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CHEMISTRY AND CHEMICAL TECHNOLOGY

EFFECTS OF PRESSURE ON OXYGEN CONCENTRATION IN SILICON SINGLE CRYSTALS

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Abstract: *Study the residual effects after all-round hydrostatic pressure (AHP) of the original and doped with nickel and gadolinium silicon samples, and study the effect of all-round hydrostatic compression on the relaxation characteristics of metal-semiconductor structures fabricated based on crystalline silicon with different resistivity. It has been shown that in n-Si with* $N_0 \approx 8 \cdot 10^{17} cm^{-3}$ *AHP up to 40 kbar will not change the peaks of oxygen and carbon absorption, while in n-Si<Ni> at P≥30 kbar, the oxygen and carbon absorption peaks gradually decrease and completely disappear at pressure of P=55 kbar; in n-Si<Gd>, the absorption spectra turned out to be more resistant to external pressure.*

In the range from 12 kbar to 38 kbar, n-Si<Ni> sample under the influence of pressure had nonmonotonic change in resistivity, with the formation of a maximum at P ≥ 35 kbar, which is associated with impurity precipitates by two acceptor levels (E_v+0.2 eV and E_c–0.4 eV). The experimentally observed non-monotonicity in the dependences ρ=f (P) is consequence of two counter processes. Mechanical stresses that stimulate the gettering of thermal defects from the bulk of the semiconductor or impurities localized in the metal-semiconductor transition layer and interacting with surface states can be responsible for changing the properties of the interface under pressure.

Keywords: *silicon, oxygen, diffusion, infrared spectrophotometer, carbon, active atoms, hydrostatic pressure, precipitates.*

INTRODUCTION. It is known that silicon single crystals obtained both by the pulling method and by zone melting contain a significant amount of oxygen atoms ($\sim 10^{18}\ cm^{-3}$) and carbon atoms ($\sim 10^{17}\ cm^{-3}$), which are mainly in an electrically inactive state [1]. At the same time, various external influences, such as heat treatment, irradiation with ionizing and high-energy particles, necessary both for obtaining semiconductor structures and inevitable during their operation, lead to the formation of a number of electrically active centers with the participation of oxygen and carbon atoms, which play a decisive role. in the degradation of the parameters of silicon and products based on it. Along with such traditional types of external influences as illumination, heat treatment, and irradiation with ionizing particles, in recent years there has been increased interest in studying the effect of high pressures on the properties of semiconductors, in particular, in the nature of the residual effects of this influence.

The work [1] studied the behavior of oxygen atoms before and after the confining pressure (HP) P=60÷80 kbar in the temperature range 600÷800°C. The authors point out that as a result of plastic deformation of silicon single crystals under a pressure of P=70 kbar and a temperature of

800°C, all optically active oxygen passes into an optically inactive state, as a result of which the absorption peak at 9.1 µm completely disappears, and after thermal annealing at 1100°C within 1÷10 seconds is restored back.

Also in [2], the authors state that in silicon single crystals grown by the Czochralski method, oxygen can be found not only as a Si-O-Si quasi-molecule, but also in various accumulations (precipitates) of SiO_2, which are formed during the growth of an ingot. It is also shown that in some crystals grown, the oxygen content in various clusters can reach up to 20% of the total oxygen concentration. It is also shown that in some crystals grown, the oxygen content in various clusters can reach up to 20% of the total oxygen concentration.

MATERIAL AND METHODS. To dissolve oxygen accumulations and transfer all oxygen to an optically active state, it is necessary to anneal the crystal at a temperature of T=1350°C for t=20 hours. However, after such annealing, during crystal cooling, a small part of oxygen is still complexed on various impurities and lattice defects, which are usually present in silicon single crystals grown by the Czochralski method.

On the other hand, high hydrostatic pressure has a significant effect on some physical parameters of single crystals, as well as on the diffusion coefficient and the solubility of impurities in them. For example, in [3-5], the authors studied the effect of high-pressure thermal annealing on the state of optically active oxygen. It is shown that the heat treatment of a single crystal of silicon with a high content of oxygen, grown by the Czochralski method, in the range from 650 ÷ 800°C at an all-round pressure (up to 70 kbar) for one hour leads to a significant (more than an order of magnitude) decrease in the intensity of the main (1106 cm^{-1}) IR bands - absorption of interstitial oxygen.

Moreover, the results of these experiments did not depend on the method of doping the silicon single crystal with oxygen. It was also established in these works that the decrease in the concentration of optically active oxygen atoms mainly depends on the strain rate. So far, the number of such studies devoted to the study of the mechanism of such transformations is limited.

The aim of this work was to study the residual effects after hydrostatic pressure (RHP) of the original and doped with nickel and gadolinium silicon samples, as well as to study the effect of hydrostatic compression on the relaxation characteristics of metal-semiconductor structures fabricated based on crystalline silicon with different resistivity.

Samples of n-type silicon with initial resistivity ρ=5÷20 Ohm•cm were studied. The n-Si<Ni> samples were obtained by nickel diffusion into silicon single crystals, and the n-Si<Gd> samples were obtained by doping during single crystal growth. Nickel diffusion and other technological cycles were carried out according to the procedure described in [6]. The samples thus obtained were subjected to all-round compression in the range P=1÷70 kbar. The samples were compressed at the Institute of High Pressure Physics of the Russian Academy of Sciences (Troitsk). At the same time, the geometric dimensions and surface quality of the samples were identical.

The IR light transmission spectra of the samples were measured using an infrared spectrometer UR-20. The concentrations of IR-active oxygen (N_O) and carbon (N_C) were determined by the magnitude of the absorption peaks at λ=9.1 µm and λ=16.4 µm, respectively, according to the formulas:

$$N_0 = 4{,}8 \cdot 10^{17} \left(\frac{\alpha_1 d - \alpha_2 d}{d_0}\right) \tag{1}$$

$$N_C = 1{,}1 \cdot 10^{17} \left(\frac{\alpha d}{d_0}\right) \tag{2}$$

Where:

α – is the absorption coefficient,

d_0 – is the sample thickness.

Before and after the removal of pressure and appropriate processing of the indicated samples, the transmission spectra of IR light were studied on an infrared spectrometer UR-20.

Table 1

Concentration of optically active oxygen atoms at different pressures

P, kbar	After HT, at T=1550K n-Si	N-Si <Ni>
0	$8 \cdot 10^{17}\ cm^{-3}$	$8 \cdot 10^{17}\ cm^{-3}$
4	$8 \cdot 10^{17}\ cm^{-3}$	$8 \cdot 10^{17}\ cm^{-3}$
8	$8 \cdot 10^{17}\ cm^{-3}$	$7{,}8 \cdot 10^{17}\ cm$
12	$7{,}8 \cdot 10^{17}\ cm^{-3}$	$7{,}6 \cdot 10^{17}\ cm^{-3}$
16	$7{,}8 \cdot 10^{17}\ cm^{-3}$	$7{,}2 \cdot 10^{17}\ cm^{-3}$
20	$7{,}7 \cdot 10^{17}\ cm См^{-3}$	$6{,}6 \cdot 10^{17}\ cm^{-3}$
30	$7{,}6 \cdot 10^{17}\ cm^{-3}$	$6{,}3 \cdot 10^{17}\ cm^{-3}$
40	$6{,}6 \cdot 10^{17}\ cm$	$5{,}3 \cdot 10^{17}\ cm^{-3}$
50	$5{,}3 \cdot 10^{17}\ cm^{-3}$	$3{,}6 \cdot 10^{17}\ cm^{-3}$
55	$4{,}7 \cdot 10^{17}\ cm^{-3}$	$1{,}8 \cdot 10^{17}\ cm$
60	$4{,}1 \cdot 10^{17}\ cm^{-3}$	$8{,}3 \cdot 10^{16}\ cm^{-3}$
65	$3{,}3 \cdot 10^{17}\ cm^{-3}$	–

Studies have shown that in n-Si samples with $N_0 \approx 8 \cdot 10^{17} cm^{-3}$ the AHP up to 40 kbar does not lead to noticeable change in the oxygen and carbon absorption peaks. At the same time, in n-Si<Ni> samples, it was found that after annealing at P≥30 kbar, the oxygen and carbon absorption peaks gradually decrease and completely disappear at a pressure of P=55 kbar (Table 1.). In contrast to the nickel-doped samples, the absorption spectra of the n-Si<Gd> samples turned out to be more resistant to external pressure.

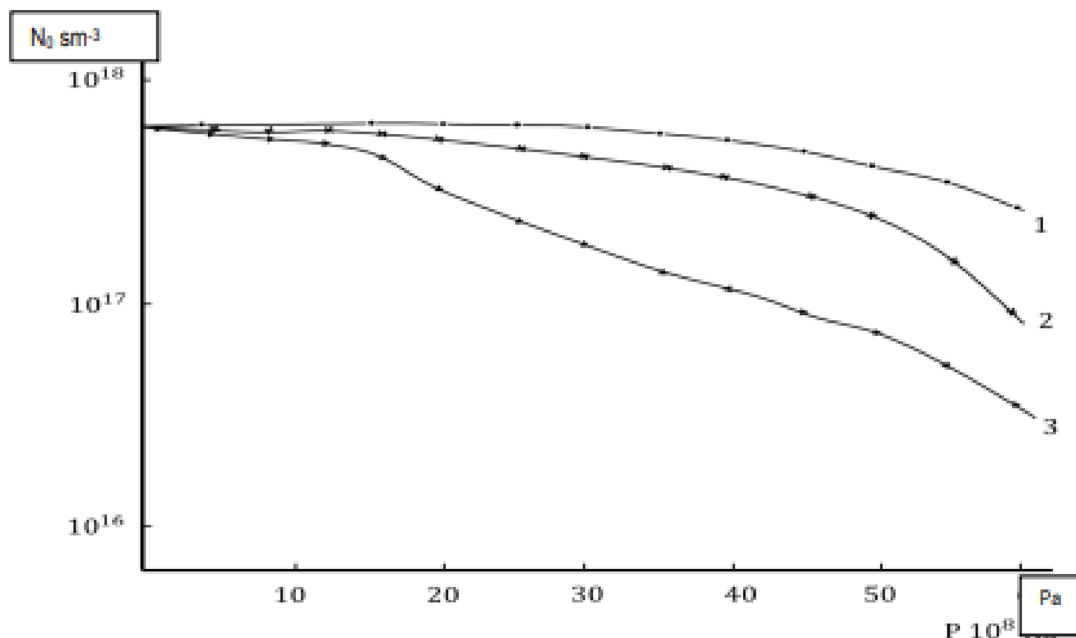

Fig 1. Dependence of the concentration of optically active oxygen atoms on the AHP for sampl of n-Si (curve 1), n-Si<Gd> (curve 2), n-Si<Ni> (curve 3)

As can be seen from Fig.1. with an increase in the value of the external pressure from 20 kbar, the peak at λ=9.1 μm gradually decreases to one order of magnitude and at P=65 kbar almost completely disappears. Comparing the curves of the dependence of the absorption intensity on P, one can see (Fig. 1) that in the n-Si<Gd> samples, the decrease in the oxygen and carbon peaks begins at a pressure value of P>10 kbar, while this value of P for n-Si<Ni> is equal to P≥30 kbar. We have carried out a qualitative assessment of the concentration of oxygen and carbon at various pressures using formulas (1) and (2), which showed that at P=40 kbar the concentration of optically active oxygen atoms in n-Si<Ni> samples decreases to one order of magnitude relative to control samples . The concentration of optically active carbon atoms in these samples in the pressure range P=1÷30 kbar practically does not change, and at P=35÷55 kbar a decrease in N_c by 45÷50% was found. Based on the results obtained, the dependence of changes in oxygen concentration on pressure was compiled (Fig. 1.).

In [6], the decay of Ni and Gd impurity precipitates under the action of external hydrostatic pressure, where it was shown that the defect formation energy of gadolinium precipitates is much lower ($E_d^{Gd} \approx 2$ eV), than the defect formation energy of nickel precipitates ($E_d^{\mathrm{Ni}} \approx 4$ eV). This means that the decomposition of gadolinium precipitates begins earlier than the decomposition of nickel precipitates under external pressure. It can be assumed that the increase in the concentration of oxygen atoms passing into the optically inactive state in n-Si<Ni> samples compared to n-Si is determined by its interaction with Ni and Gd atoms or their silicides formed after the decomposition of precipitates in the bulk of the crystal.

However, it should be noted that after annealing samples for 30 minutes at a temperature of 1000°C, the optically active concentration is restored by 80%. The rest (about 20%) of oxygen is possibly generated on the surface of the single crystal.

Simultaneous changes in resistivity and concentration of optically active oxygen and carbon atoms, in our opinion, are associated with the following processes:

a) at high value of the all-round hydrostatic pressure, the precipitates simultaneously disintegrate and new dislocation lines with various defects appear, which partially accelerates the disappearance of optically active oxygen atoms.

b) the formation of oxygen precipitation with the participation of carbon atoms.

Our experiments have shown that high hydrostatic pressure accelerates the precipitation of oxygen and carbon atoms. In this case, significant elastic stresses arise at the precipitate-matrix interface. With an increase in oxygen precipitation, the process of formation of complexes of the $C_m O_m$ type proceeds. Using the law of acting masses, E_a. According to experimental data [6] according to the formula:

$$[\mathrm{CO}] \approx \exp(\mathrm{E_a}/\mathrm{kT}) \quad (3)$$

$E_a \approx 1{,}1$ ev was estimated.

These studies are of great practical importance in the field of solid-state physics. With the help of AHP, it is possible to control the degradation of electrophysical parameters and features of the interaction of impurities with silicon.

One of the main requirements for semiconductor devices is the reliability of operation and the stability of their characteristics in relation to various external influences. In this regard, we have studied the effect of all-round hydrostatic compression on the relaxation characteristics of metal-semiconductor structures fabricated on the basis of crystalline silicon with different resistivity. To measure electrical resistance, careful temperature control is necessary. And to measure mobility, before determining changes in carrier concentration, you need to find a way to accurately correct for changes in mobility. Mobility corrections can be made either by extrapolating the pressure coefficient for mobility measured at higher temperatures (where the

electron concentration does not change under pressures, since all impurities are ionized [7]), or by calculating changes in pressure coefficients for parameters describing mobility (if they are known).

RESULTS AND DISCUSSION. The pressure coefficients for ionization, determined from changes in carrier concentration, are presented in Table-1. These coefficients are small, but they coincide with the values calculated theoretically and from the pressure coefficients for the effective mass and dielectric constant found in other studies [8].

Table 2

Ionization energy coefficients of impurities for silicon [9].

№	System	Ionization energy, eV	Pressure coefficient eV·cm²/kg
1	Antimony, donor	0,05	~ -5·10⁻⁸
2	Aluminum, acceptor	0,06	~ +1·10⁻⁸
3	Indium, acceptor	0,16	~ +5·10⁻⁸
4	Gold, acceptor¹	0,54	-1,2·10⁻⁶
5	Gold, acceptor²	0,62	-0,3·10⁻⁶
6	Gold, donor³	0,35	≤ |5·10⁻⁸|

The paper presents the results of an experimental study of the effect of pressure on the characteristics of Schottky diodes based on n-Si<Ni>. Crystalline silicon of the electronic type of conductivity (KEF-200, with crystallographic orientation <111>) was used as the starting material. The plates were subjected to heat treatment at a temperature of 1250 ^{0}C for 2 hours, followed by rapid cooling (>200^{0}C/min.). Schottky diodes were fabricated by vacuum sputtering of Au onto the surface of silicon wafers. The area of the metal contact was 7.1.10-2 cm^2.

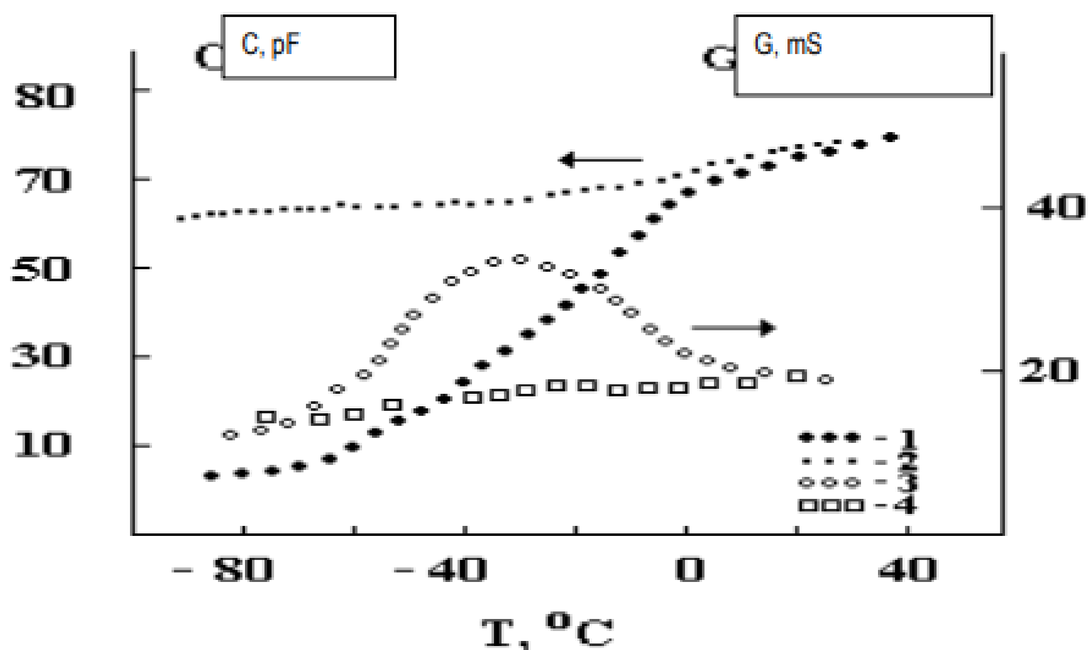

Fig 2. Temperature dependences of capacitance (1) and conductivity (2) of one of the investigated Schottky diodes before (1.2) and after (3.4) exposure to pressure [6].

Some of the manufactured diodes were subjected to all-round compression up to pressures of 70 kbar. Changes in the diode parameters were controlled by dark capacitance-voltage characteristics measured by the bridge compensation method at a frequency of 150 kHz. The temperature of the diodes during the measurement was stabilized with an accuracy of 0.5^{0}C. The measurements showed that the measured capacitance of the diodes weakly depends on the magnitude of the applied voltage, and when cooled to -80^{0}C, it decreases to the value of the geometric capacitance C_g.

Figure-2 shows the temperature dependences of capacitance (1) and conductivity (2), measured in a parallel equivalent circuit of one of the diodes under study at a reverse voltage of 4V. Thus, measurements under pressure, first of all, make it possible to separate that part of the temperature coefficient of the parameter, which is due only to the expansion of the lattice, from the additional contribution, which is the temperature effect proper [9]. Such a behavior of the capacitance and the presence of a maximum in the temperature dependence of the diode conductivity, in accordance with the theory developed in [6–12], indicates an overcompensation of the base region of the diodes under study. In this case, the relation $N_a > N_m$ between the concentrations of the acceptor deep center N_a and the donor shallow center Nm is satisfied.

Using the assumptions proposed by the authors of [6] and the equivalent circuit of a diode from an overcompensated semiconductor [12], we can obtain the following expression:

$$1{,}15\ \lg A = 2{,}3\ \lg\left(\frac{\omega \varepsilon d}{q\mu_n N_m}\right) + \frac{E_c - E_a}{kT} \tag{4}$$

$$A = \frac{C^{-1} - C_b^{-1}}{C_g^{-1} - C^{-1}}, \tag{5}$$

C - is the measured capacitance, C_b - is the barrier capacitance and C_g - is the geometric capacitance of the diode, E_c – is the energy corresponding to the bottom of the conduction band, E_a - is the activation energy of the compensating impurity, ω - is the cyclic frequency, ε - is the permittivity of the semiconductor, μ_n - is the mobility of the main charge carriers, d-dimensionless parameter.

Neglecting the temperature dependence of the parameter $(\omega\tau_0 d)$, which is equivalent to neglecting the temperature dependence of the mobility of the main charge carriers, using the tangent of the slope angle (1), we find the activation energy of the compensating deep center. In [6], the temperature dependences of the parameter - A, for two of the diodes under study are given, according to which the ionization energy of the compensating center is E_C - 0,27 $\pm$ 0,03 eV. The temperature dependence of the parameter A, for the two investigated diodes are shown in Figures -2, 3.

The solid straight line is a similar dependence according to [7]. In all diodes subjected to all-round compression to pressures of 1–4 kbar, with a step of 1 kbar, with an exposure of 10 min in each interval, the temperature dependence of the capacitance weakens, and the maximum on the temperature dependence of the conductivity gradually smoothes out. When pressure of 4 kbar is reached, the temperature dependence of the capacitance becomes comparable with the temperature dependence of the diffusion potential (see Fig. 2, dependences 3 and 4).

This behavior of the diode capacitance indicates the removal of compensation and the fulfillment of the condition $N_m > N_a$. Control measurements using the method of isothermal relaxation of the capacitance [8, 9], the applicability of which is possible under the condition Nm>Na, showed that in diodes subjected to a pressure of 4 kbar, there is a recharging of two

deep centers with ionization energies E_C - 0,27 ± 0,03 eV and E_C -0,54 ± 0,03 eV. The temperature dependences of the relaxation time constants for two Schottky diodes subjected to a pressure of 4 kbar are shown in Figure 3. in [7, 8] and in our case, the charge exchange of identical [9] centers were observed.

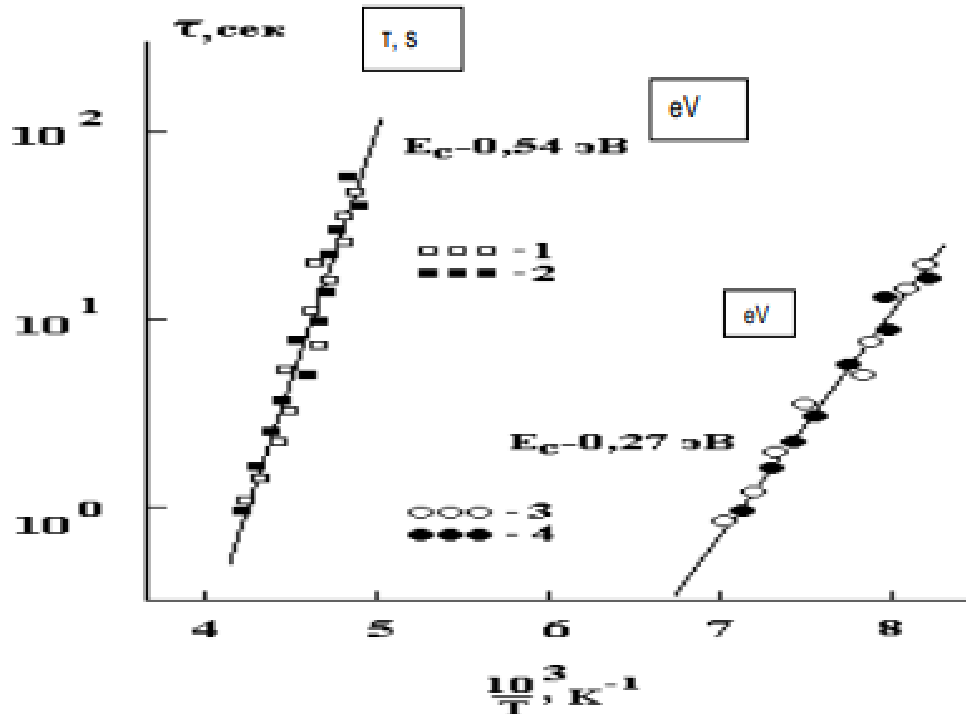

Fig 3. Temperature dependences of the relaxation time constants for two (1.3 and 2.4) Schottky diodes subjected to pressure of 4 kbar. Solid line - **τ(T)** dependences according to [7]

Considering that the diodes described in [7] were created using boron diffusion, and in our case, silicon wafers were subjected to only similar thermal effects, we can draw the following conclusion. The centers observed in the diodes studied by us and described in [7, 8] are of the same nature. The impact of pressure, without changing the structure of the center, only leads to a decrease in its concentration.

Note that the capture cross section of the majority charge carriers does not change under pressure, which follows from the invariance of the dependences **τ(T)** in the diodes, both in the presence of compensation and when it is removed. Under pressure, precipitation of oxygen atoms occurs, that is, free interstitial oxygen passes into the second phase with the formation of SiO_2 particles [12] (Fig.-5). Which, on an n-type semiconductor, when exposed to strong electric fields, many electrons quickly gain enough energy to excite 0.2 eV or more above the edge of the conduction band. In this case, it is essential to know the position of all higher lows in order to fully understand the effect.

The application of 12 kbar pressure changes the position of the oxygen atoms to settle on the SiO_2 agglomerations, as result of which they probably lose electrical activity. It can be seen from the figure that at 12 kbar the saturation of the J-V characteristics is less, but at lower pressure there is saturation. An analysis of the obtained results shows (Fig. 4) that when pressure up to 12 kbar is applied to the diode structures studied by us based on n-Si<Ni> containing impurity centers in the bulk of the semiconductor, monotonous increase in resistivity occurs in them, in contrast to the data work [13]. Hence, we can conclude that the centers we observe are structural defects of the semiconductor due to its rapid cooling.

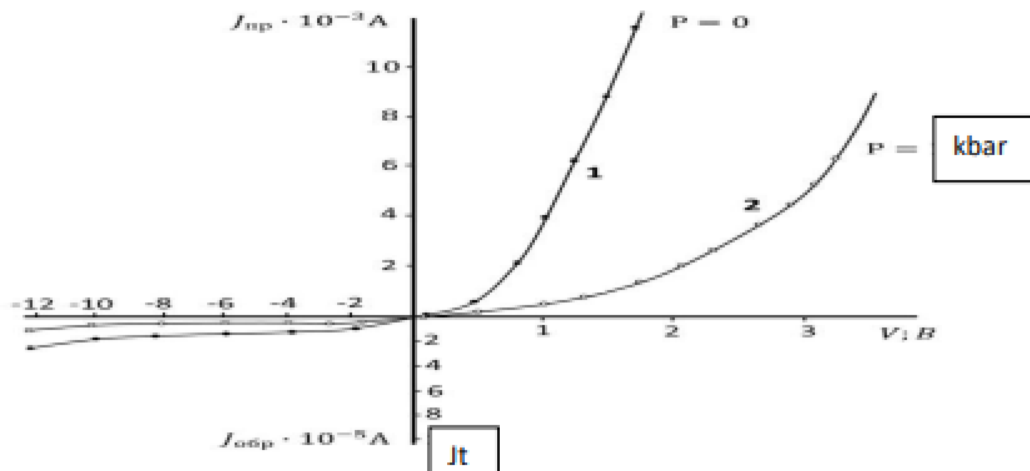

Fig 4. CVC of Schottky diodes before and after exposure to confining pressure

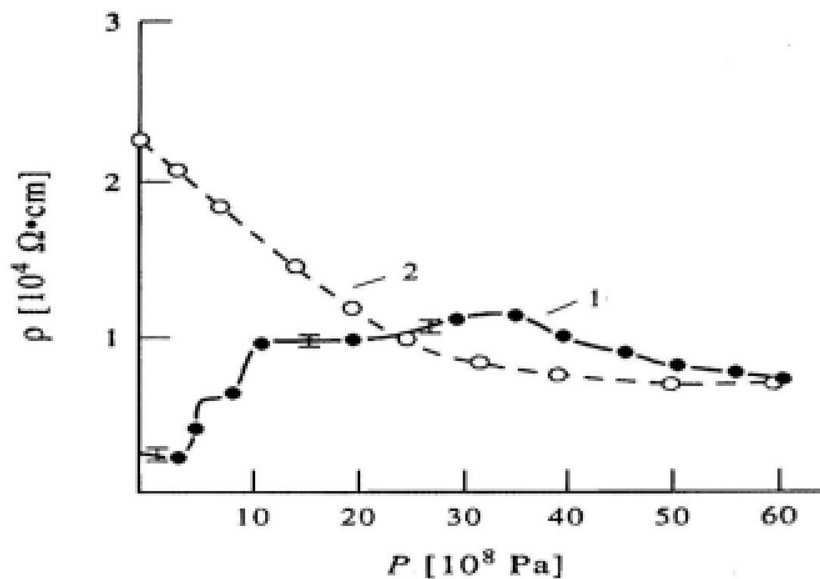

Fig 5. Strain resistance of n-Si<Ni> specimens under all-round hydrostatic compression [11]. (1 - as P increases, 2 - as P decreases).

However, as the pressure increases in the range from 12 kbar to 38 kbar, the change in the resistivity of the n-Si<Ni> samples under the influence of pressure (Fig. 5, dependences 1 and 2) has nonmonotonic character with the formation of a maximum at P≥35 kbar. The formation of this maximum is explained by the fact that, despite the high solubility of Ni atoms in silicon (7.10^{-17} cm^{-3}) Ni atoms (5.10^{-14} cm^{-3}) is electrically active. These atoms create two acceptor levels in the Si band gap (E_v+0.2 eV and E_c–0.4 eV). And with further increase in pressure (P>38 kbar), the resistivity will noticeably decrease.

CONCLUSION. It can be assumed that the experimentally observed nonmonotonicity in the dependences ρ=f(P) is the result of two counter processes: the first is a decrease in the band gap and a change in the ionization energy of deep Ni levels, which leads to an increase in conductivity, and the second is the decay of impurity precipitates with an increase in the concentration of electrically active centers, which, under the influence of pressure, are displaced

from the bulk of the semiconductor and changing the surface charge distribution spectrum at the metal-semiconductor interface. Mechanical stresses that stimulate the gettering of thermal defects from the bulk of the semiconductor or impurities localized in the metal-semiconductor transition layer and interacting with surface states can be responsible for changing the properties of the interface under pressure.

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