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INVESTIGATION OF SCANDIUM ALLOY ON THE STRUCTURE OF THE SILICON SURFACE LAYER

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Abstract

The technology of step-by-step low-temperature diffusion of scandium into silicon is developed, allowing to create clusters of impure atoms uniformly distributed over the entire volume of material. It is shown that unlike the samples obtained by high-temperature diffusion alloying, the samples produced by the new technology do not have surface erosion, the formation of alloys and silicide's in the near-surface area. The thermal and radiation stability of silicon samples containing clusters of impure scandium atoms has been increased.

Complex (methods of tagged atoms, autoradiography, conductivity measurement and Hall effect, isothermal relaxation of capacitance and current) diffusion research were conducted, solubility and electro physical properties of scandium in silicon at different annealing media and for a wide temperature range (1100÷1250$0^0$C). Diffusion parameters, solubility and acceptance nature of studied impurity in silicon, thermal stability of silicon doped with impure atoms of scandium have been determined.

Keywords

scandium, technology, cluster, erosion, low-temperature diffusion, doping, thermal stability, degradation, diffusion, tagged atoms, autoradiography, electrical conductivity, Hall effect, isothermal and current relaxation, thermal stability of silicon.

Introduction

At present, semiconductor physics is actively developing in the world, where close attention is paid to obtaining a new type of material by forming clusters on the surface and in the volume of silicon, changing its fundamental parameters. Development of the technology of formation of clusters of impure atoms, which

allows to create Nano scale structures in the volume of a crystal with a sufficiently high concentration and specified composition, structure and physical parameters, is one of the main tasks of modern microelectronics [1-5].

Semiconductor materials with formed internal structures, especially with clusters of impure atoms, are of great scientific and practical interest. In such materials, a number of new physical phenomena have been discovered, making a great contribution to the development of modern micro- and Nano electronics. The formation of clusters of impurity atoms of rare earth elements [6-12] is of particular interest in this regard.

The urgency of the work is connected with the existence of a number of unresolved issues from the point of view of the development of technology for the formation of clusters of impure atoms of rare earth elements with controlled structure and properties in a silicon grating, which is one of the actual and prospective tasks of modern Nano electronics. The creation of clusters of impurity atoms of rare earth elements in silicon allows not only to control the fundamental parameters of silicon, its magnetic properties, but also to reveal a number of new still unknown physical phenomena in it, the use of which opens up new opportunities for creating instrument structures with unique technical characteristics.

Research techniques

By implementing the technique of microprobe analysis (Jeol Super Probe JXA-8800 R/RL), IR microscopy (INFRAM-I), and AFM (Dimension 3000) the experimental team had studied the process of forming of clusters of atoms of scandium, as well as changes in their structure, size, and density. The electrical parameters of the samples were measured using the four-probe method and the Hall effect technique. Based on theoretical calculations, the authors have determined the concentrations of impurity atoms participating in cluster formation.

The state of impurity atoms of scandium in the bulk of the silicon crystal lattice was studied using an infrared microscope INFRAM-I. The INFRAM-I infrared microscope makes observes not only the surface of a silicon sample, but also practically all its layers, i.e., allowing to examine the sample throughout its entire bulk.

To measure the lifetime of minority charge carriers, the technique of transient processes of a sinusoidal current of large amplitude was used. The choice of this technique was motivated by the fact that the Si<P,Sc> samples, as well as the reference samples, had changed their resistivity in a fairly wide range during heat

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treatments. Therefore, the use of standard pulse methods made it difficult to obtain reliable information. The measurement error was in the range more than 5%.

Phosphorus doped and boron doped single-crystalline silicon samples of both n- and p-type conductivity, grown by Czochralski technique (concentration of residual oxygen was in the range of $6*10^{17}$ cm^{-3}, resistivity in the range ρ~1÷100 Ohm·cm) were taken as reference samples. The thickness of original silicon samples was 380 µm. Scandium were used as dopant elements. Doping of the samples was carried out in evacuated quartz ampoules from a thin metal layer deposited in vacuum on the surface of silicon samples. Reference silicon samples (without scandium atoms) were annealed in separate ampoules under similar conditions in order to evaluate the effect of diffusion doping on electrical parameters of the samples. Mechanical and chemical processing of all samples was carried out under identical conditions.

To study the silicon doped with scandium, the samples were prepared as follows. For electro physical studies, diffusants happened to be stable isotopes of metallic scandium or salts of trivalent scandium chloride. A metal layer of the scandium radioactive isotope ^{46}Sc was deposited on the surface of n-type silicon with a resistivity ρ= 15 Ω ·cm, or a layer was deposited from a solution of scandium chloride labeled with the radioactive isotope ^{46}Sc.

Diffusion doping was carried out on open air, in evacuated quartz ampoules (~ 10^{-5} ÷ 10^{-7} mmHg), as well as in evacuated ampoules in argon atmosphere in the temperature range of 1100÷1250^{0}C. The duration of diffusion doping varied depending on the diffusion temperature from 5 to 72 hours. After diffusion doping, the samples were repeatedly washed in hydrofluoric acid, aqua regia, and also in a boiling mixture of H_2O_2:HCl. After that, the edges of the sample were etched down to a depth of ~100 µm, thus significantly exceeding the diffusion depth of ~10 µm.

The diffusant profile was determined by the method of sectioning, i.e., etching thin layers (in a solution of HF: HNO_3 = 1:50 with washing in a mixture of H_2O_2 : HCl) and measuring the residual activity of the sample. The residual activity of the sample was measured using a UMF-1500M low-background unit with an SBT-11-type β-counter. Identification of ^{46}Sc spectra was also carried out by means of measurements on an AI-1024-type pulse analyzer. The thickness of the removed layers (0.05÷0.5µm) was determined by intermittent weighing the sample. The autoradiograms obtained before and after doping, as well as during the removal of layers, indicated a uniform distribution of Sc impurities over the cross section of the sample and the absence of embedded inclusions.

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The Figure 1 shows an analytical growth chamber was shown, where 1, 2, 3 are electron-beam evaporators; 4-effusion source; 5-sample; 6-manipulator; 7-heater; 8-thermocouple; 9-electron gun of the fast electron diffractometer; 10-fluorescent screen; 11 - a quadrupole gas analyzer; 12-cryopanel; 13.14 - deposition rate sensors (quartz); 15,16 - dampers; 17-ion pump; 18-control dampers with a servo drive; 19-power supplies of electron-beam evaporators; 20 - processor unit.

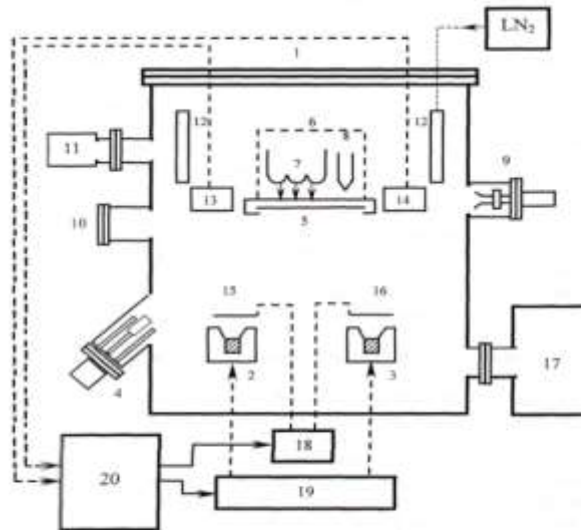

Fig. 1. Analytical growth chamber: 1, 2, 3 - electron beam evaporators; 4-effusion source; 5-sample; 6-manipulator; 7-heater; 8-thermocouple; 9-electron gun of the fast electron diffractometer; 10-fluorescent screen; 11 quadrupole gas analyzers; 12-cryopanel; 13, 14- deposition rate sensors (quartz); 15, 16 dampers; 17-ion pump; 18-control dampers with a servo drive; 19-power supplies of electron-beam evaporators; 20-processor unit.

The sample 5, mounted on a molybdenum holder, was placed inside the chamber on a manipulator 6. The manipulator was equipped with a heater 7 and a thermocouple 8, which made it possible to control the sample temperature in the temperature range from room temperature up to 1000^{0}C with an accuracy of ±0.5^{0}C. To ensure uniform heating of the sample and uniform deposition of evaporated materials, the holder with the sample was rotated using an electric motor.

The growth chamber was equipped with a reflection high-energy electron diffractometer, which made it possible to analyze the structure of the crystal surface directly in the course of growth. The diffractometer included an electron gun 9 and a luminescent screen 10.

Quadrupole gas analyzer 11 was used to control the composition of the residual gas atmosphere. The grown epitaxial structures were studied in the

analytical chamber by Auger electron and secondary ion mass spectrometry. The vacuum in the system was maintained at the level of $5 \cdot 10^{-9}$ Pa.

Results and discussion

Having investigated the clusters of impurity atoms of scandium, the authors have noted that this is not just a random accumulation of a certain number of atoms of some impurity somewhere in the lattice. This is a local region of a semiconductor enriched with impurity atoms, which, as a rule, is characterized by a clearly defined structure with an ordered arrangement of both impurities and the main atoms of the lattice. Impurity atoms in a cluster themselves enter a sublattice with a certain arrangement of atoms and ions, which is correlated with the semiconductor lattice, which explains their relatively high stability.

Under external influence, clusters of impurity atoms can change their state. The search for ways to control the state of clusters and their ordering in the crystal lattice of a semiconductor is of great scientific and practical interest. This is due not only to the chance of creating a bulk nanostructured semiconductor material, but also to obtaining new types of photonic materials and bulk super lattices with specified parameters. The study of semiconductor materials with an ordered distribution of impurity atom clusters as a whole makes it possible to determine their unique functionality.

The essence of the "low-temperature diffusion doping" technique developed by us is as follows. The studied samples and diffusant are placed into evacuated quartz ampoules (pressure in the ampule $\sim 10^{-6}$ mmHg). They are placed in a diffusion furnace at T=300K. It has been established in advance that the temperature of the furnace at the location of the ampoule gradually increases at a rate of 5 deg/min. Thereafter, the samples are heated to a temperature of T= ($550 \div 700\ ^0$C) and kept at this temperature for t = ($10 \div 20$) min, then the furnace temperature is made to rise quickly (at the rate of $150 \div 200$ deg/min) to a certain temperature (T=$1200 \div 1250\ ^0$C) and at this temperature the samples are kept for a while, after which the ampoules are removed from the furnace and cooled at a rate of 200^0/sec.

Based on the experimental results, it can be argued that at low temperatures, diffusion actually occurs via interstices, and impurity atoms are in interstitial states. An additional proof of this assumption can be the fact that at low-temperature diffusion, the calculated concentration of vacancies will be $N_v \sim 10^7$ cm^{-3}, i.e., almost 7-8 orders of magnitude less than the concentration of impurity atoms in the interstices.

One of the most important conclusions stemming from these experimental results is that the diffusion mechanism and the concept of solubility of impurity atoms under conditions of low-temperature diffusion differ significantly from diffusion at high temperatures, and the basic diffusion parameters and solubility obtained in this case require significant correction.

As the experiments showed, the new technology, in addition to the scientific novelty described above, has a number of important practical points alike: the diffusion process duration is reduced by 2-2.5 times, the power consumption for diffusion is reduced by 2 times, the formation of various alloys, silicide, both on the surface and in the near surface area and surface erosion are almost completely eliminated. All the above advantages of the new method make it possible not only to simplify the technology of obtaining samples, but also to form clusters of impurity atoms. Here, the concentration of rare earth elements gradually decreases towards the interface with silicon, which is explained by the escape of rare earth ions from the film into the silicon substrate due to diffusion. As a result, the shape of the spectrum of scattered neutrons also noticeably changes, where the peak of rear earth elements becomes lower and that of oxygen slightly higher (Figure 1).

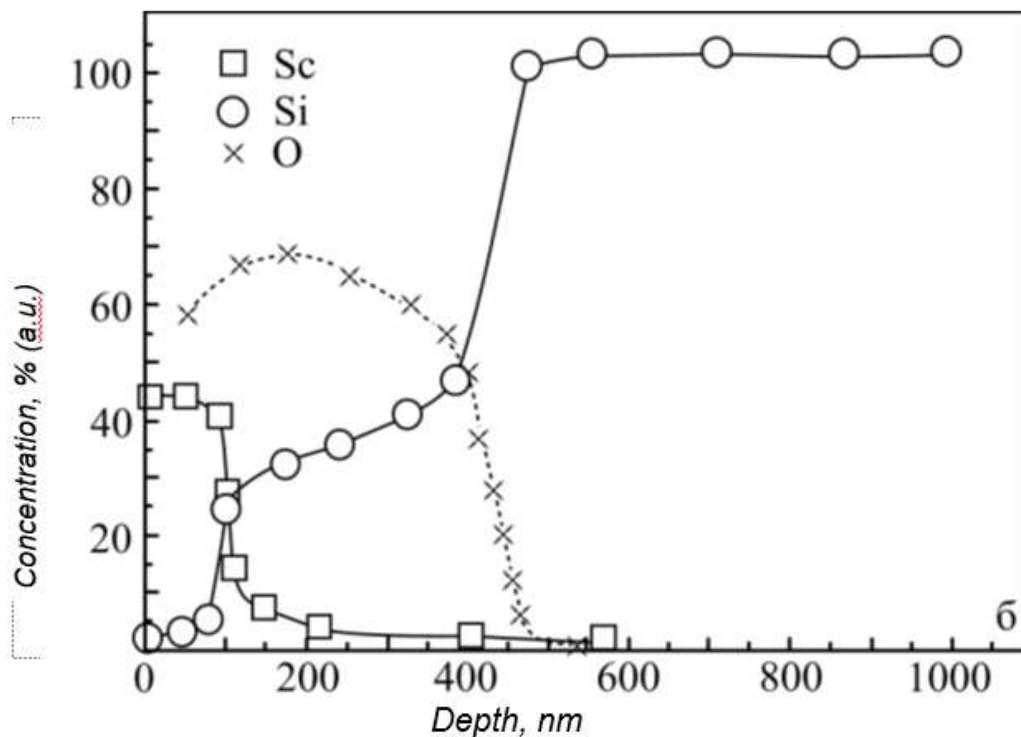

Fig. 1. Concentration profiles of Sc in silicon samples subjected to high-temperature annealing

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From the analysis of concentration profiles given in figure. 1, it can be determined that the total thickness of the mixed oxide layer for such structures is from 0.1 to 0.6 μm, and the thickness of rare earth element layers is noticeably smaller from 0.05 to 0.1 μm, which corresponds to the thicknesses of the original rare earth element layers. At greater depths of silicon, the oxygen enriched layers and rare earth elements are reduced.

The concentration of holes for all rare earth elements studied ranges from 10^{14} to 10^{17} cm^3, which is significantly less than the concentration of the elements themselves. Thus, it can be concluded that a significant proportion of rare earth elements are in the thin tenth fraction of the micrometer of the surface layer of silicon in the electrically inactive state.

In general, it was found that the total rare earth content in the surface layers of silicon, according to the elemental analysis, is quite high (up to 10^{20} cm^3), significantly higher than the values usually obtained when diffusing rare earth elements into silicon. At the same time, the concentration of electrically active acceptor impurities determined from electrophysical measurements is at least an order of magnitude lower (no more than 10^{19} cm^3). That is, a significant proportion of rare earth element atoms are electrically inactive in the surface layer of silicon.

Thus, the traditional mechanism of impurity diffusion through vacancies, which is usually involved in explaining the high temperature diffusion of rare earth elements in silicon, clearly does not take into account all the characteristics of the behavior of a rare earth element impurity in silicon at high temperatures. Based on the analysis of the results of the conducted studies, the assumption was made, That in the high-temperature treatment of silicon structures with rare earth films on the surface, one of the dominant processes is the simultaneous diffusion of rare earth elements and oxygen from rare earth film into silicon, as evidenced by the high oxygen content in the surface layer of silicon. In this case, they appear to interact with the formation of complexes in the silicon crystal lattice. Another possible process is the interaction of rare earth elements with silica to form a silicide or silicate phase, although X-ray analysis does not show the presence of such phases in significant quantities.

Conclusion

The technology of step-by-step low-temperature diffusion of scandium into silicon is developed, allowing to create clusters of impure atoms of scandium uniformly distributed over the entire volume of the crystal. Such clusters allow not only to control the properties of semiconductor material, but also allow to use such materials in the development of new designs of semiconductor devices. The results

of the surface examination of the samples after diffusion showed that, unlike the samples obtained by high-temperature diffusion alloying, there was no surface erosion in the samples produced by the new technology, formation of alloys and silicides in the near-surface area. It has been determined that, in a low-temperature stage diffusion, the diffusion temperature and time influence not only the penetration depth of the impurity, but also the size of the forming clusters.

The complex study of the physical properties of silicon containing ordered clusters of impure Scandium atoms may contribute to the discovery of new physical phenomena that have not been found only in alloyed semiconductor materials, but also semiconductors with nanostructures. Control of the state and distribution of clusters of impure scandium atoms in a silicon crystal lattice allows to create a new class of semiconductor material with unique functional capabilities, and create a new class of optoelectronics and microelectronics.

The obtained experimental data show that scandium atoms in interdimensional unstable states in the lattice, in the process of low temperature alloying, tend to a more energetically advantageous i.e. to a quasi-equilibrium state, i.e. interact with cluster formation. In this case, the crystal gets rid of single scandium atoms, and the system goes into the most energetically advantageous state. The formation and interaction of clusters, due to gadolinium atoms, substantially relieves strain in the crystal. In this case, the main stimulant are both oxygen atoms and other lattice defects, respectively, the formation of clusters of impure atoms scandium has a strong heterogeneous effect, significantly reducing the concentration of uncontrolled contaminant crystal, and suppresses the formation of thermo donors.

Analysis of the obtained results shows that diffusion coefficients and energy of activation of scandium impurities in silicon are in the range of values characteristic for diffusion of typical elements of the III group, as well as for other EREs which are impurities of substitution and diffusion on units of crystal lattice. This suggests that the Scandium atoms, the same group, are also an impurity of substitution and diffuse like other REs along the nodes of the silicon crystal lattice. Analysis of the obtained results on formation of clusters of impure atoms of scandium also showed that it is possible to move the received clusters to the right place.

Comparison of the data obtained with early results on diffusion of REE in silicon obtained by radioactive and others. It shows that the diffusion method and diffusion environment do not significantly affect the diffusion parameters of REE in silicon. Thus, it can be assumed that REE scandium impurities - elements of the third group, investigated by us, are also impurities of substitution and diffuse

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along the nodes of the crystal lattice (vacant diffusion mechanism) of silicon.

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