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ЕРЕВАНСКИЙ ФИЗИЧЕСКИЙ ИНСТИТУТ

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YEREVAN PHYSICS INSTITUTE

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THE INTERACTION OF IMPURITY OXYGEN IN SILICON
WITH VACANCIES

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Silicon specimens irradiated with 50 MeV electrons, containing along with isolated oxygen atoms more complicated oxy-quasi-molecules of SiO_n ($n = 1, 2, 3, \dots$) type are investigated. At isochronal and isothermal annealing in the temperature region 300-350°C, besides the reaction of vacancy capturing by oxygen atoms with formation of A-centres, there occur more complicated reactions with participation of vacancies, A-centres, oxygen containing quasi-molecules, and a variety of sinks. The kinetics of the processes taking place at irradiation and annealing was studied with respect to the measurement of IR absorption spectra in the region 1-16 μm . A model is suggested to describe the observed processes that differ qualitatively from those taking place in specimens containing completely dissociated oxygen.

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ВЗАИМОДЕЙСТВИЕ ПРИМЕСНОГО КИСЛОРОДА В КРЕМНИИ
С ВАКАНСИЯМИ

Исследовались облученные электронами с энергией 50 МэВ образцы кремния, содержащие наряду с изолированными атомами кислорода более сложные, включающие кислород, квазимолекулы типа SiO_n ($n = 1, 2, 3, \dots$). При изохронном и изотермическом отжиге в температурном интервале $300\text{--}350^\circ\text{C}$, кроме реакции захвата вакансий атомами кислорода с образованием А-центров, происходят реакции с участием вакансий, А-центров, кислородосодержащих квазимолекул и различных стоков. Кинетика процессов, происходящих при облучении и отжиге, изучалась по измерению спектров ИК-поглощения в области 1–16 мкм. Предложена модель, описывающая наблюдаемые процессы, качественно отличающиеся от модели процессов, происходящих в образцах с полностью диссоциированным кислородом.

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1. Introduction

Oxygen atoms in silicon may be in different states, some of which are electrically active (thermodonors SiO_4 , thermoacceptors SiO_2 and SiO_3)^[1-3], some are optically active^[4-6], and some are paramagnetic (e.g. A-centres which have captured an electron)^[7, 8]. Therefore, a reconstruction of impurity oxygen configurations in silicon essentially affects the specimen properties. In this work such a reconstruction was achieved by irradiating silicon specimens with fast electrons (50 MeV) at room temperature, producing vacancies V and "odd" silicon atoms Si_1 which were mobile under the irradiation conditions, and subsequent heating. The oxygen impurity concentration in the specimens treated, highly exceeded those of other impurities that could capture vacancies V , and the high irradiation doses used led to a substantial reconstruction of oxygen containing defects.

A similar investigation has been reported in^[5] which shows that the disappearance of A-centres following silicon irradiation at 300°C leads to the appearance of a band with a maximum 887 cm^{-1} ; this fact is naturally attributed to the reaction $A + \text{O}_2 \rightarrow \text{VO}_2$ which takes place owing to A-centre diffusion. Heating the specimens to higher temperatures resulted in a further reconstruction of IR bands, which is explained by the appearance of centres with three and more O_1 atoms.

This relatively simple picture is possible in silicon specimens where the concentration of isolated oxygen atoms is much higher than the oxygen concentration in other oxygen containing centres. However, a lot of silicon specimens exhibit a considerable concentration of more complicated oxygen containing centres in the form of isolated quasi-molecules SiO_n ($n = 2, 3, 4, \dots$); quasi-molecules containing oxygen and other impurities (carbon, boron); microcrystalline precipitates of another phase (SiO) of different dimensions. Such centres contribute to the absorption band of local vibrations of the $Si-O$ bond near $9\mu m$ (which is usually poorly resolved at room temperatures).

The present work was done on silicon specimens which included isolated oxygen atoms as well as more complicated oxygen containing centres, O^* . During irradiation the vacancies formed are captured by oxygen atoms leading to A-centres and also by O^* centres; With subsequent heating to temperatures activating A-centre motion, a variety of sinks come to participate in A-centre capture.

2. Experiment and Measurement Results

Specimens of n- and p-type silicon containing oxygen of $(0.5 \pm 1.3) \times 10^{18} \text{ cm}^{-3}$ concentration and carbon of $(0.3 \pm 0.8) \times 10^{18} \text{ cm}^{-3}$ concentration were irradiated on the linear accelerator of the Yerevan Physics Institute with 50 MeV electrons. The irradiation dose was varied from 10^{17} to 10^{18} e/cm^2 at room temperature. The dominating defects were divacancies V_2 and A-centres. The concentrations of other radiation defects connected with donors or acceptors of different chemical nature were considerably lower.

The infrared absorption spectra of the irradiated specimens showed a number of absorption bands with maxima at wavelengths 1.8, 3.3, 3.45 and

3.6 μm (divacancies), 12 μm (A-centre), and 11.6 μm whose intensity increased as the dose grew. The intensities of the 3 μm (oxygen) and 16 μm (carbon) bands observed before irradiation decreased as the dose grew.

Following irradiation, isochronal and isothermal annealing was carried out successively at temperature ranging from 20 to 350 $^{\circ}$ C. Annealing at 270 C for 8 h led to the complete disappearance of absorption due to divacancies; the absorption intensity in the 829 cm^{-1} region somewhat increased. This is obviously due to reactions between impurity oxygen and vacancies released at annealing of divacancies. Thus the subsequent study of annealing in the interval 300 $^{\circ}$ C < T < 350 $^{\circ}$ C was carried out with silicon specimens where defects containing vacancies coupled with oxygen atoms were dominant.

Fig. 1a and 1b show the absorption spectra at room temperature in the region 1106 cm^{-1} before irradiation and its variation at different irradiation doses, and during annealing, respectively. An approximation of the measured absorption curve in the form of a superposition of Gaussians is used to determine the spectrum structure. The band parameters, including the maximum positions and the number of bands, were varied and were defined from the mean-square minimum condition using a BESM-6 computer [9].

The points in Fig.1b show the elementary bands obtained after expansion of the experimental curve 1.

The kinetics of intensity variation of the elementary absorption bands during irradiation (Fig.2a) and isothermal annealing (Fig.2b) show that the absorption bands 1045, 1098, and 1106 cm^{-1} are sensitive to both irradiation and annealing. The relation between the intensities of these bands changes with irradiation and annealing; therefore, the bands must be referred to different centres. The intensities of the bands 1098 and 1106 cm^{-1} decrease with irradiation, the band 1098 cm^{-1} decreases more rapidly than the band 1106 cm^{-1} . The intensity of the band 1098 cm^{-1} grows with annealing

while that of the band 1106 cm^{-1} decreases (before they reach saturation following prolonged isothermal annealing). The bands 1150 and 1202 cm^{-1} practically vary neither during irradiation nor during annealing, i.e. they are not related to the centres participating in the reactions. The band 1045 cm^{-1} which has been identified as the complex $\text{SiO}_4^{[10]}$ was observed neither in the original specimen nor in the irradiation process at room temperature. It first appeared during annealing of the irradiated specimens at $T \approx 100 \text{ C}$.

Fig.3a shows the dose dependences of the absorption coefficient of the 829 and 862 cm^{-1} bands. The first band is identified with $O_I V$, i.e. with A-centres, and the second one, which we shall call X-band, is due to more complicated centres containing vacancies. The ratio of absorption intensities between X-band and A-band differs from specimen to specimen (in [5] the generation of A-centres with irradiation was not followed by X-centres at all); hence, the band 862 cm^{-1} appears when vacancies are captured by centres other than O_I .

The absorption spectra in this wavelength interval measured after isochronal annealing differ from specimen to specimen. The annealing leads to a decrease and vanishing of the 862 and 829 cm^{-1} bands at 300 to 340° C . In type I specimens (with oxygen concentration $\sim 1.3 \times 10^{18-3} \text{ cm}^{-3}$) this is accompanied by the appearance of the 887 cm^{-1} absorption band ($V O_2$ centre). In type II specimens (with oxygen concentration $(3 \text{ to } 6) \times 10^{17-3} \text{ cm}^{-3}$) the annealing of 862 and 829 cm^{-1} bands results in only weak traces of an absorption increase at 887 cm^{-1} . The main processes in annealing are those of an intensity decrease of the $1106 \text{ cm}^{-1} O_I$ band and an intensity increase of the 1098 cm^{-1} band (Fig.2b).

Fig 3b shows experimental values of the absorption coefficient during isothermal annealing of the 862 and 829 cm^{-1} bands at temperatures 310 to 340° C for both type I and type II specimens, and also the appearance kine-

tics of the 887 cm^{-1} band in type I specimens. The annealing of A- and X-centres does not correlate with the annealing of the $9\mu\text{m}$ band and does not lead to the restoration of the original oxygen absorption band. The annealing of type II specimens results in a broader $9\mu\text{m}$ integral band, but the absorption in the maximum of the summary envelope curve does not increase; it decreases in type I specimens. This means that following annealing of an A-centre its component oxygen O_I atoms do not return to their original pre-irradiation positions.

It is notable that the absorption of O_I centres at 1106 cm^{-1} in a definite time interval of isothermal annealing decreases not only in specimens where the absorption of VO_2 centres occurs, at 887 cm^{-1} , because isolated O_I atoms disappear after capturing A-centres, but also in specimens where such a reaction is ineffective and VO_2 centres do not appear.

The IR spectra of the examined specimens were measured also at low temperatures (dotted curve in Fig.1a in the helium temperature region). Here the fine structure of the $9\mu\text{m}$ band is well resolved; the maximum positions on the dotted curve fit well those given in [4, 11].

3. Discussion

According to Section 2, in non-irradiated silicon specimens one should take into account two types of centres which capture vacancies after irradiation, viz. isolated O_I atoms (1106 cm^{-1}) and O^* centres (1098 cm^{-1}). The latter apparently are some microscopic oxygen atom conglomerations, such that the oscillation frequency of the Si - O bond in the conglomerate near the value of 1098 cm^{-1} depends only slightly on the number of oxygen atoms in the conglomerate. The capture of a vacancy by a conglomerate or the pre-

sence of an impurity atom in it may substantially distort the oscillation frequency of the corresponding bond; but it affects little the frequencies of the other conglomerate bonds. Therefore, Si - O bonds of oxygen conglomerates, which differ somewhat by the number of oxygen atoms or by the presence of other impurity atoms, may be responsible for the absorption at 1098 cm^{-1} .

As we see from Section 2, O^* centres have a somewhat larger summary V capture cross-section than O_I centres. When V is captured by an O_I atom the absorption at 1106 cm^{-1} decreases and an A-centre appears, which gives rise to absorption at 829 cm^{-1} . When V is captured by an O^* centre the absorption at 1098 cm^{-1} decreases owing to the distortion of a Si - O bond in the O^* -centre and the absorption of the distorted bond occurs at 862 cm^{-1} (X-band). The other Si - O bonds in the O^*V centre remain as before.

To interpret the annealing results we considered several alternative mechanisms.

However, we were led by the experimental evidence to dwell on the mechanism of A-centre annealing, which consists in the activation of the motion of an A-centre as a whole, at $T \approx 300^\circ \text{ C}$, and its subsequent capture by other centres.

According to our scheme, at $T = 300$ to 310° C when A-centre motion is activated, one can expect A-centre detachment from the O^* centre. But it follows from the results of Section 2 that the binding energy of an oxygen atom in the conglomerate is sufficiently high so that an isolated vacancy rather than an A-centre is detached from the O^*V centre. The absorption of the Si - O bond near the vacancy in the O^*V centre disappears; the O^* centre absorption is restored at 1098 cm^{-1} and the X-band disappears.

A secondary trapping of the free vacancy by some O^* centre is possible but this position of V is unstable again, and such a process only prolongs the annealing time. If, on the other hand, the free vacancy is captured by an O_I atom, an A-centre appears which is identical to those formed directly at irradiation and is mobile at the annealing temperature.

The diffusion of A-centres leads to their capture by O_I and O^* centres with the attendant decrease in A-centre absorption at 829 cm^{-1} . VO_2 centres (887 cm^{-1}) result from the $A + O_I$ reaction. With the A-centre capture by O^* there appears an O^*V centre which contains, however, an odd oxygen atom. This circumstance, as mentioned before, distorts little the O^* - centre absorption at 1098 cm^{-1} , but changes the absorption from the A-centre at 829 cm^{-1} to the 862 cm^{-1} X-band (Si - O bond in O centre + V). Then V is again detached from the O^*V centre and so on. This type of centre which releases vacancies after capturing A-centres can be called a vacancy release centre.

The above picture explains in qualitative terms the effect of decreased O_I atom absorption during annealing even in specimens where A-centre capture by O_I atoms is not very effective owing to strong A-centre sinks on O^* . The vacancies which are released from O^*V centres act as catalysts to coagulate O_I atoms into conglomerates O^* by transporting the atoms to O^* in the form of A-centres.

If no vacancy capture centres other than O_I and O^* would exist, the annealing should result in the complete disappearance of isolated oxygen atoms and the 1106 cm^{-1} absorption band. But capture takes place; dislocations, specimen surface, or other impurity centres which are either optically inactive or have lower concentrations, may be effective V capture centres. Here a vacancy recombination is possible, i.e. its filling by either

a silicon atom or an impurity atom; such a centre can be called a vacancy annihilation centre. In case a vacancy is strongly bound to the centre but does not annihilate, it can participate in various reactions at higher temperatures⁵. These centres may be called vacancy trapping centres at appropriate temperatures.

The availability of both vacancy annihilation and vacancy trapping centres leads to a gradual decrease in the free V concentration and stop the decrease in O_I band intensity during annealing which was mentioned in Section 2.

4. Kinetics of Bound Vacancy Annealing

The simplest case examined in [5] corresponds to A-centre capture by O_I atoms with formation of VO_2 centres. The kinetics of this process is described by

$$\frac{dC_A}{dt} = -\chi_{AO} C_A C_{O_I}, \quad \frac{dC_{O_I}}{dt} = \chi_{CA} C_A C_{O_I}, \quad \frac{dC_{VO_2}}{dt} = \chi_{AO} C_A C_{O_I}, \quad (1)$$

where $C_A = C_A^\circ$, $C_{O_I} = C_{O_I}^\circ$ at $t=0$ (beginning of annealing). Here and further on C_A , C_{O_I} , C_{VO_2} are concentrations of corresponding centres, $\chi_{\alpha\beta}$ are probabilities of elementary acts of the reaction between α and β . The integration of system (1) results in the solution

$$C_A = C_A^\circ \frac{C_{O_I}^\circ - C_A^\circ}{C_{O_I}^\circ \exp[\chi_{AO} (C_{O_I}^\circ - C_A^\circ)t] - C_A^\circ}; \quad (2)$$

$$C_{O_I} = C_{O_I}^\circ \frac{C_{O_I}^\circ - C_A^\circ}{C_{O_I}^\circ - C_A^\circ \exp[-\chi_{AO} (C_{O_I}^\circ - C_A^\circ)t]}$$

As one can see, the kinetics of A-band degradation and VO_2 band growth differ from each other and at some t noticeably differ from a first-order kinetics. In particular, there exists some "delay" in C_A and C_{O_I} variations at the beginning of annealing: $C_A, C_{O_I} \approx \text{const}$ at $t \leq [\gamma_{AO}(C_{O_I} - C_A^0)]^{-1}$. This corresponds to experimental observations described in [5].

To interpret the annealing results on specimens having a $9 \mu\text{m}$ band of complex structure in the light of the facts described in Sections 2 and 3 we should consider the following system of equations:

$$\frac{dC_{O_I^*}}{dt} = \omega_x C_x - \gamma_{O^*V} C_{O^*} C_V; \quad \frac{dC_{O_I}}{dt} = -\gamma_{OV} C_{O_I} C_V + \omega_A C_A; \quad (3)$$

$$\frac{dC_x}{dt} = \gamma_{O^*V} C_{O^*} C_V - \omega_x C_x + \gamma_{AO^*} C_{O^*}; \quad \frac{dC_A}{dt} = \gamma_{OV} C_{O_I} C_V - \gamma_{O^*A} C_{O^*} C_A - \omega_A C_A;$$

$$- \frac{dV}{dt} = \omega_x C_x - \gamma_{OV} C_{O_I} C_V - \gamma_{O^*V} C_{O^*} C_V - \omega_s C_V + \omega_A C_A,$$

where ω_x is inverse X-centre lifetime relative to O^* and V decay; ω_s is the inverse lifetime of a free vacancy relative to the capture by a vacancy annihilation or trapping centre; C_{O^*} is the concentration of oxygen atoms in all the O^* centres.

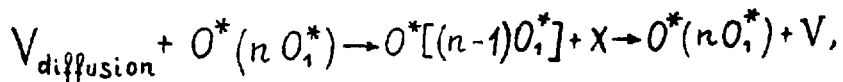
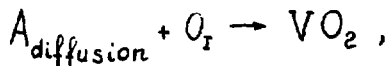
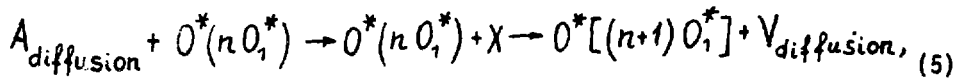
The rates of the processes which take place during annealing are written in the following form: $\omega_x C_x$ for the thermal decomposition of X-centres into O^* and V; $\gamma_{O^*V} C_{O^*} C_V$ for the free-vacancy capture by O^* centre when an O atom in O^* centre begins absorbing at X-centre frequency; $\gamma_{OV} C_{O_I} C_V$ for the free-vacancy capture by an O_I atom leading to an A-centre; $\gamma_{AO^*} C_A C_{O^*}$ for the A-centre capture by the O^* complex, here an O atom used to absorb at the A-centre frequency will absorb at the O^* centre frequency; $\omega_s C_V$ for the free-vacancy

capture by an immobile annihilation or trapping centre.

Among the variables $C_{O_1^*}$, C_{O_I} , C_X , C_A , C_V only four are independent, since there is a linear relation describing the law of conservation of the total number of C_O° oxygen atoms:

$$C_X + C_{O_1^*} + C_{O_I} + C_A = C_O^\circ \quad (4)$$

The complete set of reactions under consideration in the temperature range 300 to 350° C can be presented in the form



V (diffusion) \rightarrow trapping and annihilation of V .

Here $O^*(n O_1^*)$ means an O^* centre which consists of atoms of O .

This set of reactions leaves the total number of oxygen atoms in the specimen constant,

$$C_X + C_A + C_{O_I} + C_{O_1^*} + 2C_{VO_2} = C_O^\circ, \quad (6)$$

where $C_O^\circ = \text{const}$, C_X , C_A , C_{O_I} are the concentrations of the respective centres.

The solution of the system of equations describing the set of reactions (5) sets up considerable problems. An approximate solution for type II

specimens, where the VO_2 accumulation is negligible, is discussed in [12]. In particular, it can be shown that in this case at $t \rightarrow \infty$ the solution has the form:

$$C_A \rightarrow 0; C_X \rightarrow 0; C_V \rightarrow 0; C_{O_I} \rightarrow C_{O_I}^0 - C_{O_I}^\infty;$$

$$C_{O_I} \rightarrow C_{O_I}^\infty \equiv C_{O_I}^0 \exp[-\gamma_{OV} \tau_s (C_A^0 + C_X^0)],$$

where $C_{O_I}^0$, C_A^0 , C_X^0 are the initial concentrations of O_I -, A-, and X-centres at annealing. γ_{OV} is the probability of vacancy capture by an O_I atom, τ_s is the vacancy lifetime with respect to the capture by an annihilation or trapping centre.

5. Conclusions

In the temperature range 300 to 350° C the diffusion activation of A-centres occurs, which results in their capture by different types of centres. Some of them hold vacancy (vacancy trapping centres) which can participate in reactions between point defects at higher temperatures. On other centres capturing A-centres the vacancy annihilates. A third type of capture centres holds the oxygen atom alone. A vacancy is set free following A-centre capture by O_I atoms on other defects. A part of the released vacancies captured on O_I transports isolated oxygen atoms in the form of A-centres to other centres, i.e. acts as a catalyst for the reaction of oxygen atom coagulation in silicon at temperatures at which in the absence of vacancies O_I atoms are immobile and do not react. The number of released vacancies decreases in time, owing to the capture by annihilation and recombination centres. Such a decrease in the number of free vacancies can be attributed to their capture by interphase boundaries, surfaces,

dislocations, or other defects. The set of reactions with A-centre participation is determined by the assortment of defects in the original specimen and can vary widely with production conditions, doping, and thermal treatment of the specimens.

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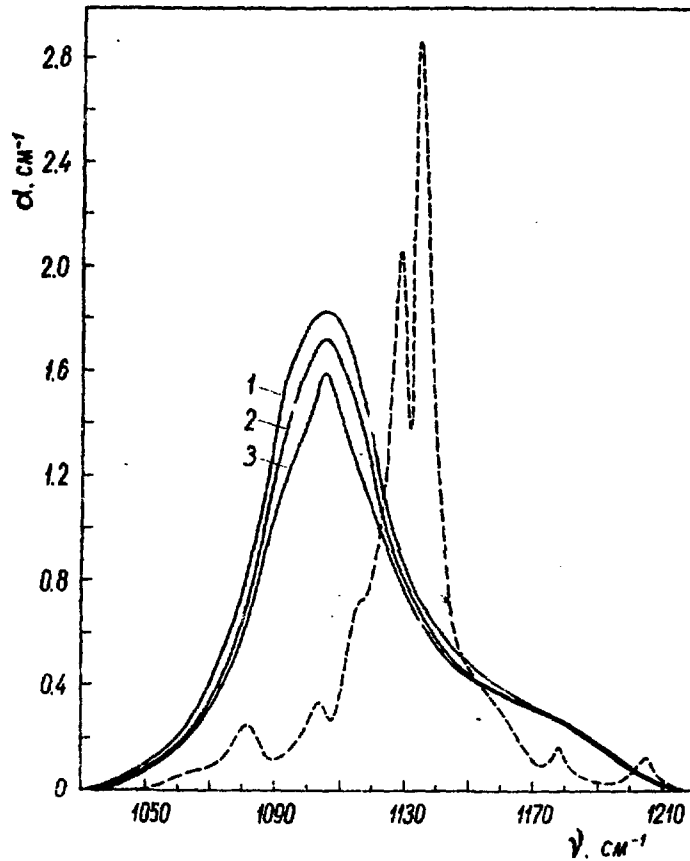


Fig.1. Change of the shape of the $9\ \mu\text{m}$ spectral absorption band in p-type silicon specimens with oxygen concentration of $6 \times 10^{17}\ \text{cm}^{-3}$ as a result of irradiation with 50 MeV electrons measured at room temperature.

- (1) - Before irradiation, (2) $1.3 \times 10^{17}\ \text{el/cm}^2$,
 (3) $1.1 \times 10^{18}\ \text{el/cm}^2$, Dotted curve: the $9\ \mu\text{m}$ absorption band obtained at liquid helium temperature after irradiation by $1.1 \times 10^{18}\ \text{el/cm}^2$;

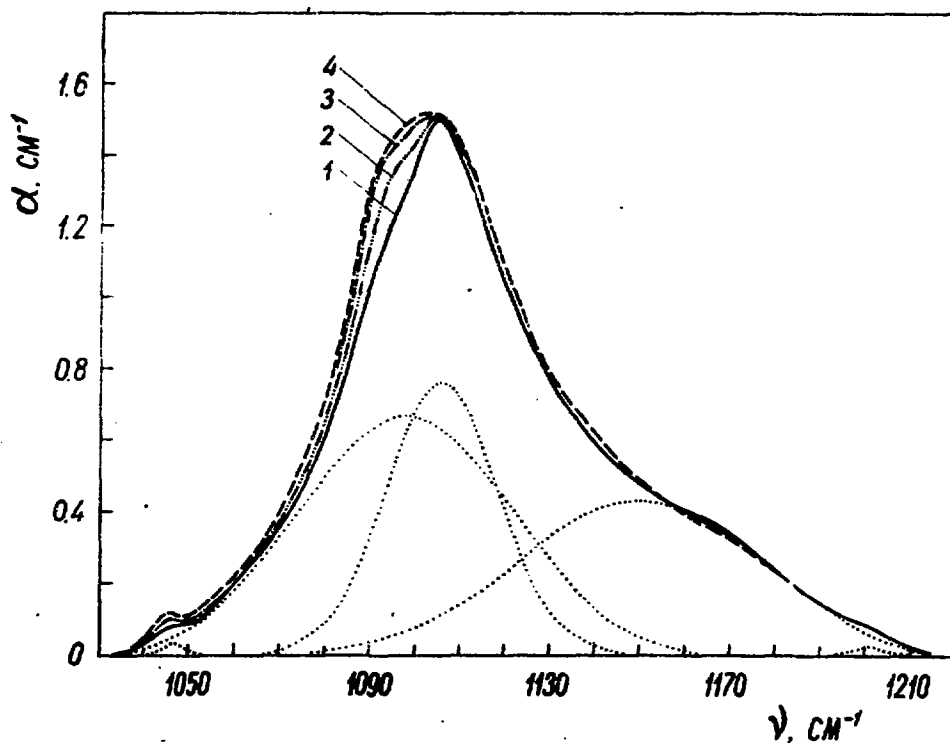


Fig.2 During isothermal annealing at $T = 320^{\circ} \text{C}$:

(1) Initial point (the specimen irradiated with $1.1 \times 10^{18} \text{ eI/cm}^2$ and annealed at $T=275^{\circ} \text{C}$ for 460 min), (2) 30 min later, (3) 80 min later, (4) 380 min later. The dotted line shows the elementary bands obtained after decomposition of the band (1).

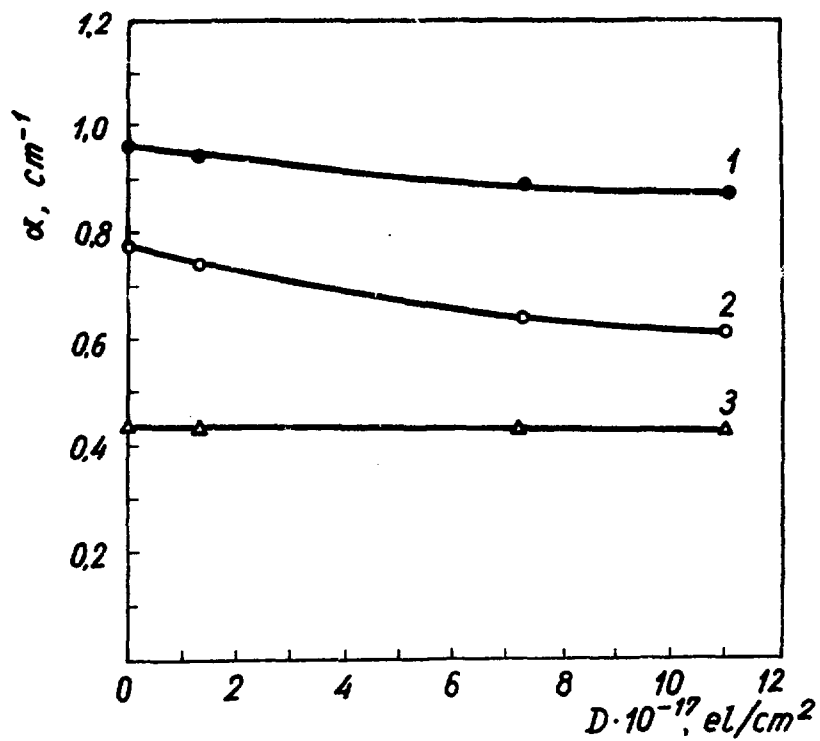


Fig.3 Dose dependence of the absorption coefficients of the decomposed elementary bands with maxima at frequencies (1) $\nu = 1106$, (2) 1098 , (3) 1150 cm^{-1} .

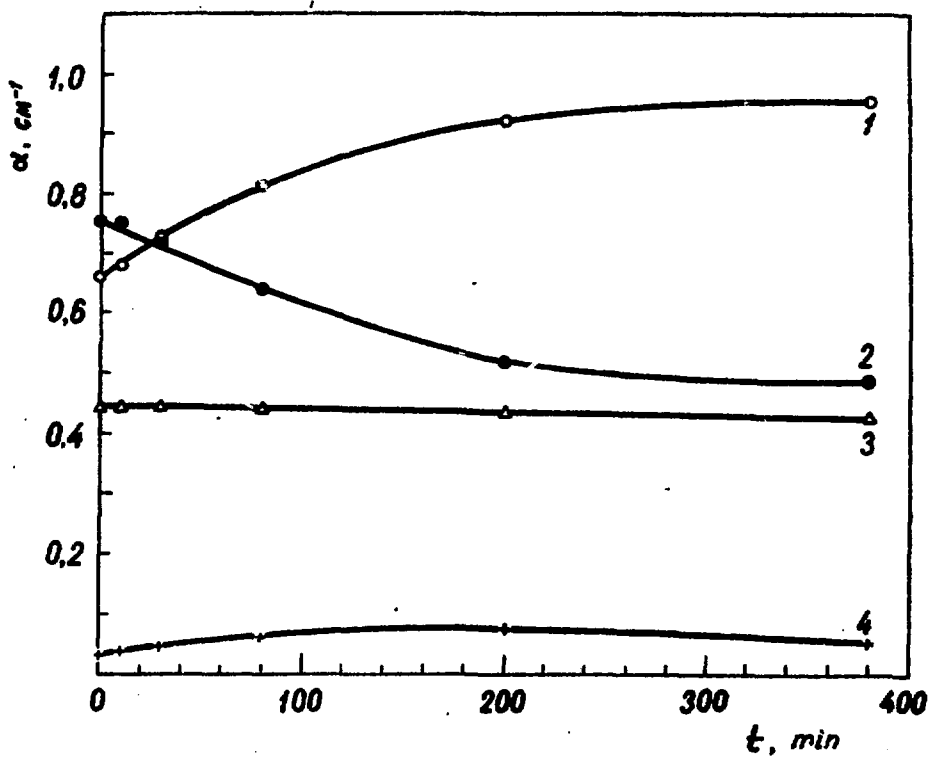


Fig.4 Kinetics of 320°C isothermal annealing of the decomposed elementary bands with maxima at frequencies
 (1) 1098, (2) 1106, (3) 1150, (4) 1045 cm^{-1} .

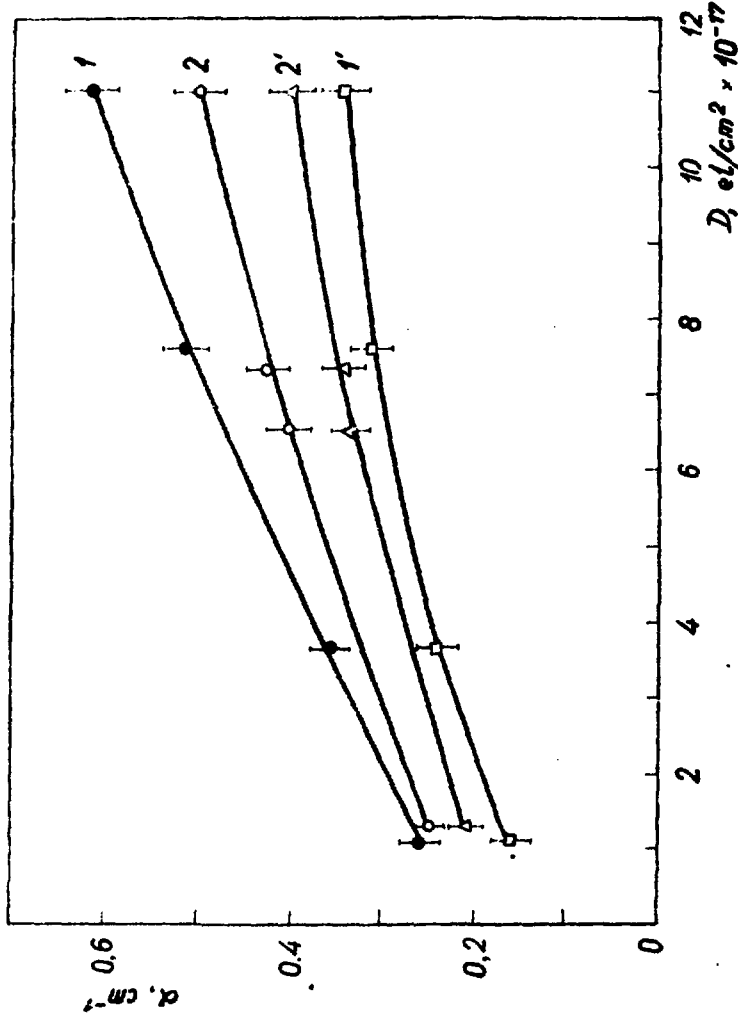


Fig.5 Dose dependence of absorption coefficients of bands: (1), (2)

829 cm^{-1} and (1'), (2') 862 cm^{-1} . (1) and (1') Si-p-20

($\rho \approx 20 \Omega$ cm, with concentrations of oxygen $\approx 1.3 \times 10^{18} cm^{-3}$ and carbon $\approx 1.7 \times 10^{17} cm^{-3}$); (2) and (2') Si-p-250

($\rho \approx 250 \Omega$ cm, with concentrations of oxygen $\approx 6 \times 10^{17} cm^{-3}$, carbon $\approx 6 \times 10^{17} cm^{-3}$);

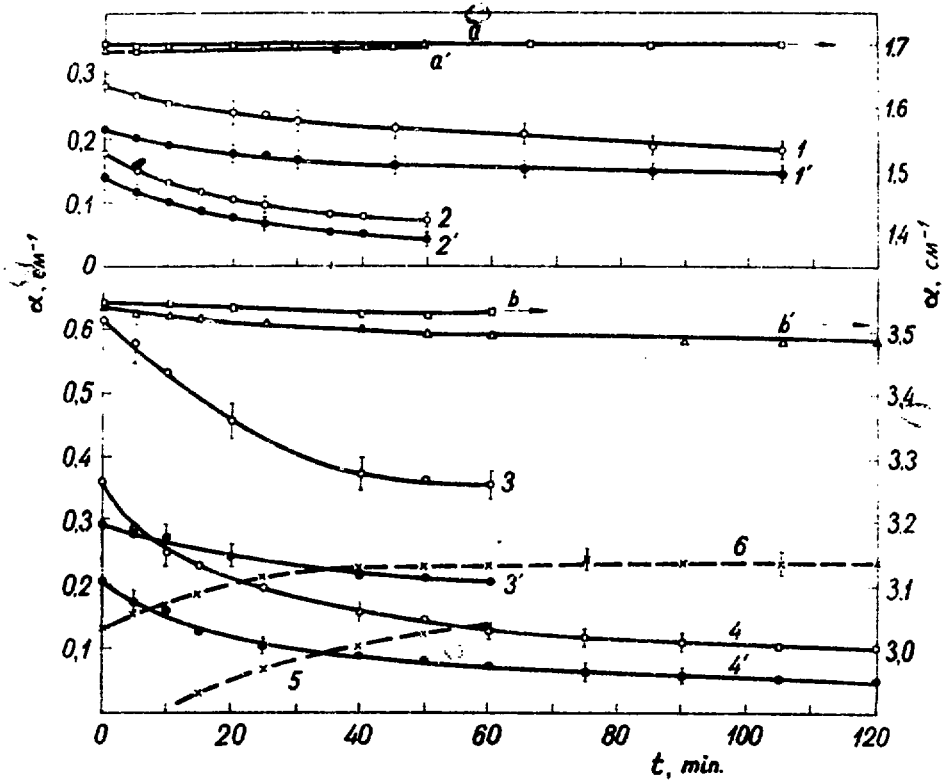


Fig.6 Kinetics of isothermal annealing of absorption bands 9, 11.6 and 12 μm of the specimens Si-p-250:

$T = 310^{\circ}\text{C}$, (a) 9 μm , (1) 829 cm^{-1} , (1') 862 cm^{-1} ,

$T = 340^{\circ}\text{C}$, (a') 9 μm , (2) 829 cm^{-1} , (2') 862 cm^{-1} ,

Si-p-20: $T = 320^{\circ}\text{C}$, (b) 9 μm , (3) 829 cm^{-1} , (3') 862 cm^{-1} ,

$T = 340^{\circ}\text{C}$, (b') 9 μm , (4) 829 cm^{-1} , (4') 862 cm^{-1} .

397 cm^{-1} band (5) $T = 320$, (6) 340°C .

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