

INCREASING OF RADIATION HARDNESS OF SILICON FOR DETECTORS BY PRELIMINARY IRRADIATION.

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1. Introduction

The necessary long term operation of semiconductor devices in nuclear radiation fields is the reason to pay attention to the problem of increasing of their radiation hardness. For example, in a series of experiments at CERN with beams of charged particles the detecting system consists of many different detectors. It is clear, that the increase of a their long-term stability will essentially improve efficiency of such experiments. The situation with magnetic sensors using, based on semiconductor materials A_3B_5 , at charged particle accelerators is similar. That's why investigation of radiation hardness of semiconductor materials and devices on their basis was always in the focus of researchers attention.

2. Methods of increasing semiconductor materials radiation hardness

As it is known, during irradiation of semiconductors, the wide spectrum of defects are formed: point defects of vacancy and interstitial types, their complexes with impurities and clusters of defects. And while point

defects are rather mobile under experimental conditions, clusters are less mobile. There is a space charge around clusters, it may essentially effect on the characteristics of materials and devices based on its.

The larger part of electrically active defects, induced under irradiation are the impurity-defect complexes. So during γ - irradiation silicon, vacancies and interstitial atoms interacting with the impurity atoms and with each other, participate in formation of A - centers (V-O); E- centres (P-V); divacancies (V-V) and di-interstitials (I-I). These defects are efficient scattering and recombination centers for charge carriers in semiconductors.

In addition to point defects, high-energy nuclear particles and fast neutrons of reactor create defect clusters surrounded with a space charge blocking a current of charge carriers. Radiation hardness of silicon semiconductor detectors is first of all determined by a rate of introduction of point defects and aggregation of defects into clusters.

In [1-3] the influence of various impurities, such as B, O, isovalent impurities Sn, C, Ge etc. on radiation hardness of silicon were studied.

Below we shall discuss general methods of silicon hardness radiation increasing. Briefly it is possible to extend the influence of various impurities on radiation hardness and reasons increasing it. So, the introduction of electrically inactive impurity of oxygen promotes the derivation of a vacancy stream from a doping phosphorus impurity. It is also known, that the probability of phosphorus capture by vacancy is approximately in two orders higher, than for oxygen. Thus, in spite of the greater radius of capture of vacancies by phosphorus atom, high concentration of oxygen will suppress the formation of E-centres. As detectors operate at room temperature and, hence, the Fermi level is close to the middle of a forbidden gap in high-resistivity Si, though the level of A- centre ($E_C - 0,164$ eV) will not influence on removal of carriers from a conduction band. Compared to phosphorous, boron has larger radius of vacancy capture, which rises radiation hardness of silicon, highly doped with boron. [1] Interstitial atoms of silicon displace boron in an interstitial position, where boron is efficiently captured by divacancies or recombines with vacancies.

The temperature of the sample during irradiation also efficiently influences on formation of radiation defects. So the irradiation at temperature lower than 120 K reduces of removal rate of the carriers, if measured at room

temperature, in tens of times [4]. In [5] it is shown, that intensity of near - edge absorption and rate of postradiation annealing of disordered regions are much higher in the crystals of silicon irradiated by neutrons at 100K. It can be connected with high rate of introduction of non-oriented divacancies, that are intensively annealed at the temperature above 140K. The low-temperature irradiation also suppresses participation of oxygen in formation of electrically active defects, containing oxygen. That is one method more of increasing the semiconductor materials radiation hardness.

In the course of silicon doping with isovalent impurities Sn and Ge, the mechanical fields of strains caused by their large covalent radius are formed around the places of localization of these impurities. These fields of deformation strains may serve as sinks for vacancies and interstitial atoms, which causes suppression of vacancy complexes and divacancies formation. At high concentrations of these impurities ($> 10^{18} \text{ cm}^{-3}$), in silicon a carriers removal rate measured at room temperature is suppressed. So, the silicon doping with Ge suppresses the introduction rate of E- centres and divacancies by two orders, and Sn three times more effectively interacts with vacancies than with oxygen. However, such large concentration of impurities (Sn, Ge) may result in material homogeneity deterioration and decreasing of charge carrier lifetime, which determines quality of Si detectors.

Dislocations, free from the impurity atmospheres, are also capable to interact efficiently with radiation defects [6,7]. At density 10^6 ?m^{-2} the dislocations in Si create deformation fields, under the influence of which vacancies and interstitial atoms generated by irradiation can migrate. Thus, radiation defects can lead to reorganization of a dislocation structure. But their small quantity and a high-temperature method of their generation interfere their use as efficient method for increasing detector material radiation hardness.

Growing microdefects (A, B, D) also efficiently interact with radiation defects. It is known, that when silicon single crystals are grow with small velocity ($\leq 3 \text{ mm/min}$), excess atoms of silicon in the range of a crystallization are observed, so A - microdefects are formed. They are dislocation loops of interstitial type, deposited in a planes (110) and (111) and strongly directed along the direction {110}. They are easily determined by the selective etching and usually have the size $(1-3) 10^{-4} \text{ cm}$ at concentration $(10^4-10^6) \text{ cm}^{-3}$. The

elastic deformation fields around A- microdefects may influence on radiation defects at distances no more than five diameters of a loop, as the electrostatic energy decreases as cube of distance.

At mean velocities of growing (~4 mm/min) B-microdefects of interstitial type are formed, representing flat aggregations in the shape of quadrate or rhomb deposited in planes (100) with sizes (200-500) Å and concentration (10^6 - 10^8) cm⁻³.

In the crystals grown up with high velocity (> 5 mm/min), when in the crystallization range the deficit of silicon atoms is created, D-microdefects of vacancy type, with sizes (40-60) Å and concentration of 10^{12} cm⁻³ are observed. D defects are centres of capture for Li⁺ atoms at their drift during the production of Si (Li) detectors. Routinely after annealing of neutron-doped silicon, used for creation of detectors, the formation of all types of microdefects sharply increases. Therefore, the method of gettering, offered in [8], on using a phosphoric glass (POCl₃) appears to be useful for NTD Si. Getter covering a surface of silicon, generates interstitial atoms in the crystal bulk and thus, D-microdefects are destroyed. Increasing of radiation hardness of the neutron-doped silicon is also found [9-11].

3. Experiment and discussion of results

Given below are experimental results concerning use the method of a preliminary irradiation and neutron transmutation doping of silicon for increasing of radiation hardness of silicon as well as indium antimonide.

3.1. Preliminary neutrons irradiation influence on radiation hardness of silicon in accordance with the results of optical investigations.

Infrared absorption study in the range of wavelengths behind intrinsic band (near edge absorption) is an effective research technique for researching radiation effects in Si. According to [12] near edge absorption after the irradiation by large doses of neutrons ($\sim 10^{19}$ n/cm²) is caused by aggregations of vacancy and interstitial types. These becomes apparent after influence of isochronous annealing in the interval of 400-500° ?

temperatures. Decay of interstitial aggregations is accompanied by formation of complexes of interstitial atoms with oxygen. The occurrence of the whole spectrum of narrow bands of an oscillatory nature testifies it on a background of two-phonon absorption. There is a full annealing of these defects at 700⁰ temperature.

At the repeated irradiation of "oxygen" samples the annealing gives too much smaller formation of oxygen complexes. It is probably due to the fact that interstitial atoms instead of formation of complexes with oxygen prove to be more effective sinks, than the dissolved atoms of oxygen. That's why, the silicon with the great content of oxygen at the repeated irradiation by neutrons and annealing behaves as low oxygen material [13].

Above-mentioned results show the influence of the repeated irradiation mainly on the processes of radiation defects annealing.

Certainly, we are more interested in the influence of the preliminary irradiation on silicon radiation hardness.

We have carried out the test of the influence of various radiation processings with subsequent annealing on radiation hardness of FZ Si. As preliminary radiation processings by fast neutrons ($\Phi = 10^{16}$ n/cm²), by thermal neutrons, in combination with fast and thermal neutrons, and also thermal neutrons with γ quanta were used. After radiation processing all samples, including initial, were annealed during two hours at 800⁰ temperature. Then samples were irradiated with fast neutrons by fluence 10^{16} n/cm² at research reactor WWR-M.

After irradiation the investigations were carried out by the method of infrared spectroscopy using spectrophotometers UR-20 and SVU-23.

The most sensitive to such irradiation is FZ Si in the spectral range near the edge of intrinsic absorption of Si. The so-called near-edge absorption, monotonously decreases with wavelength. The selection absorption band with a maximum at 1,8 μ m that related with divacancies is imposed on it. After irradiation with the fluence $\sim 10^{16}$ n/cm² the near-edge absorption in Si which is caused by disordered areas that are preferentially of vacancy type [12]. The investigations of absorption spectrums in spectral range 2-1,1 μ m on a large set of samples subjected to various combinations of preliminary radiation processings were carried out.

The typical transmission spectrum in the spectral range 2-1,1 μ m is shown in Fig.1 for sample annealing at 800⁰ C during 2 hours. In Fig.2 the transmission spectrum are shown for sample annealed at 800⁰C 2 hours and

irradiated by fast neutrons $\Phi = 10^{16} \text{ n/cm}^2$ (curve *a*) and for sample preliminary irradiated by fast neutrons ($\Phi = 10^{16} \text{ n/cm}^2$), then annealed and once more irradiated by the same dose of fast neutrons (curve *b*). From comparing curves *a* and *b* one can see the decreasing additional absorption of the sample which was preliminary irradiated.

The results of these investigations are shown at the following two curves of figure 3, where differential transmission spectrum are given for clarity.

The curve *a* on fig. 3 represent the differential transmission spectrum of reference Si sample not subjected to preliminary radiation treatment in comparison to a sample, preliminary irradiated with fast neutrons. Both samples were annealed and then were irradiated with neutron fluence $\Phi = 10^{16} \text{ n/cm}^2$.

The samples were cut off from one ingot. They had the identical thickness and identical surface treatment. It is known, that preliminary 800°C annealing provides full annealing of the radiation defects, fixed by infrared absorption, thus the apparent distinction in intensity of their transmission spectrums should be caused only due to preliminary radiation treatment by fast neutrons. As it follows from curve *a*, the intensity of absorption in reference sample is higher.

Figure 3 curve *b* represent the differential transmission spectrum of two samples irradiated with fast neutrons $\Phi = 10^{16} \text{ n/cm}^2$, and preliminary subjected to various radiation treatments. As seen from this curve *b*, the intensity of absorption in these samples does not depend on the method of preliminary radiation treatment used in the given experiment (an irradiation with fast neutrons, both thermal and fast neutrons, as well as gamma-quanta and thermal neutrons).

Thus, the given results evidently show, that preliminary radiation treatment yields in increasing of radiation hardness due to formation sinks for radiation defects.

3.2 The influence of preliminary irradiation with charged particles on radiation hardness of Indium Antimonide

Let us consider the results of investigation of efficiency of a defect formation in preliminarily irradiated

semiconductors on the example of indium antimonide, irradiated by 47 MeV protons.

As a material was well studied and frequently used as a model material indium antimonide: sample 1-not doped n-InSb with initial concentration of charge carriers $n_{0K} = 1.6 \cdot 10^{14} \text{ cm}^{-3}$; sample 2-n-InSb <Sn> (tin) with initial concentration of charge carriers $n_{0K} = 9 \cdot 10^{15} \text{ cm}^{-3}$; sample 3 - p-InSb <Cd> with initial concentration of charge carriers $n_{0K} = 7 \cdot 10^{15} \text{ cm}^{-3}$. It was decided to test a variant of introduction of sinks. Samples were irradiated by 47 MeV protons on isochronous cyclotron U-240 of KINR (Kiev Institute for Nuclear Research of the National Academy of Sciences of Ukraine) with beam intensity $(1-6) \cdot 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ up to a certain fluence (a preliminary irradiation), then irradiated samples were annealed at room temperature up to some intermediate state and then the irradiation was recommenced (for sample 1 - at room temperature; for samples 2 and 3 - at $T=120^\circ\text{C}$) with the purpose to compare efficiency of defect formation in samples at the intermediate state with defect formation in irradiated initial samples with the same characteristics, as they are at the samples in the intermediate state (preliminary irradiated and annealed). From Fig. 4 one can see, that after recommencing of irradiation the rate of radiation defects introduction in samples has sharply decreased. In our opinion it was possible due to the influence of sinks for newly induced radiation defects as well as to radiation defects, induced by preliminary irradiation and annealing, which play the role of sinks. Analyzing the received result, it is possible to draw a conclusion, that a combination of preliminary irradiation with annealing, should establish the mode that may probably become a basis for radiation-thermal technology of increasing radiation hardness of semiconductor materials.

3.3 Radiation hardness of neutron transmutation doping silicon.

As a matter of fact, the neutron transmutation doping of silicon, is one of variants of the raise of radiation hardness of silicon by the help of preliminary irradiation.

The neutron transmutation doping of silicon (NTD) was carried out in a thermal column of a reactor WWR-M. Initial samples p-Si had a specific resistivity $\rho \sim 2-10 \text{ k}\Omega\cdot\text{cm}$. After irradiation by various doses of thermal neutrons and annealing at 850°C within 2 hours were obtained samples p-Si with a specific resistivity (12-

40) kOhm-cm and the samples n-Si with concentration of carriers $n = 1,4 \cdot 10^{12} \text{ cm}^{-3}$. Investigations of radiation hardness NTD silicon n- and p-type were carried out at the irradiation by γ -quanta ^{60}Co and by fast neutrons of the reactor with efficient energy ($E_n \sim 1 \text{ MeV}$). In NTD silicon of n-type conductivity the carrier removal rate after neutron irradiation is much less, than in reference samples of silicon. In tables 1 and 2 carrier removal rate, measured at room temperature for some radiation doses is shown. From tables 1 and 2 it is visible, that NTD silicon n - type has more higher radiation hardness to γ -radiation, than to the neutron irradiation. So, at γ -irradiation the carrier removal rate in NTD silicon more than one order less, than in a reference specimen, but at a neutron irradiation carriers removal rate is approximately two of times less. In high-resistivity NTD p-Si ($\rho \sim 10\text{-}40 \text{ kOhm-cm}$) it is not revealed apparent difference of radiation hardness from reference specimens p-Si. Apparently, it is connected with a small radiation dose needed for obtaining high-resistivity p-Si and, as a consequence, with small concentration of sinks created by irradiation.

When doping by neutrons, an irradiation of crystals by thermal neutrons, by fast neutrons and by γ -quanta takes place too. After neutron transmutation doping silicon and its subsequent annealing at 850°C , defects of vacancy type combine and form electrically inactive vacancy complexes, and defects of an interstitial type under annealing are transmuted into complexes of an interstitial type (microdefects, such as A, B and D).

Because distortions of crystal lattice around such aggregations there are elastic stress fields of squeezing. Under action of these fields vacancies generated by radiation and interstitial atoms migrate to aggregations where they may annihilate or form complexes with each other (for example - divacancies). For this reason concentration of initial vacancies, capable to participate in formation of complexes with the impurity atoms in the crystal volume, it appears much less, than in an FZ material. It yields in smaller efficiency of V -centres formation in NTD silicon.

Impurity-defect aggregations may also consist from electric active complexes $\text{C}_i - \text{C}_s$. Except for them aggregations is entered with own interstitials, interstitial carbon C_i or their associations, which in our experiment are electrically neutral. By estimates mean radius of area is about $\sim 1,3 \cdot 10^{-4} \text{ cm}$.

Naturally, that radiation hardness of NTD silicon will depend also both on concentration of sinks and that of phosphorus injected by transmutation. And for the best effect, it is necessary to have their optimum relation in

each concrete case. Thus, received by us NTD silicon n-type for manufacturing detectors of nuclear radiations is more radiation hard to γ - radiations Co^{60} and fast neutrons. It is caused by activity of high concentration of sinks having different nature and generated during a nuclear doping and after annealing.

3.4. Influence of oxygen on the formation of radiation defects in Si.

The temperature dependencies of carrier concentration in n-Si FZ and OFZ/G, at different doses 24 GeV protons is shown on fig.5 and fig.6.

In FZ Si n-type small concentration of oxygen at low doses of irradiation the level $(E_c - 0.47)\text{eV}$ is observed which corresponds to E-center (VP) (Fig.5). At higher doses the level $(E_c - 0.49)\text{eV}$ is observed which we identified with three-vacancies (V_3). The introduction rate of this level is $v = 0,16 \text{ cm}^{-1}$. Probably such changing of level spectra connect with predominating process formation many vacancy complexes at high doses, as concentration of vacancies is much more then phosphorus concentration and the most phosphorus atoms were used to formation of E-centers at low doses.

It oxygenated Si (OFZ/G) at low doses the level $(E_c - 0.43)\text{eV}$ is observed, which identified as divacancy (V_2) (Fig.6). At increasing the dose, level $(E_c - 0.455)\text{eV}$ is observed. But E-center do not observe. This connect with drawing away the flow of vacancies from phosphor to oxygen as its concentration is more high than phosphorus.

In the case the concentration of lower level is approximately equal to carrier concentration in conductivity band, efficient slope will be observed with E_{eff} equaled to the half-sum of energies of two centers:

$$E_{\text{eff}} = (E_{0,43} + E_{0,49}) / 2 \sim E_{0,455}$$

One can see that in oxygenated Si the formation of E-centers $(E_c - 0.47) \text{ eV}$ is depressed and the radiation hardness of such material is increased.

From the other hand, in oxygenated crystals centers of precipitation are formed after preliminary irradiation and annealing. These centers may act as the sinks for radiation defects.

Therefore, the preliminary irradiation and annealing of oxygenated silicon material leads to the increasing of the radiation hardness as well as also due to the introduction of the precipitates which are the sinks for radiation

defects. Now the influence radiation on formation of the precipitates and on the kinetic of precipitation of oxygen at different temperatures are performing in our department.

4. Conclusions

The preliminary irradiation of silicon by fast neutrons and the annealing leads to the formation the sinks for primary radiation defects. These sinks are the complexes of radiation defects with neutral impurities, such as C,O, that always contains in silicon crystals. Such method of preliminary irradiation leads to increasing the radiation hardness of silicon. For achievement the best effect it is necessary to induce the optimum concentration of sinks in each concrete case.

The radiation hardness of the neutron transmutation doping silicon increases in ten times to gamma-irradiation and in two times to neutron irradiation due to sinks, which has been created at neutron doping and at technological annealing of silicon. The neutron transmutation doping is one of the ways of the increasing of radiation hardness of Si by preliminary irradiation.

This method can be applicable for increasing of radiation hardness of many semiconductors, but it is the most advanced for silicon that has been used in development of detectors and other semiconductor sensors working in fields of nuclear radiation.

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Table 1
g-irradiation

Fluence $^{60}\text{Co}, \text{cm}^{-2}$	NTD		Reference sample	
	concentration N, cm^{-3}	removal rate $\text{DN}/F, \text{cm}^{-1}$	concentration N, cm^{-3}	removal rate $\text{DN}/F, \text{cm}^{-1}$
0	$1,4 \times 10^{12}$	–	$3,4 \times 10^{12}$	–
$1,6 \times 10^{14}$	$1,4 \times 10^{12}$	0	$2,67 \times 10^{12}$	$4,5 \times 10^{-3}$
$4,8 \times 10^{14}$	$1,33 \times 10^{12}$	$1,5 \times 10^{-4}$	$2,16 \times 10^{12}$	$2,6 \times 10^{-3}$
$1,6 \times 10^{15}$	$9,8 \times 10^{11}$	$2,6 \times 10^{-4}$	$1,7 \times 10^{12}$	$1,1 \times 10^{-3}$

Table 2
Irradiation by fast neutrons

Fluence fast neutrons, $n \times \text{cm}^{-2}$	NTD		Reference sample	
	concentration N, cm^{-3}	removal rate $\text{DN}/F, \text{cm}^{-1}$	concentration N, cm^{-3}	removal rate $\text{Dn}/F, \text{cm}^{-1}$
0	$1,4 \times 10^{12}$	–	$1,0 \times 10^{13}$	–
$4,6 \times 10^{11}$	$1,1 \times 10^{12}$	0,66	$9,2 \times 10^{12}$	1,74
$9,6 \times 10^{11}$	$6,4 \times 10^{11}$	0,79	$8,5 \times 10^{12}$	1,56

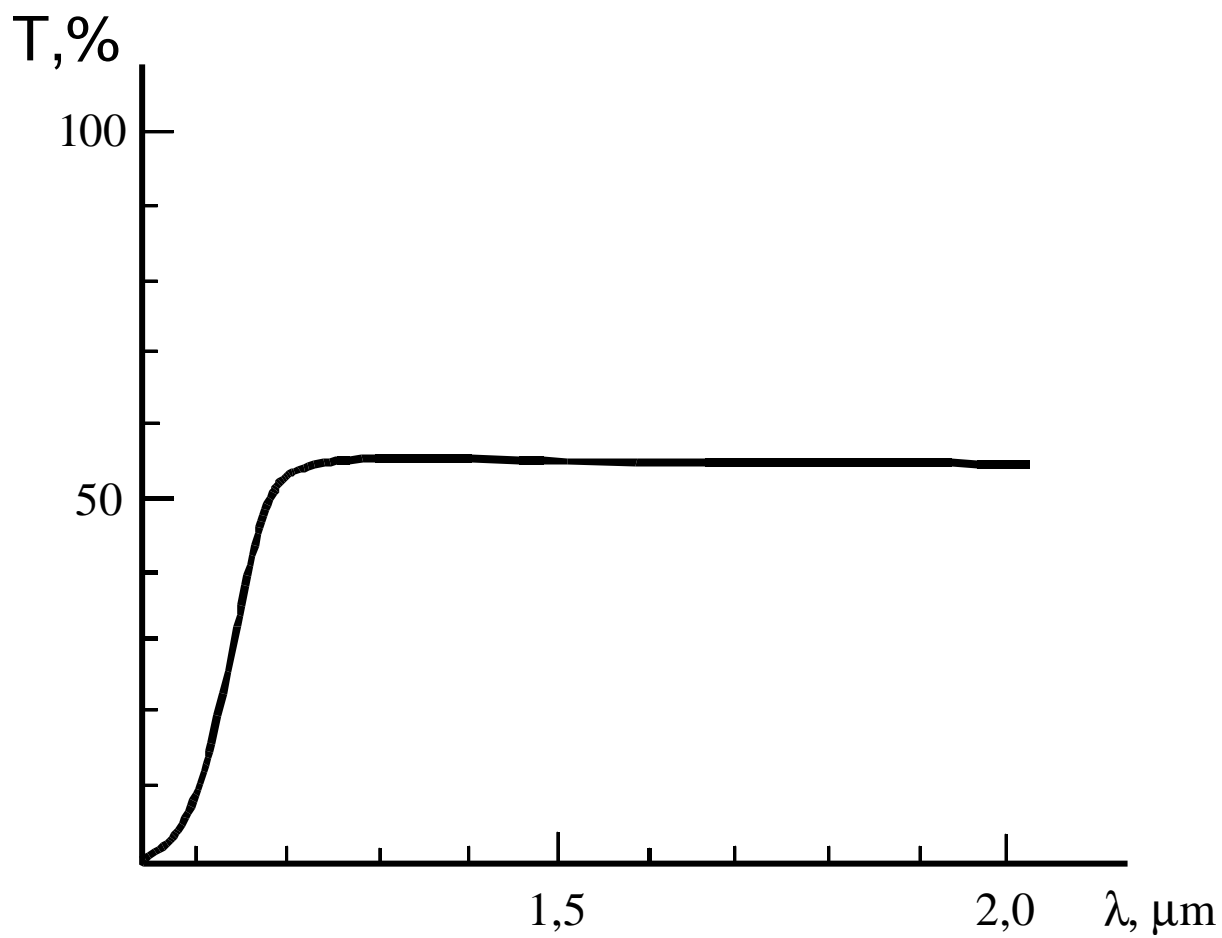


Fig 1. Dependence of transmission on wavelength for FZ Si sample annealing 2 hours at 800°?

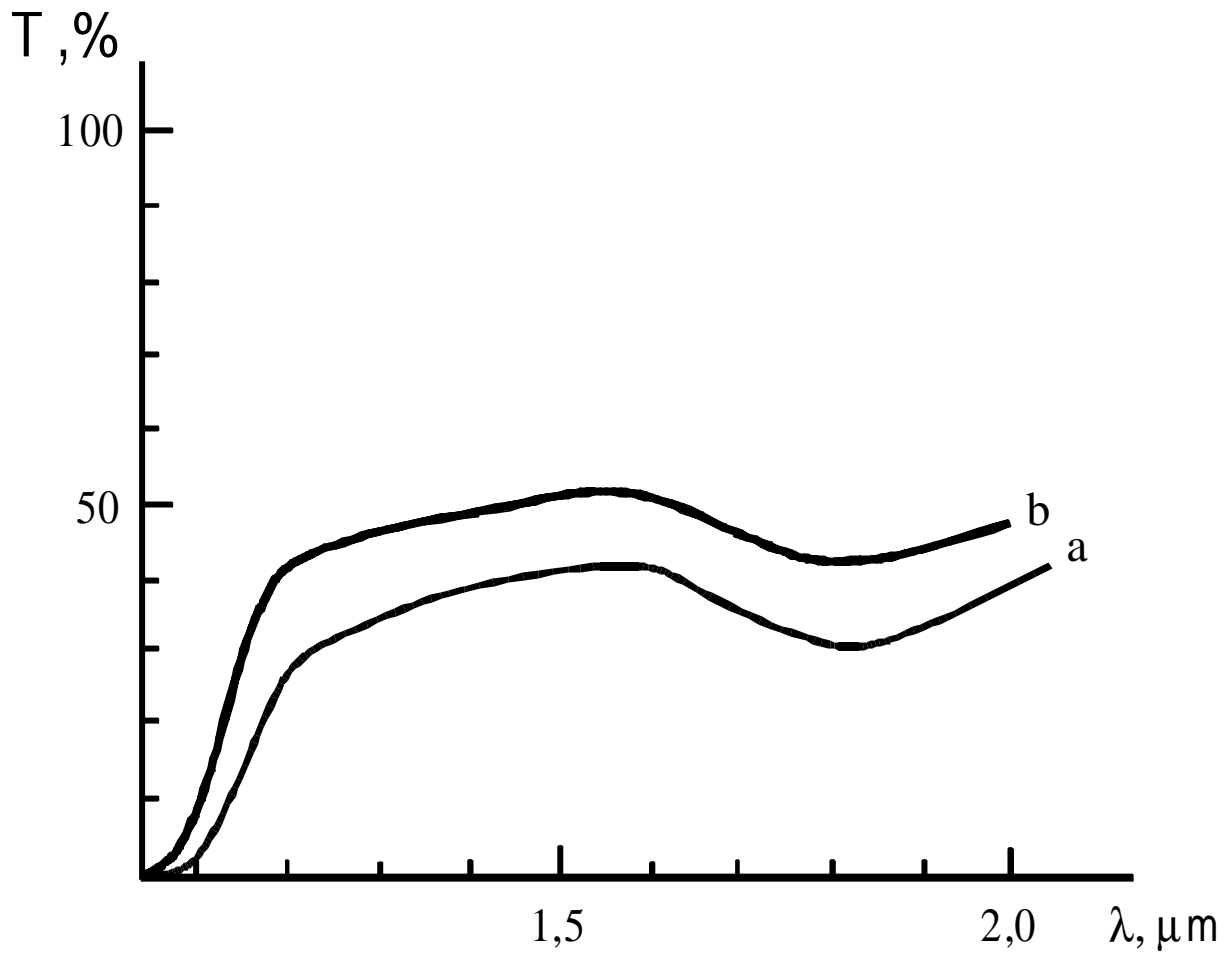


Fig 2. Transmission spectrum of Si sample:

a — annealed at 800°C and irradiated by fast neutron ($\Phi = 10^{16} \text{ n/cm}^2$)

b — preliminary irradiated by fast neutron ($\Phi = 10^{16} \text{ n/cm}^2$) then annealed and irradiated by fast neutrons ($\Phi = 10^{16} \text{ n/cm}^2$)

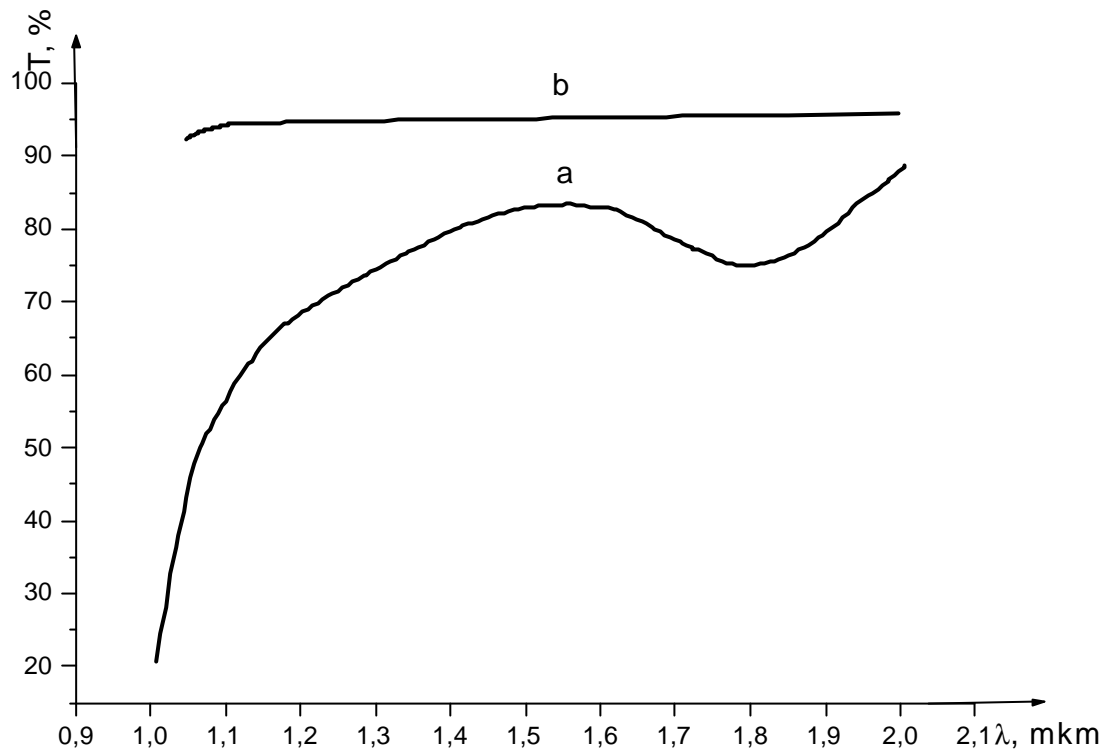


Fig 3. Differential transmission spectrum dependence for Si samples after 800⁰? annealing and irradiation with neutron fluence $\Phi = 10^{16} \text{ n/cm}^2$ on wavelength $T=f(\lambda)$

- a** — for the reference sample in comparison with a sample preliminarily irradiated with fast neutrons. Thickness of samples is $d=0.434 \text{ cm}$
- b** — for a sample subjected to preliminary radiation treatment (by fast and thermal neutrons) in comparison with a sample preliminarily irradiated by gamma-quanta and thermal neutrons.

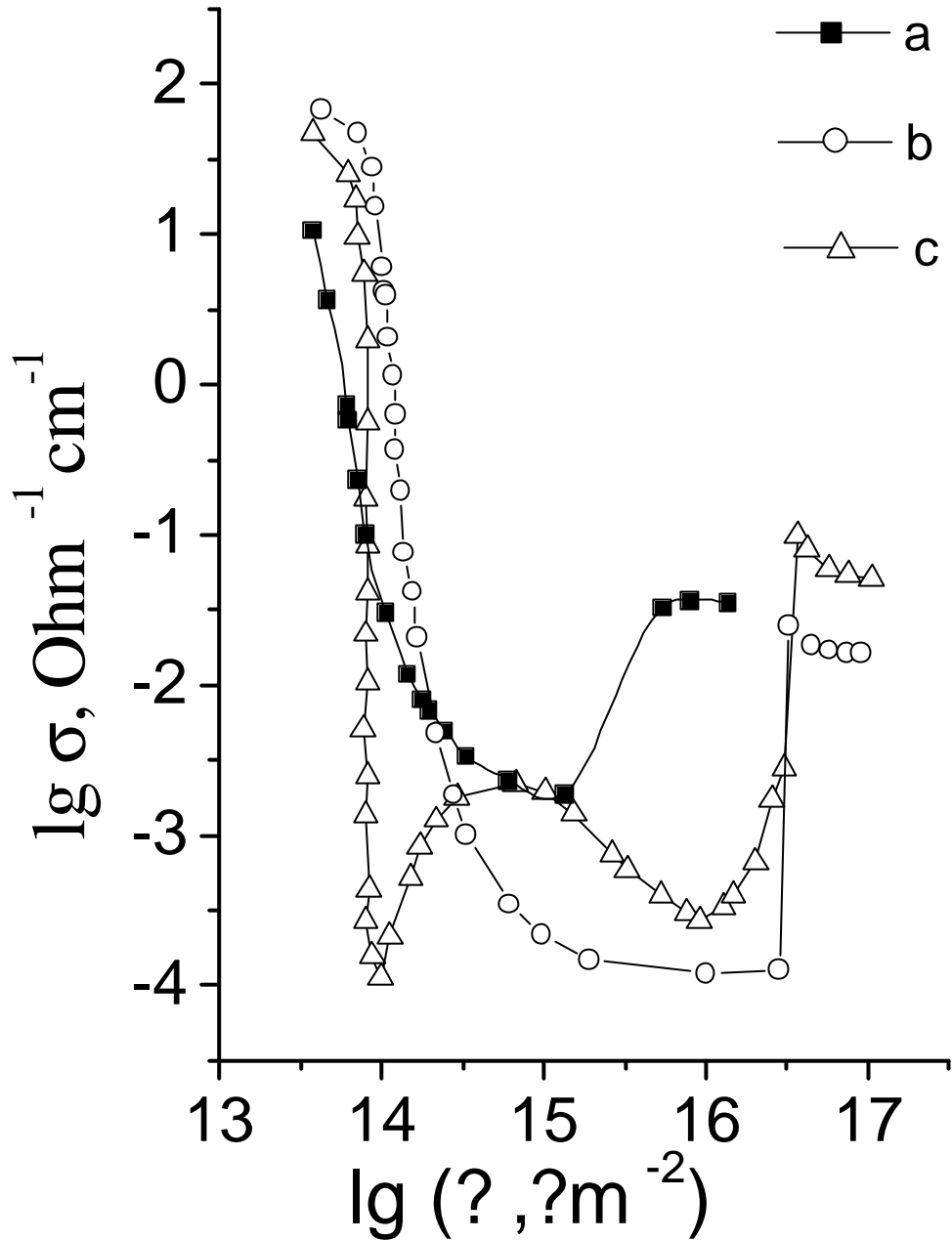


Fig 4. Dose dependences of conductivity σ_{300K} in irradiated by 47MeV protons Indium Antimonide:

a- n-InSb; irradiation at 100K and 300K;

b- n-InSb <Sn>; temperature of an irradiation 120K

c- p-InSb <Cd>; temperature of irradiation 120K

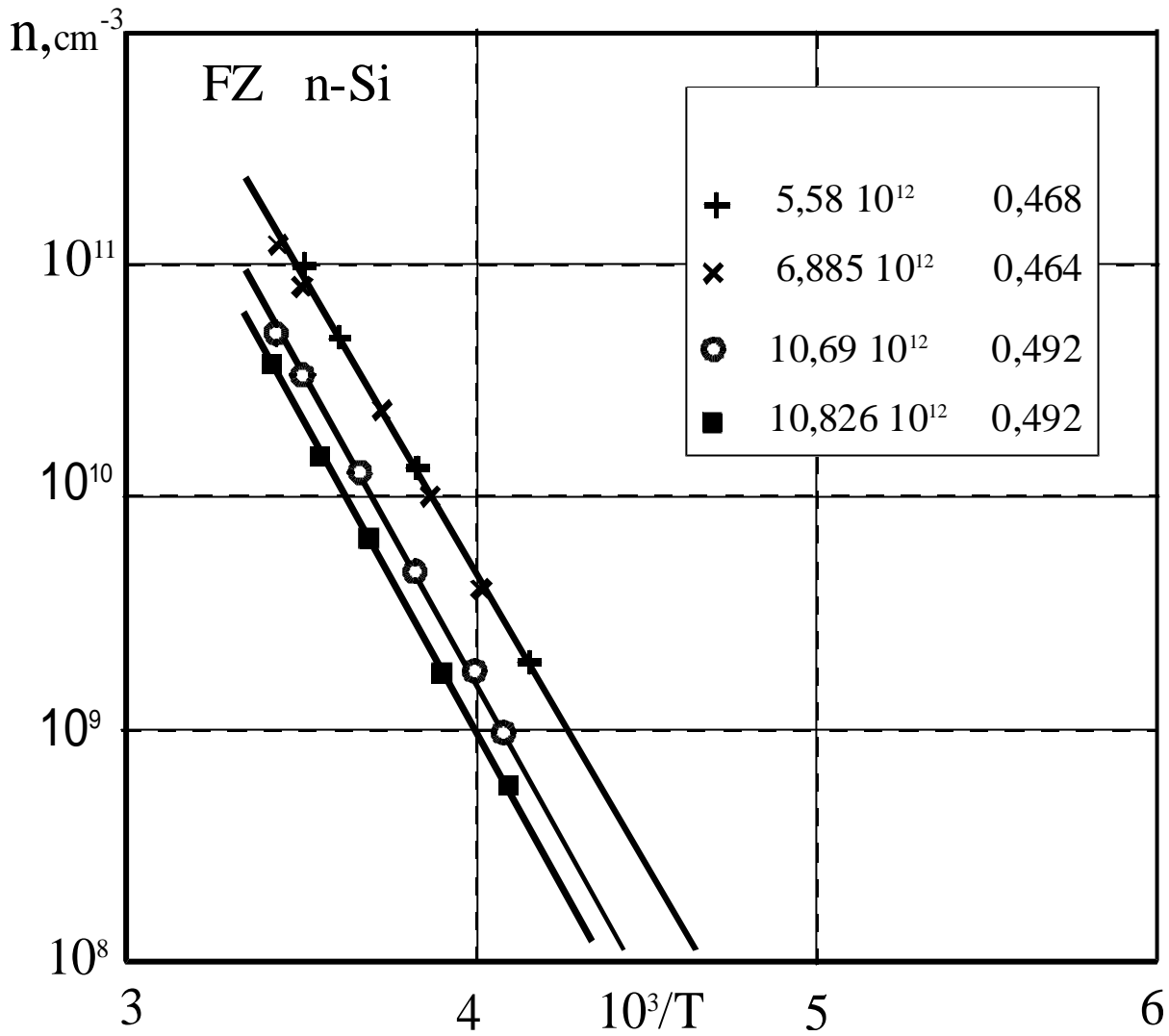
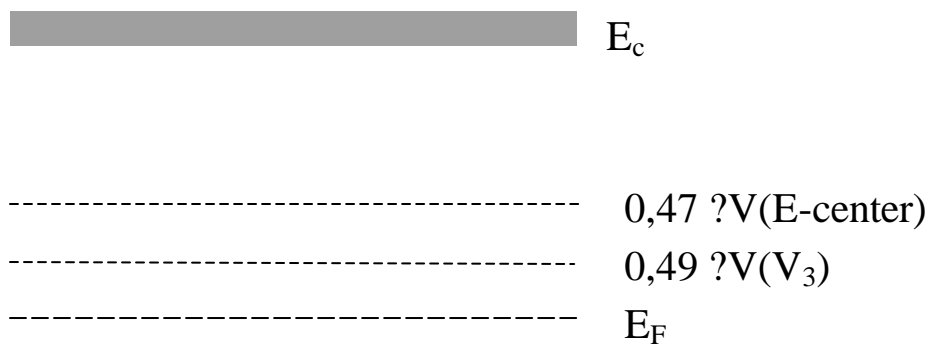


Fig 5. Temperature dependence of carrier concentration of FZ n-Si at different doses 24 GeV protons



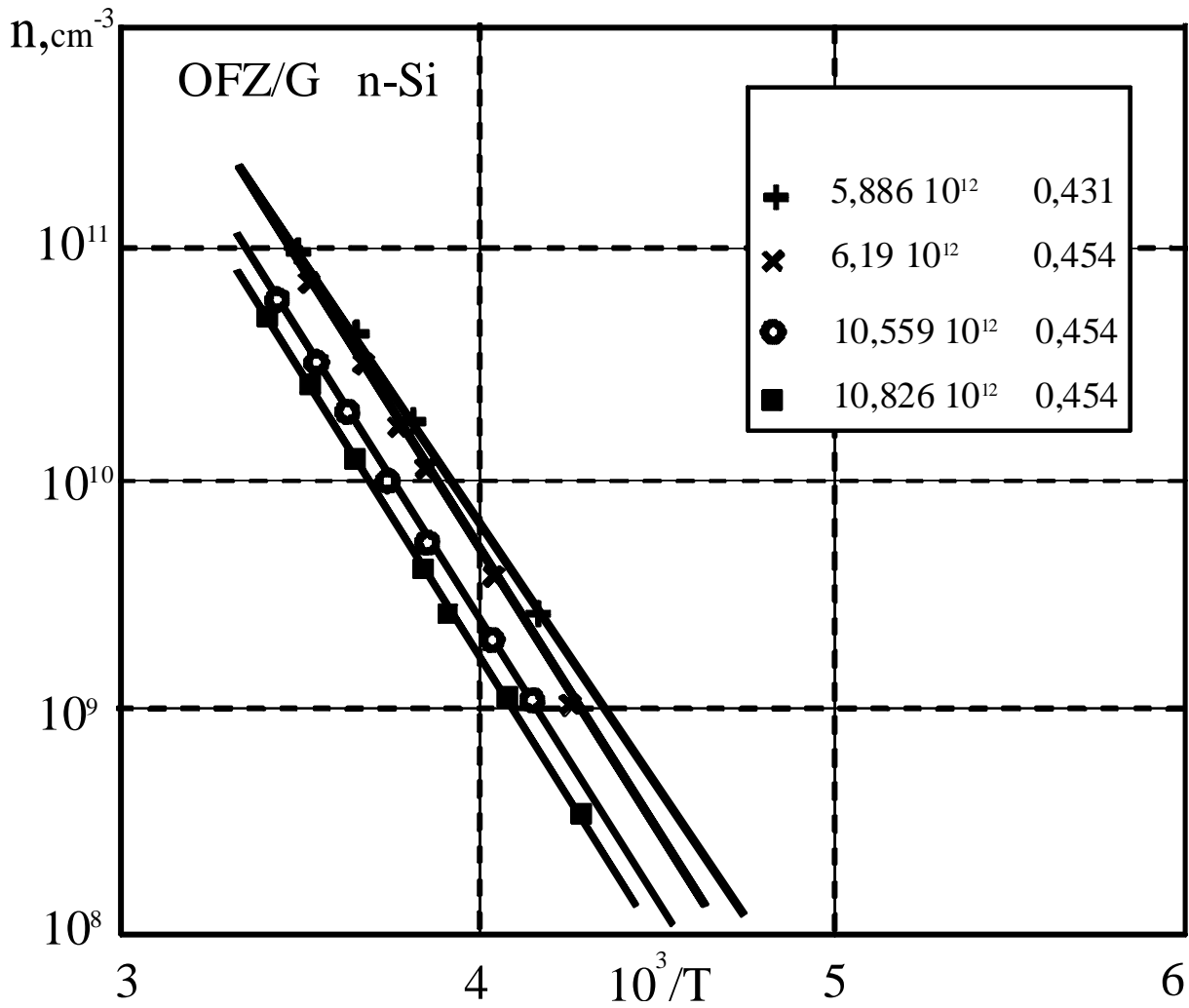


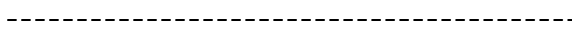
Fig 6. Temperature dependence of carrier concentration of OFZ/G n-type Si at different doses 24 GeV protons

$$n = N_{0,49}$$

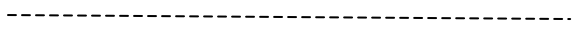
$$E_{\text{eff}} = \frac{E_{0,43} + E_{0,49}}{2} \sim E_{0,455}$$



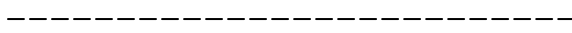
E_c



0,43 $V(V_2)$



0,45 $V(E_{\text{eff}})$



E_F