Investigation of dislocations in silicon by the photo-ESR method

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An investigation was made of the influence of monochromatic light on the ESR spectrum of dislocations in samples of plastically deformed silicon. It was found that illumination increased the number of paramagnetic centers and improved the resolution of the ESR spectrum. The spectral dependence of the effect indicated the occurrence of transitions at energies of $+0.38$ eV and $+0.6$ eV. The effect became saturated at high illumination intensities. An increase in the area under the ESR signal was 3–6% under saturation conditions. A study was made of the kinetics of the recovery of the spectrum observed in darkness. The results were explained by a model of a dislocation energy spectrum of electrons.

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INTRODUCTION

Investigations of the positions of electron levels that appear in the band gaps of semiconductors as a result of plastic deformation have been usually made on the basis of measurements of electrical properties or photoconductivity.[1-12] On the other hand, using the ESR method, we have been able to show that introduction of dislocations in silicon crystals produces paramagnetic centers whose number is equal to the number of broken bonds in dislocation cores.[13-19] However, it has not yet been finally established whether the electron levels associated with broken dislocation bonds and responsible for the ESR signal are the same as those manifested in electrical measurements. This aspect is of fundamental importance in the model of the energy spectrum of electrons at dislocations.

Further information on this question was obtained by us in an investigation of the influence of monochromatic illumination altering the populations of the dislocation energy levels on the ESR signal of broken dislocation bonds in silicon.

SAMPLES AND EXPERIMENTAL METHOD

We used silicon single crystals grown by the floating zone method in vacuum. Our crystals were doped with 2×10^{13} cm^{-3} of boron and before deformation they exhibited p-type conduction with a resistivity of 3×10^{3} Ω·cm at 300°K. Our samples were parallelepipeds with edges along the $\langle 112\rangle$, $\langle 111\rangle$, and $\langle 110\rangle$ directions, and their dimensions were $3.5\times4\times9$ mm. Dislocations were introduced by plastic deformation resulting from compression by a constant load along the $\langle 110\rangle$ axis at 690°C in an argon atmosphere. The degree of deformation (strain) was 3 – 5%. After deformation a layer 1 mm thick was removed from the ends, and then the samples were subjected to mechanical and chemical polishing. Details of the preparation methods were given earlier.[5,13] Samples were similar to those used in Refs. 5 and 6. The dislocation density found by electron-microscopic examination amounted to $(3-5)\times10^{9}$ cm^{-2} and the concentration of broken dislocation bonds, deduced by the ESR method, was $(3-6)\times10^{16}$ cm^{-3}. The concentration of broken bonds was in good agreement with that calculated on the basis of geometric considerations from the dislocation density. Changes in the deformation conditions, particularly an increase in the temperature and duration of deformation, reduced the ESR signal due to dislocations. This probably accounted for the fact that in samples used in Refs. 19 and 20 the concentration of the ESR centers was only a few percent of that calculated on the basis of geometric considerations, so that the ESR signal observed in Refs. 19 and 20 was associated with point defects and its value was of the order of a dislocation signal. Investigation of such samples was outside the scope of the present study because we were interested primarily in the properties of a one-dimensional dislocation system of broken bonds in its "purest" form.

A sample was placed at the center of a cylindrical cavity resonator and cooled to the required temperature by a stream of gaseous nitrogen. Temperature was varied within the range 300 – 110°K and was maintained to within at least 1.0°K. The working frequency of an ESR spectrometer was 9.3 GHz (x range). Since at the selected temperatures the spin relaxation times τ_1 and τ_2 of broken dislocation bonds in Si were less than 10^{-6} sec ($\tau_1, \tau_2 < 10^{-6}$ sec), in accordance with Ref. 16, we modulated the magnetic field at a frequency of 100 kHz and employed the slow passage regime. The modulation amplitude was 0.2 Oe and the microwave power was 0.5 mW. A sample was illuminated through a CaF_2 window by means of a monochromator with an LiF prism. The source of light was a Globar. The absorption of light in water vapor at the wavelength of 2.8 μ was reduced by blowing dry nitrogen continuously through the optical channel. This channel included a mechanical chopper controlled by a spectrum storage unit. The ESR signal was stored in this unit and analyzed there (the operations included subtraction, integration, accumulation, etc.).

EXPERIMENTAL RESULTS

The ESR spectra observed for our samples were practically unaffected by temperature variations in the range 300 – 80°K and were qualitatively similar to the spectra obtained in Refs. 13 – 15 and 19 at $T < 60$°K.

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FIG. 1. ESR spectra showing the signal $\partial\chi''/\partial H(H)$ due to dislocations in darkness (1) and the same signal in the presence of illumination (2). $T = 170°K$, $H_0 \parallel \langle 111 \rangle$.

When a sample was illuminated at temperatures in the range $T < 260°K$ there was a considerable change in the form of the spectrum. Figure 1 shows a record of the ESR signal $\partial\chi''/\partial H(H)$ before and during illumination. Figure 2 shows the spectra of $\chi''(H)$ before and during illumination obtained by integration of the $\partial\chi''/\partial H$ spectra (see Fig. 1). The lower curve in Fig. 2 gives the difference $\Delta\chi'' = \chi_0'' - \chi_d''$, i.e., the change in the $\chi''(H)$ spectrum as a result of illumination. On the right, we can see the fourth line of a standard source (Mn^{2+} in MgO). This standard source was in thermal contact with a sample and was located in the same place in the resonator; it was used to monitor the sensitivity in the operation of subtraction of the spectra. It should be noted that the form of the spectrum $\Delta\chi''(H)$ observed in the range 270 – 110°K was independent of temperature and illumination intensity. Neither the form of the spectrum nor its amplitude

$$\Delta\chi''(H) \Big/ \int_{H_0-30\,\text{Oe}}^{H_0+30\,\text{Oe}} \chi_0''\,dH$$

were affected by reduction in the microwave power by 10 dB (from 1 to 0.1 mW), indicating the absence of influence of saturation.

Figure 3 shows the dependences of the signal $\Delta\chi''$ on the power of light of the 1.3 μ wavelength (selected with a filter which had a transmission maximum at 1.3 μ). The effect of illumination showed saturation at high intensities and the optical power from which saturation began fell rapidly as temperature was lowered. The intensity of light p from which saturation began was approximately 500 times less at $T = 120°K$ than at $T = 200°K$. At $T < 120°K$ this intensity became so low that special measures had to be taken to avoid the influence of scattered light.

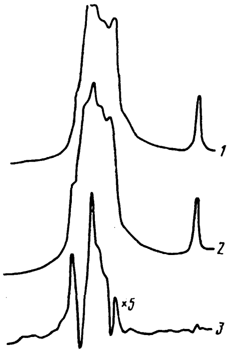

FIG. 2. ESR spectra showing $\chi''(H) = \int(\partial\chi''/\partial H)dH$ in darkness (1) and during illumination (2). Spectrum 3 is the difference ($\Delta\chi''$) between spectra 2 and 1.

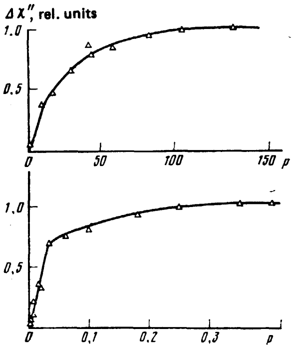

FIG. 3. Dependence of the change in the ESR signal on the optical power p; $\lambda = 1.3\ \mu$; the upper curve was recorded at $T = 200°K$ and the lower at $T = 120°K$.

The quantity

$$f' = \int_{H_0-30\,\text{Oe}}^{H_0+30\,\text{Oe}} \Delta\chi''(H)\,dH \Big/ \int_{H_0-30\,\text{Oe}}^{H_0+30\,\text{Oe}} \chi_0''(H)\,dH,$$

representing the relative change in the concentration of paramagnetic centers as a result of illumination amounted to +3–6% under saturation conditions and did not vary greatly from sample to sample. The sign of this quantity always indicated an increase in the number of paramagnetic centers as a result of illumination.

Figure 4 shows the kinetics of recovery of the dark ESR spectrum after the end of illumination. Fast scanning of the magnetic field was used and the spectra $(\partial\chi''/\partial H)_t$ were recorded after a time t from the end of illumination. Next, a calculation was made of the quantity $\Delta(t)$ representing the peak-to-peak amplitude of the difference spectrum $(\partial\chi''/\partial H)_t - (\partial\chi''/\partial H)_\infty$, where $(\partial\chi''/\partial H)_\infty$ was the steady-state spectrum in darkness. The process of recovery of the dark spectrum could not be described by a single exponential function (Fig. 4). We attempted to describe it by a sum of two exponential functions, i.e., by

$$\Delta(t) = (\Delta_0 - \Delta_s)\exp(-t/\tau_f) + \Delta_s \exp(-t/\tau_s), \tag{1}$$

where $\Delta_0 \equiv \Delta(0)$; Δ_s is a temperature-dependent parameter. Since the values of τ_f and τ_s differed by almost two orders of magnitude, it was quite easy to separate the fast decay stage $\Delta(t)$ from the very slow tail. Fig-

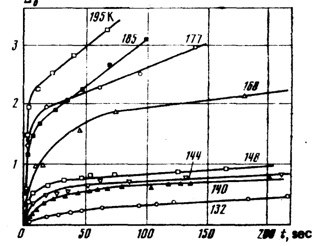

FIG. 4. Recovery of the dark ESR signal after the end of illumination: Δ_0 is the effect during illumination and Δ_t is the effect after a time t from the end of illumination.

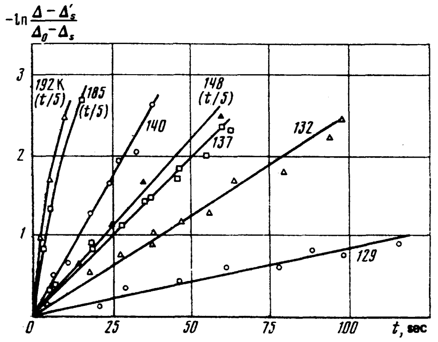

FIG. 5. Fast component of the recovery of the dark ESR spectrum (τ_f).

ure 5 shows the dependences of $\ln[(\Delta(t)-\Delta_s')/(\Delta_0-\Delta_s)]$ on t recorded at several temperatures; Δ_s was in this case deduced from the curves of the type shown in Fig. 4 by extrapolation of the slow stage to $t=0$ using $\Delta_s'=\Delta_s\exp(-t/\tau_s)\approx\Delta_s$, and τ_s was estimated from the slope of the curves $\ln[\Delta(t)/\Delta_0]$ (Fig. 4) at high values of t. Figure 6 shows the dependences of $\ln[(\Delta(t)-\Delta')/\Delta_s]$ on t, where $\Delta'=(\Delta_0-\Delta_s)$. It is clear from Figs. 5 and 6 that the experimental points fit well straight lines, indicating that Eq. (1) is obeyed. The slopes of these straight lines give τ_f and τ_s, respectively.

Figure 7 shows our dependences of $\log\tau_f$, $\log\tau_s$, and $\log[\Delta_s/(\Delta_0-\Delta_s)]$ on the reciprocal of temperature.

Figure 8 gives the spectral dependence of the effect. The ordinate represents, on a logarithmic scale, the quantity $\Delta_0=(\partial\chi''/\partial H)_0-(\partial\chi''/\partial H)_\infty$, i.e., the amplitude of the change in the spectrum $\partial\chi''/\partial H$ due to illumination divided by the number of photons N incident on a sample per unit time. The photon flux was calculated from the optical power transmitted by the monochromator and measured with a thermopile. The illumination intensity was selected so as to operate in the linear part of the dependence of Δ_0 on the illumination intensity (Fig. 3). The sensitivity threshold of the method is identified by a dashed curve. The upward bending of this curve at low energies ($h\nu<0.35$ eV) is due to a reduction in the optical power resulting from the spectral characteristic of the light source and the optical system employed. In the range $h\nu\lesssim 0.27$ eV the experimental points do not lie above the sensitivity threshold so that, within the limits of the experimental error, we cannot separate the photo-ESR signal from the noise. Throughout the spectral range where the effect was observed the form of the spectrum of $\Delta\chi''(H)$ was constant. In view of the long relaxation times, we heated the sample after each point in order to obtain the dark spectrum.

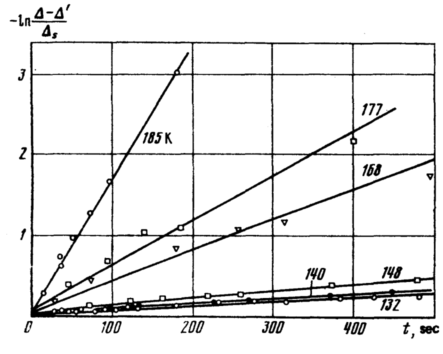

FIG. 6. Slow component of the recovery of the dark ESR spectrum: $\Delta'=(\Delta_0-\Delta_s)\exp(-t/\tau_f)$.

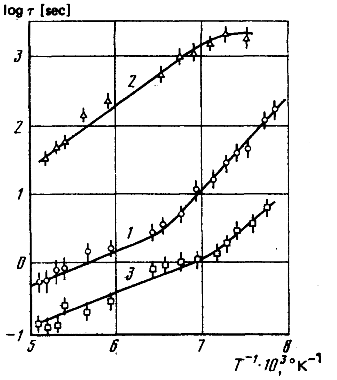

FIG. 7. Dependences of $\log\tau_f$ (1), $\log\tau_s$ (2), and $\log[\Delta_s/(\Delta_0-\Delta_s)]$ (3) on the reciprocal of temperature.

DISCUSSION OF RESULTS

The main experimental result in which—in our opinion—requires explanation is the increase in the intensity of the dislocation-induced ESR signal as a result of illumination. As pointed our earlier (Figs. 1 and 2) this increase amounts, under saturation conditions, to 3 – 5% of the initial integrated intensity.

Following the conclusions of earlier investigations,[13-19] we shall assume that the investigated ESR spectra represent broken bonds in dislocation cores. The number of such bonds, both deduced from the ESR spectra and calculated from the experimental data on the dislocation density, is $(3-6)\times10^{16}$ cm^{-3}. Consequently, illumination increases the number of broken bonds by $(1-3)\times10^{15}$ cm^{-3}.

At first sight it might seem that the simplest explanation is as follows: electrons initially captured from broken dislocation bonds by the boron acceptor levels and leaving behind a certain number of holes are excited optically back to the broken bonds and this increases the number of uncompensated spins, manifested as an increase in the ESR signal. However, it is clear that since the total concentration of the boron acceptor impurities is only 2×10^{13} cm^{-3} and the increase in the number of these centers responsible for the ESR signal

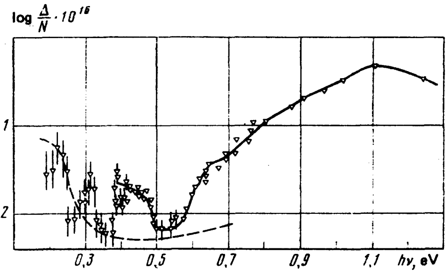

FIG. 8. Dependence of $\log(\Delta/N)$ on the photon energy $h\nu$: Δ is the change in the ESR signal due to illumination and N is the flux of the incident photons. $T=130$°K.

is by ~2×10^{15} cm^{-3}, this process cannot be responsible for the experimentally observed illumination-induced increase in the intensity of the ESR signal representing broken dislocation bonds. Consequently, we shall assume that the acceptor centers from which electrons are transferred by illumination to dislocation levels in amounts of at least 2×10^{15} cm^{-3} are formed, like the dislocation levels themselves, directly as a result of plastic deformation. A self-consistent explanation of the increase in the photo-ESR signal can be provided if the acceptor levels are included in the general scheme of the dislocation energy spectrum of electrons.

The main experimental data needed to construct such an energy scheme and the model representations are already largely available in our previous papers reporting investigations of electrical properties of plastically deformed silicon and germanium.[1,2,5,6,9-12] We shall now recall the main conclusions reached there. Investigations of the Hall effect and conductivity of silicon containing dislocations, carried out under dc conditions and in the microwave range, have led to the conclusion that the results can be accounted for by a two-band model of the dislocation spectrum of electrons which follows from the model of dislocations regarded as a Mott–Hubbard insulator.[5-6,21] The position of the first acceptor level (band) in silicon is 0.42 ± 0.03 eV below the conduction band, whereas the lower donor level (band) is 0.43 ± 0.03 eV above the valence band.

It follows that the Hubbard gap between these narrow levels or bands is 0.25 eV in the case of silicon.[5,6] It is also clear from these data that a certain number of acceptor levels, estimated as 0.03 – 0.05 of the total number of donor dislocation states,[5,6] adjoins the lower donor band, i.e., it is separated by no more than 0.03 eV from this band. A study of the microwave conductivity associated with the dislocation spectrum of electrons in germanium has also led us to a three-band model and the mobility of carriers in the upper acceptor and lower donor bands has been sufficient to observe significant microwave dislocation conductivity, whereas the conductivity in the middle acceptor band has been found to be practically negligible.[11] Numerical data on the positions of the donor and acceptor dislocation bands of germanium have also been obtained from the Hall effect measurements.[2,12] The dislocation electron spectrum deduced from the above electrical measurements is shown in Fig. 9.

The same electron spectrum is proposed for silicon in Refs. 6 and 21. However, it fails to account for the main result of the present study, namely for the increase in the intensity of the dislocation ESR spectrum as a result of illumination, since the acceptor states E_1' lie in this scheme above the state E_1 responsible for the dislocation ESR signal. We can explain all the results obtained in investigations of electrical properties of crystals with dislocations and the ESR due to dislocations by a more general dislocation energy spectrum of elemental semiconductors which is given below and which represents an extension of the scheme proposed earlier. Before considering the actual structure of the scheme, we shall make some assumptions in the model of dislocation chains.

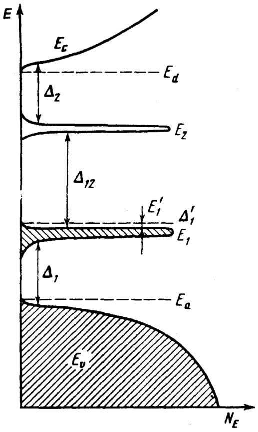

FIG. 9. Preliminary energy-level scheme of silicon: $\Delta_1 = 0.42$, $\Delta_2 = 0.43$, $\Delta_{12} = 0.25$, $\Delta_1' \leq 0.03$ eV; in the case of germanium the corresponding values are $\Delta_1 = 0.07$, $\Delta_2 = 0.5$, $\Delta_{12} = 0.18$, and $\Delta_1' \leq 0.05$ eV.

As before, we shall assume that electrons on broken bonds of atoms belonging to a dislocation core form a narrow one-dimensional donor band whose width depends on the degree of overlap of the wave functions of the broken-bond electrons along a dislocation chain. These broken dislocation bonds may capture excess electrons which are present in a crystal and are associated either with chemical donors or other defects or are excited externally by incident light or a strong electric field. These excess electrons may be captured by the dislocation acceptor levels forming a narrow acceptor dislocation band. We shall assume, as before,[5,6] that the Coulomb interaction between two electrons at one site gives rise to a gap between these two one-dimensional bands and the presence of this gap means that a dislocation chain with broken bonds can be described as a Mott–Hubbard insulator.

Investigations of the structure of dislocations in real crystals have shown that the length of regular parts of a dislocation line are sufficiently short (amounting to a few tens or hundreds of interatomic distances) and the regular parts of dislocations alternate with local defects, such as jogs, points of intersection with other dislocations, etc. Moreover, we can expect defects in the form of impurity (for example, oxygen) atoms located near a dislocation core. Such local defects near or on dislocation lines may give rise to additional electron acceptor and donor states. The number of such states is related to the number of local defects. We shall assume that local acceptor and donor levels lie below the main dislocation donor band. Then, in the case of a pure (free of shallow impurities) semiconductor with dislocations some of the electrons are transferred from the broken bonds to acceptor levels of these local defects and form holes in the dislocation donor band. These holes are mobile and they should move toward electrons captured by local defects because of the Coulomb attraction, and they should remain near these defects ceasing to be mobile.

Since the gap between E_1 and E_1' does not exceed 0.05

eV (Refs. 5 and 6), the average distance between a hole at E_1' and an electron at ε_2 should be of the order of 5 lattice constants. Such a dislocation "exciton" may be stable because of, for example, an energy barrier at a dislocation or because of spatial separation if a local defect does not lie quite inside a dislocation core. Thus, a narrow band of holes interacting by the Coulomb force with electrons captured at acceptor levels of local defects at dislocations splits off from the main dislocation donor band. Clearly, these holes are characterized by a low mobility, in contrast to other holes in the donor dislocation band formed as a result of capture of electrons from the broken dislocation bonds by chemical acceptors. We thus obtain the energy band scheme of the dislocation energy spectrum shown in Fig. 10. The abscissa gives the density of states at a given energy. The difference between this scheme and the one discussed earlier (Fig. 9) is the presence, below the E_1 band, of acceptor ε_2 and donor ε_1 states associated with local defects at dislocation lines. In the case of an undoped semiconductor or in the presence of a small number of acceptor or donor impurities both ε_1 and ε_2 bands are filled in the dark state. We shall now relate the proposed energy-level scheme to the spectral dependence of the photo-ESR signal and to the kinetics of relaxation of the spectrum after the end of illumination.

The step observed at $h\nu = 0.38\,\text{eV}$ (Fig. 8) represents the excitation of electrons from the valence to the E_1' band, whereas the step at $h\nu = 0.58-0.60\,\text{eV}$ represents the $E_v \to E_2$ transition followed by the transfer of an electron from E_2 to E_1'. These processes reduce the hole occupancy of the dislocations and increase the ESR signal. The value of $E_1 - E_v$ obtained from the Hall data in Ref. 5 is overestimated by $0.04-0.05\,\text{eV}$ and this may be due to modulation of E_v by the deformation potential so that the percolation level for holes in the valence band lies below the optical edge E_v. Recovery of the spectrum after the end of illumination takes a longer time (Figs. 3 and 4).

We can account for the observed long relaxation time by assuming that holes appearing in the valence band are captured by deep centers different from the usual dislocation broken bonds and that electrons captured by these broken bonds cannot recombine with such holes. In our scheme these deep centers are identified as ε_2 and they are associated with local defects at dislocations whose concentration is, as pointed out above, $(0.03-0.06)N_D$ and which are filled with electrons from E_1 under equilibrium conditions. The low-temperature part of curve 1 in Fig. 6 is described by the law $\tau_f^{-1} \approx 10^8 \exp(-0.25\,\text{eV}/kT)\,\text{sec}^{-1}$, which resembles strongly the law describing thermally activated release of holes from a level of depth $\approx 0.25\,\text{eV}$. It should be noted that formation of acceptor levels with an energy $E_v + 0.27\,\text{eV}$ in silicon crystals deformed at high temperatures is reported in Refs. 7 and 8. We can assume that these are the centers labeled as ε_2. At temperatures $T > 150\,^\circ\text{K}$ the slope of curve 1 decreases and this may be due to limitations imposed on the rate of recombination by the transfer of holes from the valence band to the dislocation band E_1, and not by the activation of holes from the ε_2 levels. The slow component τ_s of the relaxation process is characterized by an activation energy of 0.18 eV, as indicated by the data represented by curve 2.

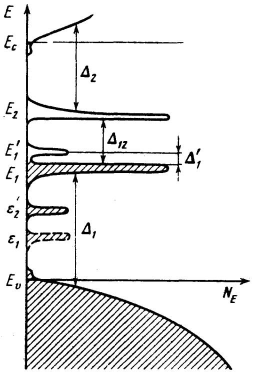

FIG. 10. Energy level scheme of the electron spectrum of dislocations.

We shall now consider the improvement in the resolution of the structure of the ESR spectrum as a result of illumination. The proposed model allows us to interpret this effect as follows. In the absence of illumination the holes in the E_1' band and the negatively charged ε_2 acceptors create strong inhomogeneous electric fields in a dislocation core and these fields should result in additional inhomogeneous broadening of the spectral lines as well as in a shift due to the deformation of the wave functions of the broken bonds and consequent changes in the g tensors. The line shift is governed by the average value of the field and the orientation of the dislocations giving rise to a specific ESR line relative to an external magnetic field. Illumination reduces these inhomogeneous Coulomb fields and this should result in narrowing and shift of the lines.

In general, there are other mechanisms which can give rise to a strong photo-ESR signal. For example, it is suggested in Ref. 17 that some of the broken dislocation bonds become paired to form a state $s = 0$ as a result of exchange interactions, so that the ESR signal is less than the concentration of the broken bonds calculated on the basis of geometric considerations. Excitation of electrons to the band E_2 may result in dissociation of such pairs and it may increase the ESR signal. Secondly, experiments of this kind may result in formation of magnetic polarons at dislocations when an electron captured by a broken bond creates a ferromagnetic region because of exchange interactions and each such region contains several nearest broken bonds.[18] An ESR signal due to such a ferromagnetic region may lie within the observed ESR spectrum of dislocations, since in this case we can clearly ignore the influence of the demagnetization factor, whereas the influence of the crystal field and other factors responsible for inhomogeneous broadening and splitting can only decrease as a result of exchange interactions. However, these mechanisms are suggested only tentatively.

We are planning to carry out a more detailed investigation of the kinetics of the photo-ESR effect and to compare it with the kinetics of the microwave photoconductivity and also dc conductivity, and to study the spectral dependence of the effect exhibited by samples differing in respect of doping.

We shall conclude by pointing out that Erdmann and Alexander[22] recently observed also the influence of light on the ESR in deformed silicon crystals. However, the results given in Ref. 22 differ considerably from those presented above. In particular, the ESR spectrum of dislocations in pure samples was found to be weakened severalfold by illumination without a change in the form of the spectrum. Moreover, the spectral dependence found by Erdmann and Alexander also differs considerably from that given in Fig. 8, although the characteristic energy of the red edge of the photo-ESR is still 0.57 eV, in agreement with the second characteristic energy found in the present study. Clearly, the differences are due to the very different structure of dislocation cores, which is indicated by the fact that even at a dislocation density of $2\times10^9\,\mathrm{cm}^{-2}$ Erdmann and Alexander[22] observed an ESR signal corresponding to just $6\times10^{14}\,\mathrm{cm}^{-3}$ of spins, which is two orders of magnitude less than that observed by us.

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