Trapping and anomalous scattering of majority carriers by interacting centers in plastically deformed *n*-type silicon

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An experimental investigation was made of the Hall effect in deformed *n*-type silicon single crystals with an initial conduction electron density $n = 5\times10^{13}$–2×10^{14} cm^{-3}. The photo-Hall effect was used in an analysis of the energy spectra of crystals with dislocations. It was found that when the dislocation density was $N_D = 10^7$–10^8 cm^{-2}, the scattering of carriers was anomalously strong but could be reduced by monochromatic illumination with photons of a certain energy $h\nu$. Such illumination ionized electrostatically the interacting deep centers concentrated in the point-defect "atmospheres" at dislocations, i.e., illumination reduced the trapping of carriers and the depth of the minimum in the temperature dependence of the electron mobility $\mu_n(T)$. The results obtained for crystals with dislocations were compared with the data on the scattering of carriers by *A* and *E* centers generated by irradiation. It was established that the minimum of the dependences $\mu_n(T)$ exhibited by deformed and irradiated crystals was due to the trapping of conduction electrons.

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The solution of the problem of the electrical activity of dislocations in germanium and silicon meets with considerable difficulties, primarily because it is not possible to generate "pure" dislocations, i.e., dislocations without "atmospheres" of impurity atoms and point defects, particularly vacancies formed by plastic deformation. Covalent crystals with the diamond lattice undergo brittle fracture at low temperatures $T < 200°$C. Therefore, if plastic deformation is desired, germanium has to be heated to temperatures of $T > 350°$C and silicon to $T > 450°$C.

Recent investigations carried out on deformed high-purity germanium and silicon crystals demonstrated the existence of new effects: 1) the dominant role of the formation and diffusion of vacancies in dislocation glide [1,2] and 2) the appearance (at a dislocation density $N_D = 10^7 \times 10^8$ cm^{-2}) of a sharp minimum in the temperature dependences of the carrier mobility in p-type germanium at 16°K (Ref. 3) and in n-type silicon at 100–150°K (Ref. 4). Such a minimum was observed in n-type Si with carrier densities $n = N_d - N_a = (3-4) \times 10^{15}$ cm^{-3}, in which the presence of dislocations in a density of $N_D = 2 \times 10^8$ cm^{-2} reduced the conduction electron density only slightly (< 10%) but did not affect the temperature dependence which still corresponded to the ionization of shallow donors. Therefore, it was suggested in [4] that the anomalous scattering of carriers was due to an increase in the concentration of complexes (trapping centers) in the point-defect atmospheres surrounding the dislocations.

Similar effects in neutron-irradiated silicon were observed by Wertheim [5] who suggested, because of the low concentration of deep centers $N_{dc} \le n = 10^{14}$ cm^{-3} introduced by irradiation, that disordered p-type regions appeared in the n-type material and the dimensions of these regions were comparable with the wavelength of the scattered carriers.

Subsequent investigations of germanium irradiated with γ rays and fast electrons [6,7] demonstrated a considerable reduction in the carrier mobility μ in the range of temperatures in which conduction electrons were scattered by charged centers and this reduction was not correlated with the concentration of such centers (a reduction in the mobility in the range $T < 200°$K for $N_{dc} \sim 10^{14}$ cm^{-3} was equivalent to a reduction in the electron mobility μ_n corresponding to a charged donor N_d or acceptor N_a concentration of the order of 10^{17} cm^{-3}). Sometimes the dependences $\mu_n(T)$ had a minimum at $T \le 100°$K. Wertheim [6] and Crawford and Cleland [7] explained these phenomena by proposing a model in which they allowed for the inhomogeneity of the irradiated crystals; this model was developed further and discussed in [8]. The anomalously strong scattering of carriers in deformed and irradiated germanium crystals with $n = 5 \times 10^{14}$–5×10^{15} cm^{-3} had much in common with the corresponding scattering in silicon but the solution of the problem could not be reduced simply to the appearance of an inhomogeneity and behavior of conduction electrons in an inhomogeneous crystal. [6,7]

The reduction in $\mu_n(T)$ in the temperature range corresponding to the scattering by charged centers could not be explained by introducing a parameter z, representing the charged state of the centers, as was done by Golubev et al. [9] For example, in the case of germanium irradiated with 2.5 MeV electrons in a dose of 8×10^{14} cm^{-2}, it was found [9] that μ_n decreased strongly in the range $T < 150°$K and the calculations carried out on the assumption that $z = 3$ gave values of μ_n which were an order of magnitude higher than the experimental mobilities and could not explain the appearance of minima in the dependences $\mu_n(T)$.

Bearing in mind all these effects, we decided to investigate the influence of charging (low excitation rates) of some of the deep centers in deformed silicon on the anomalous increase in the strength of scattering. The nature of the electrical activity of dislocations and the type of scattering centers in the point-defect atmospheres surrounding dislocations were identified by comparing the influence of dislocations on the dependences $\mu_n(T)$ with the influence of A and E centers created by irradiation.

EXPERIMENTAL METHOD

We used dislocation-free n-type silicon single crystals doped with phosphorus in which the electron density was in the range $n = 5 \times 10^{13}$–2×10^{14} cm^{-3} and the oxygen concentration was $N^O < 10^{16}$ cm^{-3} (for crystals prepared by zone melting) and $N^O \sim 4 \times 10^{17}$ cm^{-3} (for crystals grown by the Czochralski method). Plastic deformation increased the dislocation density to $N_D = 10^7-10^8$ cm^{-2} and it was performed by uniaxial compression along the $[\bar{1}\bar{2}3]$ axis at T = 700°C under a stress of σ = 2.5 kg/mm^2 acting in the principal slip plane $(1\bar{1}1)$. After deformation, the samples were cooled either slowly (30 min) together with the oven or fast (30 sec) by directing a stream of nitrogen vapor onto a sample. The dislocation density was deduced from the etch pits by averaging over many fields of view and the error in this determination was $\pm 5 \times 10^6$ cm^{-2}.

The influence of dislocations was compared with the influence of radiation defects by irradiating some of the samples with 2.2 MeV electrons (2×10^{14} cm^{-2} dose) or initially with ~24 MeV protons (10^{11} cm^{-2} dose) and then with electrons. Preliminary proton irradiation with a dose of ~10^{11} cm^{-2} did not affect the values of n and μ_n throughout the investigated temperature range but helped to induce (at lower electron doses) a minimum in the dependences $\mu_n(T)$ at T = 100°K. After irradiation with just electrons, a minimum in $\mu_n(T)$ did appear but it was located at T < 77°K possibly because fewer interacting centers were formed.

The role of charging of deep centers was investigated by the photo-Hall effect: electrons from levels of a given type were excited to the conduction band by light which passed through an IKS-21 monochromator. The temperature dependences of n and μ_n investigated in the range T = 77–450°K by the compensation method in a magnetic field of H = 10 kOe and the Hall factor was assumed to be unity. Changes in point-defect atmospheres surrounding dislocations were produced by isothermal annealing at T = 700°C and by isochronous annealing at 70–550°C, in which case cooling was fast ($\leq$10 sec).

RESULTS

The dependences n(1/T) exhibited by the original samples in the range T = 77–300°K corresponded to total ionization of shallow donors (phosphorus atoms): n = const (depletion region) and the carrier mobility

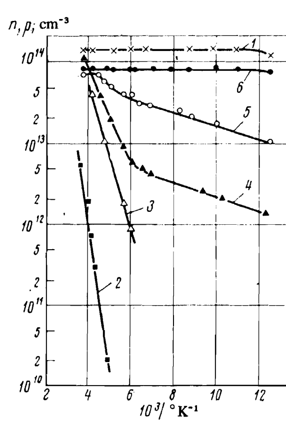

FIG. 1

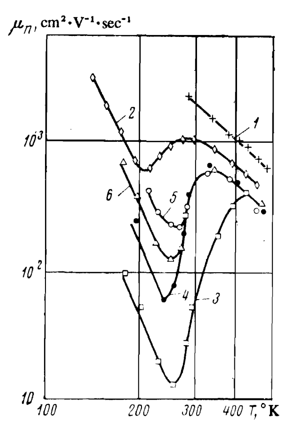

FIG. 2

FIG. 1. Temperature dependences of the carrier density in a silicon sample with $N_D = 5 \times 10^7$ cm^{-2}: 1) original sample; 2) after deformation; 3)–6) after annealing at the deformation temperature for 1, 6, 26, and 37 min, respectively.

FIG. 2. Temperature dependences of the mobility of the majority carriers in darkness: 1) original sample; 2) after deformation producing $N_D = 5 \times 10^7$ cm^{-2}; 3) $N_D = 2.2 \times 10^8$ cm^{-2}; 4) same as 3) but in the presence of hν = 0.4 eV (2.8 μ) illumination; 5) in the presence of hν = 0.17 eV (6.2 μ) illumination; 6) in the presence of hν = 1.15 eV (1.13 μ) illumination.

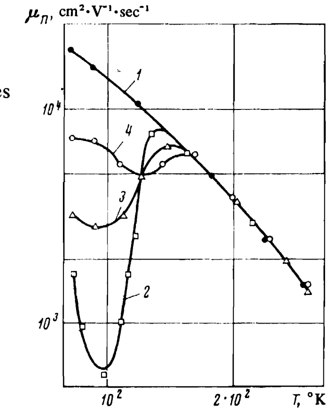

FIG. 3. Temperature dependences of the mobility of the majority carriers in samples irradiated with 24 MeV protons (10^{11} cm^{-2} dose) and with 2.2 MeV electrons (10^{14} cm^{-2} dose): 1) initial mobility; 2) mobility in darkness after irradiation; 3) mobility during illumination with hν = 1.15 eV photons; 4) during illumination with hν = 0.4 eV photons.

$\mu_n(T)$ corresponded to the scattering by phonons $\mu_n \propto T^{-2.6}$, as demonstrated in Figs. 1–3 (curves denoted by 1). After plastic deformation followed by slow cooling (30 min), the dislocation density in the samples became $N_D = 2 \times 10^8$ cm^{-2} and the n-type samples were converted to p-type conduction provided $n \leq 5 \times 10^{14}$ cm^{-3}. All the electrons from the conduction band were captured by deep centers and the hole density p in the valence band was governed by the concentration of deep acceptors N_{dc}, which was comparable with N_d and N_a (curve 2 in Fig. 1).

Since all the possible complexes which could be formed by vacancies and impurity atoms in the course of plastic deformation and during cooling were stable in silicon up to temperatures not exceeding 600°C, heating to $T_{ann} = T_{def}$ = 700°C could ensure their full dissociation in a deformed sample. If, without waiting for the liberated vacancies to be annihilated as a result of condensation at sinks so that the bulk concentration of vacancies would reach the equilibrium value (at T_{ann} = 700°C), a new state was "frozen" after annealing for 1 min by quenching in glycol, the crystals recovered their n-type conduction and the electron density in the conduction band was found to be governed by the ionization of centers whose energy was $E_c - 0.44$ eV, as demonstrated by curve 3 in Fig. 1.

A gradual increase in the annealing time at T = 700°C revealed that there were stages during which more stable centers with an ionization energy $E_c - 0.2$ eV predominated (curve 4) and then—after annealing for t $\geq$ 37 min—a quasiequilibrium was established in which the carrier density in the conduction band was again governed by shallow donors (curve 6). The presence of dislocations in the bulk of a crystal was now manifested only by a reduction in the carrier density in the conduction band by an amount $\Delta n = n_{init} - n_{def}$ as a result of the transfer of carriers to acceptor levels with $\Delta E \geq 0.44$ eV and the appearance of an additional scattering mechanism.

The additional scattering of carriers in the T $\geq$ 300°K range was proportional to the dislocation density, depended weakly on temperature, and was similar to scattering by cylindrical space-charge regions surrounded by dislocations, considered theoretically by Read [10, 11] (curves 1–3 in Fig. 2). Moreover, an anomalously strong scattering of carriers was observed and this had a maximum at T = 200°K ($N_D = 5 \times 10^7$ cm^{-2}, curve 2) and a minimum at T = 250°K ($N_D = 2.2 \times 10^8$ cm^{-2}, curve 3).

The nature of the electrical activity of dislocations and the role of "broken" chemical bonds, whose concentration was governed by the total length of the dislocations in 1 cm^3 and could not change with time at room temperature, were determined by investigating the influence of ageing which could alter the point-defect atmosphere surrounding the dislocations and could facilitate vacancy migration. We used samples from which all the complexes were removed by annealing at T = 700°C, as shown in Fig. 1 (curve 6). The state represented by curve 6 could be obtained by single annealing at T = 700°C for 50–60 min, followed by rapid cooling.

Natural ageing at T = 20°C for 27 days resulted in a change from the state represented by curve 6 in Fig. 1 ("state 6") back to "state 4." In the latter state, we again observed the 0.2 eV levels (in addition to the levels at 0.44 eV) and the total density of these levels did not exceed the initial density of electrons in the conduction band. Further ageing for 65 days produced "state 3" of Fig. 1, in which electrons from the conduction band and those from the 0.2 eV levels were transferred to the newly formed deeper levels at 0.44 eV. There was a parallel change in the dependence $\mu_n(T)$. As long as the samples were in the state 6 (curve 6 in Fig. 1), the minimum of $\mu_n(T)$ was located at T = 100°K. When the filling of the 0.44 eV levels was accompanied by the filling of the levels at 0.2 eV (not shown in Figs. 1 and 2), the dependences $\mu_n(T)$ had two minima: one at T = 100°K (shallower) and the other at T = 250°K. Finally, when deep centers of the 0.44 eV type predominated (curve 3 in Fig. 1), the minimum of $\mu_n(T)$ was located at T = 250°K.

In the case of the samples in which the high-temperature state after the end of deformation was "frozen" by cooling in 30 sec and the vacancies could not reach the sinks (dislocations) in the available time, we found that complexes were formed (the majority of excess vacancies was removed when the temperature reached the range of stability of the A centers, which was ~300°C) and the temperature dependences of n were very similar to curve 6 in Fig. 1; a minimum was probably exhibited by the $\mu_n(T)$ dependences but it was outside the investigated range of temperatures because μ_n decreased right down to T = 77°K. According to our estimates, this state corresponded to the minimum radius of the point-defect atmospheres at dislocations.

Isochronous annealing of the deformed samples was carried out in the range T = 70–550°C. The dependence of μ_n(77°K) on T_{ann} obtained for the crystals grown by the Czochralski method, i.e., with the oxygen concentration $N^O = 4 \times 10^{17}$ cm^{-3}, was similar to the dependences exhibited by the zone-grown crystals (with $N^O < 10^{16}$ cm^{-3}) but the relative changes in μ_n(77°K) were greater in the crystals with the higher oxygen concentration. When the annealing temperature passed through T_{ann} = 150, 250, and 350°C—which were the characteristic temperatures of the dissociation of the E centers, divacancies, and A centers—the dependences μ_n(77°K) on T_{ann} exhibited inflections. This was one of the proofs that the dislocation atmospheres contained the complexes mentioned above.

If the complexes formed in the dislocation atmospheres were indeed A or E centers and divacancies, and if, being bound by the electrostatic interaction, they were responsible for the anomalous scattering of carriers, we could use monochromatic illumination to influence the depth of the minima of the dependences $\mu_n(T)$ by charging a negligible proportion of such centers.

In fact, we found that the regions of the maximum influence of external illumination on the dependences $\mu_n(T)$ corresponded to the photon energies hν = 1.15, 0.4, and 0.17 eV.

Figure 2 gives not only the dependence $\mu_n(T)$ obtained in darkness for the initial crystal but also the dark dependences $\mu_n(T)$ of deformed samples with $N_D = 5 \times 10^7$ and 2.2×10^8 cm^{-2}. The excitation of electrons to the conduction band from the ~0.4 eV levels reduced the depth of the minimum and shifted it slightly toward lower temperatures (curve 4); the excitation of electrons from the ~0.2 eV levels resulted in an even stronger reduction in the depth of the minimum (curve 5) and shifted it toward lower temperatures. The interband excitation (hν = 1.15 eV) had somewhat less effect than the excitation of 0.2 eV levels (curve 6, compared with curve 5 in Fig. 2).

The photon energy hν corresponded to the ionization energies of the predominant centers, which were deduced from the slopes of curves 3 and 4 in Fig. 1. The injection rate was estimated from the change in the carrier density on the basis of the dependences n(1/T) obtained in darkness or in the presence of illumination, and it did not exceed 1%, i.e., $\Delta n \sim 10^{10}$ cm^{-3}. We also found (Fig. 2) that illumination influenced the nature of the dependence $\mu_n(T)$ only in the region of the minimum but not in the range where carriers were scattered by phonons T = 300–450°K.

For comparison, we included in Fig. 3 the temperature dependences of μ_n obtained from samples which were first irradiated with 24 MeV protons (10^{11} cm^{-2} dose) and then with 2.2 MeV electrons (10^{14} cm^{-2} dose). A preliminary proton irradiation increased the proportion of the electrostatically interacting divacancies (V_2), A and E centers because it produced primary defects located along tracks and capable of condensing to form closely spaced vacancy complexes and clusters. An anomalous scattering of carriers with a minimum at T = 100°K was observed in such samples.

The excitation of electrons from the E centers (hν = 0.4 eV for the same injection rate Δn = 1%) resulted in a considerable change in the depth of the minimum and shifted it toward higher temperatures (T = 115°K, curves 2 and 4 in Fig. 3). Clearly, illumination with hν = 0.4 eV photons maintained the influence of the unexcited deeper centers, such as divacancies with levels at 0.54 eV, whose concentration was $N_{V_2} < N_E$. The interband excitation (hν = 1.15 eV) shifted the minimum to T = 95°K and had a somewhat smaller influence on its depth. A complete suppression of the anomalous scattering of carriers could be achieved by increasing the illumination intensity but this was not possible when the IKS-21 monochromator was used.

DISCUSSION OF RESULTS

The well-known radiation defects in silicon, such as E centers (vacancy + phosphorus atom, ΔE = 0.4 eV), divacancies (ΔE = 0.54 eV), and A centers (vacancy + oxygen atom, ΔE = 0.17 eV) are stable to annealing temperatures T_{ann} = 150, 250, and 300°C, respectively. Vacancies liberated as a result of dissociation of complexes condense at sinks or, if the annealing temperature is in the range $150°C < T_{ann} < 300°C$, they form more stable defects such as a vacancy joined to two oxygen atoms, but even they are stable only to temperatures not exceeding ~600°C. This is why the annealing temperature ($T = 700°C$) and duration (~50 min) selected in our study are sufficient to ensure full dissociation of all complexes in the bulk of a crystal and to establish an equilibrium vacancy concentration (the concentration of complexes in the bulk after irradiation or deformation followed by slow cooling is always higher than the equilibrium vacancy concentration). After the removal of complexes from the bulk of a crystal, the presence of dislocations is manifested by the change in the carrier density $\Delta n = n_{init} - n_{def}$ (curves 1 and 6 in Fig. 1).

If, following Read, [11] we assume that electrons from the conduction band are captured by "broken" bonds and if we define the degree of occupancy of these bonds f_c by

$$f_c = (N_d - N_a - n)\, c/N_D, \quad (1)$$

where $N_d - N_a$ is the concentration of the uncompensated donors, n is the density of electrons in the conduction band after deformation and annealing, c is the distance between the "broken" bonds at the edge of the extra atomic half-plane, and N_D is the dislocation density, we obtain a reasonable value $f_c \approx 0.032$ (if we use our data represented by curves 1 and 6 in Fig. 1). However, since the occupancy f_c cannot vary with time and electrons continue to leave the conduction band in the course of ageing of a sample, the concentration of centers in the dislocation atmospheres should vary with time. The concentrations of the centers with the levels at 0.2 and 0.44 eV increase so much that, after 65 days of storage at $T = 20°C$, the samples return to the state 3 of Fig. 1 and electrons then leave both the conduction band and the 0.2 eV levels and are transferred directly to the deeper levels at 0.2 eV whose density now exceeds the initial electron density. This is why we are assuming that the point-defect atmospheres at dislocations are in continuous motion and contain vacancies and complexes whose concentrations are comparable with or exceed the number of "broken" chemical bonds $N_V + N_c \geq N_D/c$.

Vacancies are capable of migrating within an atmosphere and they can condense into divacancies or may be captured by impurity atoms forming complexes such as the A and E centers. The vacancy migration energy ΔU_m is governed by the vacancy charge and, in the case of a vacancy V^{2-}, it amounts to ~0.18 eV, whereas, for V^- and V^0, it is ~0.33 eV. [12] These energies are comparable with the ionization energies of electrons captured by deep centers and may be acquired from recombination between electrons and holes. It is known [12] that charged vacancies are mobile in silicon crystals at 77°K and even at 4.2°K. They are also mobile at 20° C and, therefore, the dislocation atmospheres become modified as a result of the formation of the A and E centers and of divacancies. Vacancies seem to "evaporate" from a dislocation atmosphere and its radius increases since, in the case of a more or less homogeneous distribution of phosphorus atoms ($N^P \sim 10^{14}\ cm^{-3}$) and oxygen atoms ($N^O \sim 10^{16}\ cm^{-3}$), this is the only way that complexes can be formed. Estimates show that the radius of an atmosphere then changes from 0.25 to 0.75 μ, i.e., the impurity atoms located close to the core of a dislocation may interact with the vacancies.

We can prove that a dislocation atmosphere consists of the A and E centers and of divacancies by considering the energy of ionization of deep centers in deformed samples (curves 3–5 in Fig. 1). The depth of the levels is only slightly affected by the electrostatic interaction. According to the Read theory, [11] the energy of the electrostatic interaction of electrons in "broken" bonds forming a chain depends on the occupancy of these bonds and can be represented in the form

$$\mathscr{E}_s(f) \approx f_c \mathscr{E}_0 [^3/_2 \ln (f/f_c) - 0.866], \quad (2)$$

where $\mathscr{E}_0 = e^2/\epsilon c \sim 0.5$ eV for silicon, f is the Fermi function, $f_c = c[\pi(N_d - N_a - n)]^{1/2}$, e is the electronic charge, ϵ is the permittivity, and the other symbols have the same meaning as before.

If c (the distance between broken bonds) is comparable with c_c (the distance between complexes in a dislocation atmosphere) we can ignore the second term in the brackets in Eq. (2) and apply this expression to an atmosphere of complexes, which gives $\mathscr{E}_S \sim 0.03$ eV, in good agreement with the observed change in the depth of the 0.4 eV level of the E centers. However, since the occupancy of deep centers in an atmosphere (f'_c) tends to unity (in the case of broken bonds, we have $f_c \to 0.1$), the dislocation atmosphere itself must change and the interaction energy may reach a value $\mathscr{E}_S \sim f'_c \mathscr{E}_0$, which is comparable with the dissociation energy of complexes and with the vacancy migration energy.

In the range $T = 300-450°C$, the complexes in a dislocation atmosphere are partly ionized and the scattering of carriers by cylindrical space-charge regions surrounding dislocations is proportional to N_D and agrees satisfactorily with the conclusions reached by Read [11] and Matare, [13] according to whom the ratio $\mu_n(N_D)/\mu_n$ is ~0.3 for $N_D = 2 \times 10^8\ cm^{-2}$ and ~0.5 for $N_D = 5 \times 10^7\ cm^{-2}$.

The minimum in the dependences $\mu_n(T)$ can be explained bearing in mind the possible trapping of conduction electrons by electrostatically interacting centers in dislocation atmospheres or in irradiated samples. These centers are responsible for the anomalous scattering of carriers because an electron trapped for a time $\tau(T)$ is again released to the conduction band, which reduces the mean free time: $t = t_0 - \tau(T)$. The time τ of the resonance of an electron at a center increases when the temperature of the crystal T is lowered and lies within the range $0 < \tau(T) \geq t_0$; in the limit $\tau(T) \to \infty$, an electron remains trapped, i.e., it ceases to participate in the conduction process. Continuous illumination with photons of a certain energy $h\nu$ transforms the centers from the state in which they give rise to the anomalous carrier scattering, i.e., it reduces the time $\tau(T)$ during which the carrier resides in a localized state before being released to the conduction band.

Under trapping conditions, the mean free time t decreases in accordance with the expression

$$t \approx t_0 [1 - \tau(T)(1 - f_{f'_{ci}})/t_0], \quad (3)$$

where $t_0 = l_0/v$, l_0 is the mean free path, v is the thermal velocity of electrons, and f'_{ci} is the occupancy of complexes of a given type. Since $\mu_n \propto (e/m)T^{-2.6}t$, where m is the effective mass of an electron and t is given by Eq. (3), it follows that, in the temperature range in which the centers become filled with electrons, the mobility μ_n decreases, passes through a minimum, and then begins to rise again with increasing degree of occupancy, when f'_{ci} tends to unity. The temperature corresponding to this minimum is a function of the type of predominant center and of its ionization energy and of the concentration of the noninteracting centers in the bulk of the crystal because the trapping effect is not manifested until completion of the filling of the centers which do not interact electrostatically.

A change in the position of the minimum along the T axis as a result of illumination is clearly due to the fact that, in dislocation atmospheres present in deformed and irradiated crystals, the electrostatic interaction affects deeper (divacancies and E centers) and shallower (A centers) complexes. The filling of centers of each type with electrons from the conduction band then begins and the value of f'_{ci} reaches a value close to unity at some specific temperature and this is the temperature of the minimum in the dependence $\mu_n(T)$ for centers of this type. Therefore, the $\mu_n(T)$ curves may have not one but two minima and, if the temperature is extended toward lower values or a different set of charge centers is present, additional minima may also be observed.

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