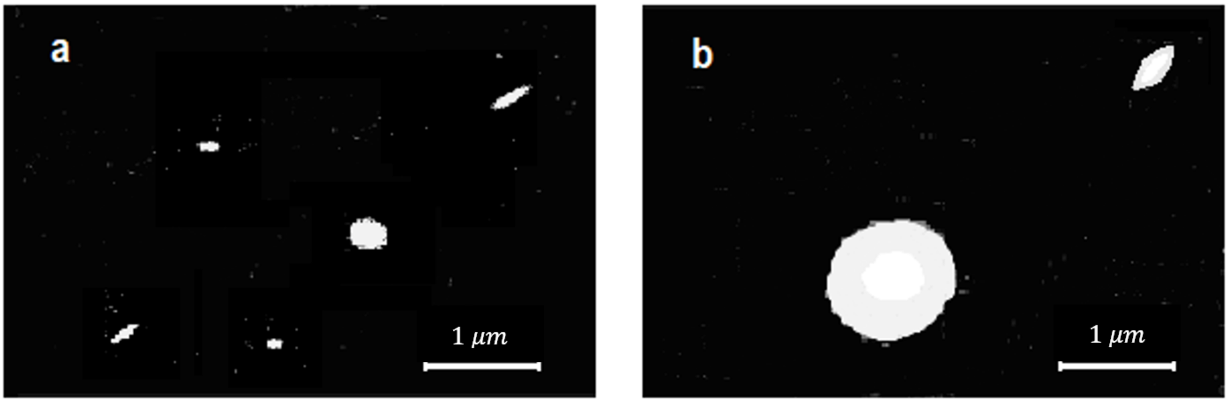

Figure 2. Photographs of impurity precipitates in p-Si<Co> samples: a – with a cooling rate υ_{cool}=200 °C/s, b – with a cooling rate υ_{cool}=0.05 °C/s.

Studies of the structure of impurity precipitates have shown that the main part of cobalt precipitates with larger sizes (≥700 nm) have a multilayer structure. Micrographs obtained using an electron probe microanalyzer show that such precipitates consist of two or more silicide layers. They clearly distinguish the boundaries between the silicide layers of cobalt. In contrast, precipitates with relatively small sizes (<700 nm) have a single-layer structure.

The results of analyzes of the chemical composition of needle-shaped and disk-shaped precipitates in n-Si<Co> samples showed that impurity cobalt atoms are distributed almost uniformly throughout the volume of such precipitates. In Figure 3 shows a graph of the percentage of cobalt atoms depending on the length of the major axis of the lens-shaped precipitate. As can be seen from the volume of this precipitate, the percentage of cobalt impurity atoms is 30÷33%, and in the near-surface regions of the precipitate this value sharply decreases. This ratio of cobalt impurity atoms and the main element silicon shows that such precipitates consist of cobalt silicide $CoSi_2$ [6].

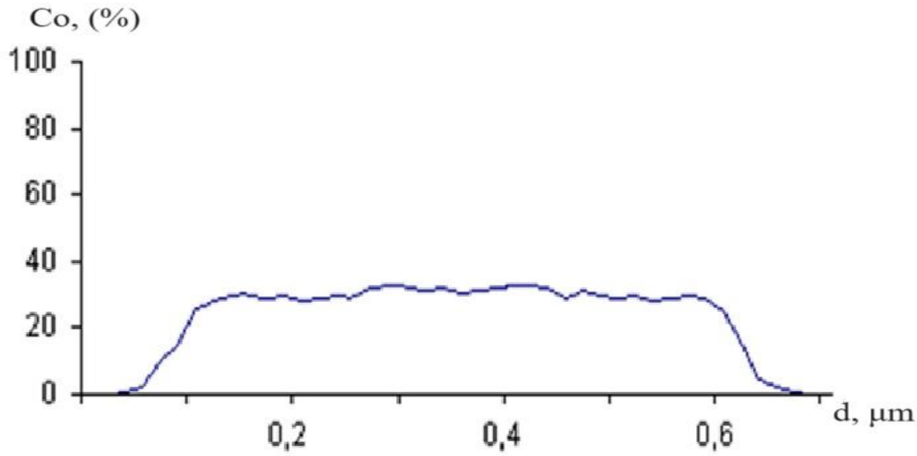

Figure 3. Distribution of impurity cobalt atoms over the volume of a single-layer lens-shaped precipitate in n-Si<Co> samples.

The results of studying the structure of relatively large cobalt precipitates with a size of d≥700 nm, having lens-shaped and spherical shapes, showed that they consist of several layers of Co_xSi_y-type silicides. Depending on the shape and size of the precipitate, these layers can have different thicknesses.

It should be noted that when studying the processes of formation of impurity precipitates in the bulk of semiconductor materials, during high-temperature diffusion doping with various impurities, special attention was not paid to the role of technological impurity atoms in this process, whose diffusion coefficient is much higher than that of the main dopant impurities. However, in the literature there is some information indicating the possible participation of atoms of technological impurities in the process of formation of impurity precipitates [7,8].

The results of comprehensive studies of quantitative indicators of the elemental composition of various impurity precipitates confirm that, by volume of cobalt precipitates in silicon, there are atoms of technological impurities. The results of studies of the distribution of atoms of technological impurities of iron and copper over the volume of a lens-shaped precipitate with a size of d = 600 nm formed in the volume of the n-Si<Co> sample are shown in Figure 4. From the graphs shown in Figure 4 it can be seen that the maximum percentage of Fe atoms by volume of the precipitate is ~0.2%, and for Cu atoms it is ~0.15%. As was observed, in the near-surface regions of the precipitates, the percentage of Fe and Cu atoms is significantly lower compared to this value in the volume of the precipitate.

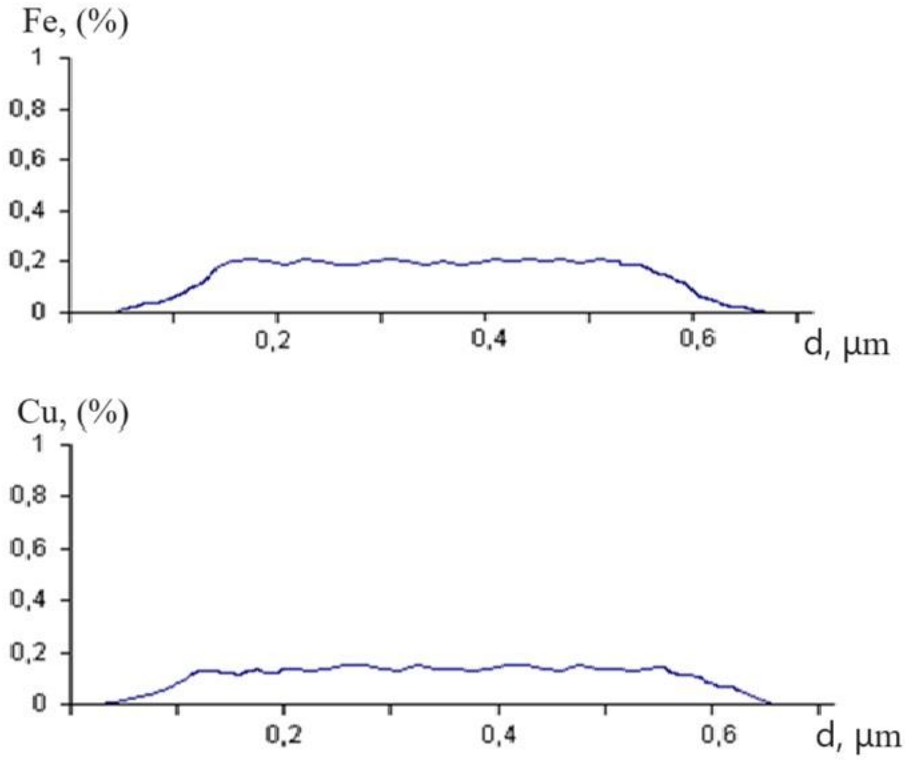

Figure 4. Distribution of atoms of Fe and Cu technological impurities over the volume of a single-layer lens-shaped precipitate in n-Si<Co> samples.

In Figure 5 shows a graph of the dependence of the percentage of cobalt atoms on the diameter of a spherical precipitate with a diameter of d=1 µm, which consists of two layers. As can be seen from the graph, in the surface layer of such precipitates the percentage of cobalt atoms is ~30%, and in the central part ~50%. Therefore, it turns out that the central layer of this precipitate consists of CoSi silicide, and its near-surface layer consists of $CoSi_2$ silicide.

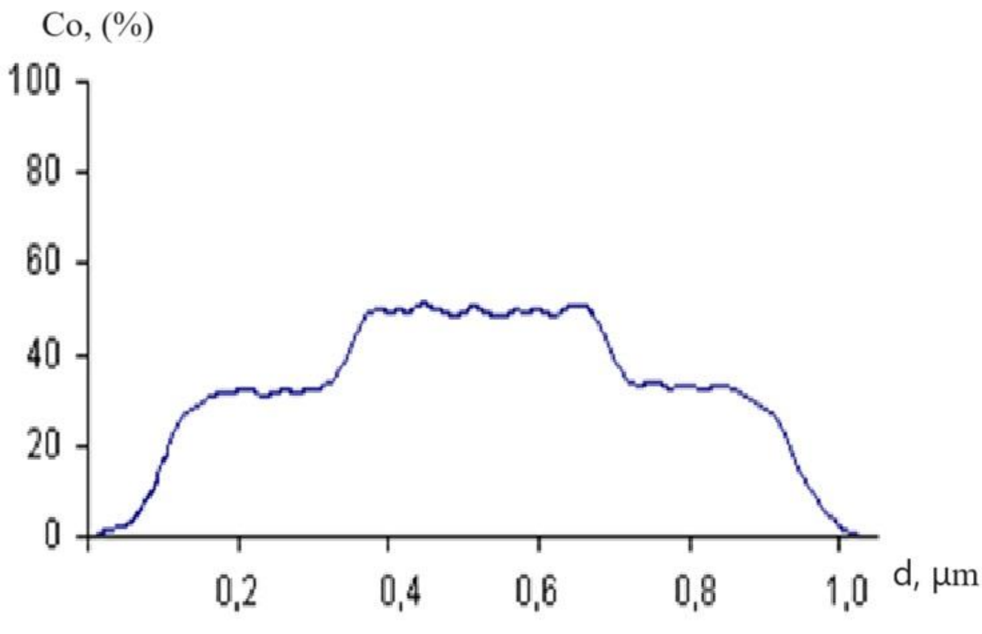

Figure 5. Distribution of cobalt impurity atoms along the diameter of a two-layer spherical precipitate with a diameter of d=1 µm, formed in the p-Si<Co>

sample.

The study of the chemical composition of impurity precipitates with relatively large sizes (d≥700 nm) with a spherical shape showed that in their volume there are also atoms of technological impurities Fe, Cu, etc. In the course of analyzes of the elemental composition over the volume of precipitates, it was found that in the distribution of atoms technological impurities, there is a certain pattern in the diameter of the precipitates. When analyzing each silicide shell separately, it turned out that the maximum fraction of atoms of uncontrolled impurities is located in the central part of the precipitates. In Figure 6 shows experimental data on the distribution of atoms of technological impurities of iron and copper over the volume of a spherical precipitate with a diameter of d=1 μm formed in the volume of the p-Si<Co> sample. This precipitate has a two-layer structure. As can be seen, the percentage of Fe atoms in the central layer of the precipitate is ~0.35%, and in the near-surface layer of the precipitate this value is ~0.1%. The percentage of Cu atoms in the central part of the precipitate is ~0.25%, and in the near-surface layer ~0.05%. The data obtained show that in each layer of the precipitate the technological impurity atoms of Fe and Cu are distributed almost evenly, but their values differ significantly from each other.

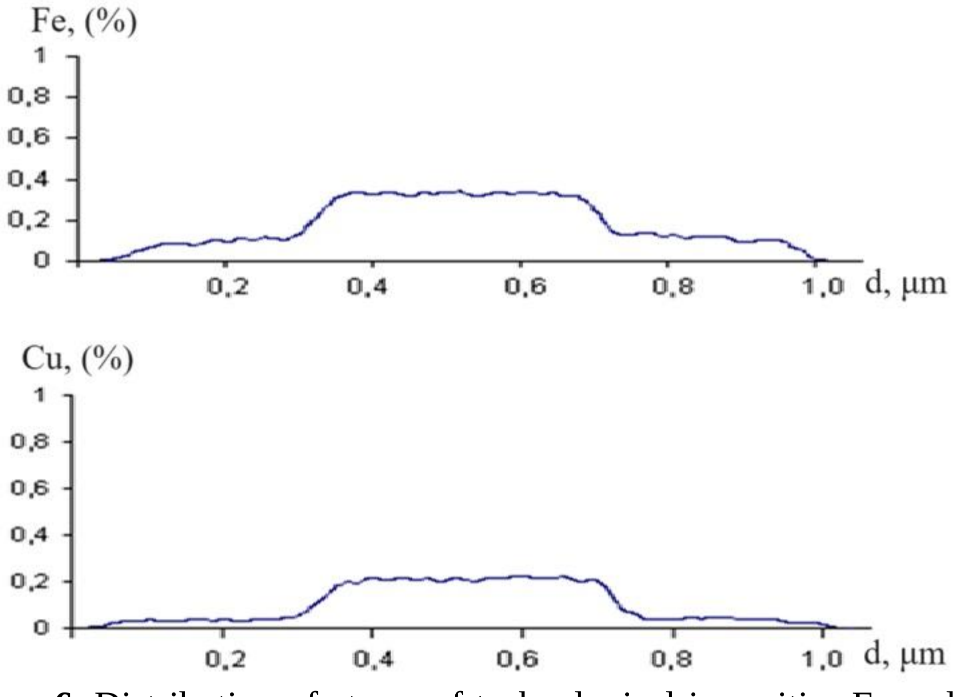

Figure 6. Distribution of atoms of technological impurities Fe and Cu over the diameter of a spherical precipitate of size d=1 μm, formed in the p-Si<Co> sample.

CONCLUSIONS

Thus, it was revealed that when diffusion doping of cobalt in silicon at a temperature of 1250 ℃ for 4 hours, impurity precipitates with different morphological parameters are formed in the bulk of the samples. Based on the results obtained from studying the chemical composition of precipitates in Si<Co> samples, it was established that the number and distribution of impurity atoms over the volume of precipitates depends on their size and shape. The geometric shapes and sizes of impurity precipitates, therefore, depend on the cooling rate of the samples after diffusion annealing. In samples with high cooling rates, impurity precipitates of various shapes are formed, with relatively small sizes (<700 nm) and they consist of one type of cobalt silicide. At low cooling rates, spherical precipitates with sizes up to ~1.2 μm, which have a multilayer structure, are mainly formed. It was found that in the volume of impurity precipitates there are atoms of technological impurities such as Fe, Cu, etc. It was also found that in the volume of multilayer precipitates, the value of the percentage of atoms of both the main impurity Ni and technological impurities Fe and Cu decreases in the direction from the center to the surface of the precipitate.

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