ELECTRON PARAMAGNETIC RESONANCE OF DISLOCATIONS IN SILICON

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The electron paramagnetic resonance spectra of centers formed as a result of plastic deformation of Si single crystals (D-centers) are investigated in the temperature range $1.3-300^{\circ}$ K. It is shown that with variation of temperature the D-center spectral intensity obeys the Curie law ($T = 1.3-4.2^{\circ}$ K). Throughout the temperature range investigated, spin-lattice interactions do not contribute significantly to broadening of the fine-structure lines; the line width is determined by the dipole-dipole and exchange interactions. An estimate of the spin-spin relaxation time based on the fine-structure line widths is $\tau_2 \sim 10^{-7}$ sec. The spin-lattice relaxation time is measured at helium temperatures. At $T = 1.3^{\circ}$ K, it is $\tau_1 \approx 70$ sec. Kinetics of D-center formation in single crystals with various dopants (P, B) is studied. D-centers arise in all plastically deformed single crystals regardless of the concentration or nature of the dopant or of the crystal-growing method. In n-type crystals the D-centers are acceptors and the "filling factor" of D-centers for electron capture is $f \leq 0.2$. An interpretation of the results based on the "dislocation" model is proposed.

INTRODUCTION

 ${
m T}$ HE investigation of plastically deformed semiconductor crystals by electron paramagnetic resonance (EPR) is a great interest both in connection with the theory of dislocations and from the point of view of studying the magnetic properties of linear spin chains, if, of course, it is assumed that each broken bond at the dislocation site in these crystals can have an unpaired spin. We therefore performed our experiments on Si monocrystals. Some of our results were published earlier $^{(1,2)}$ It was observed that in the plastic deformation of Si crystals paramagnetic centers arise, the concentration of which grows with increasing dislocation density (degree of deformation in the active stage). The EPR spectrum of these centers is anisotropic and possesses an incompletely resolved fine structure with a line width of several oersteds. At $T = 4.2^{\circ}K$ the g factors lie in the range 2.003-2.009 and shift to somewhat higher values as the temperature is increased.

There is no doubt that the centers described $in^{(1,2)}$ are associated with defects in the crystal lattice. However, it should be kept in mind that under plastic deformation not only dislocations but also point defects arise. The latter can give different paramagnetic centers just as in the irradiation of Si. A comparison of the EPR spectra of the defects arising during deformation and during irradiation of the crystals by fast electrons, as well as a comparison of some of their physical properties, showed that we have to do with defects that usually do not appear under irradiation. Because of this it was proposed $in^{(1,2)}$ that the centers formed during plastic deformation are due to electrons of broken bonds located in the nuclei of dislocations with edge components.

To establish finally the nature of these centers, we undertook a further investigation of the structure of the spectrum, as well as of the processes of formation of the centers in n- and p-type monocrystals with different concentrations of impurities. The present paper sets forth the results. Since the investigated centers are associated with lattice defects that arise under plastic deformation and are evidently due to dislocations, we shall call them D-centers for brevity.

EXPERIMENTAL METHOD

Since the general method was described in sufficient detail $in^{(2)}$, we give here only details that are specific for the present paper.

We used monocrystals of Si having the form of a parallelepiped the edges of which coincided with the crystallographic directions [112], [111], and [110] and had dimensions 3.5, 4, and 9 mm, respectively. The deformation was produced along [110] by compression at constant pressure and temperature of 700°C. After deformation the samples in an unloaded state were cooled to about 300°C in about a minute by a blast of argon and then were left to cool by themselves in air together with the carrier in which the deformation was produced. The total time of the cooling process did not exceed 30 min. With this treatment of the samples annealing of the centers did not amount to more than 10%.^[21]

After deformation a layer of thickness about 1 mm was first eliminated from each end of the samples, after which they were mechanically and chemically polished^[2]. The final volume of the samples was about 90 mm³. An X-band spectrometer was used for the measurements. At helium temperatures we usually observed the dispersion signal and at 77 and 300°K the absorption.

EXPERIMENTAL RESULTS

1. Investigation of EPR spectra of D-centers at different temperatures can give information about the spin-spin and spin-lattice interactions characteristic of these centers and in the final stage about the structure of the centers themselves. High-resistivity crystals are the most suitable ones to use for these investigations, since heavily doped crystals can be used only at low temperatures because of the small skin depth. We therefore used p-type Si crystals which had a room-temperature resistivity of about 3×10^4 ohm cm and were grown by zone melting in vacuum without a crucible. The initial dislocation density was about 5×10^3 cm⁻².

In Fig. 1 are the EPR spectra of the D-centers in these crystals recorded at different temperatures. As can be seen, the width of the fine-structure lines remains practically unchanged with temperature change in the whole range from 1.3 to 300°K. However, the intensity of the spectrum depends strongly on temperature. Quantitative measurements of the temperature dependence of intensity were made in the helium region. Here the dispersion signal was recorded in adiabatic fast passage (AFP)^[3]. Since under otherwise equal conditions the signal amplitude is proportional to the difference in population of the energy levels, then we in fact obtain the function $\chi_0(1/T)$ in relative units, where χ_0 is the static magnetic susceptibility. As is seen from Fig. 2, the function for the D-centers satisfies the Curie law in the helium region.

2. Investigation of the structure and form of the D-center spectrum for different modes of registration and orientations of the sample relative to the magnetic field direction showed that the lines are exchange narrowed. In fact, as is seen in Fig. 3, where the spectrum and a calculated Gaussian are shown, the D-center spectrum has long wings of the Lorentzian type. As is known^[4], this is evidence for exchange interactions.

It is of interest to study the processes of saturation of different portions of the spectrum to see directly whether there exists any interaction between the electrons whose resonance fields lie within one line and within different lines of the fine structure. For this the spectrum was recorded by the dispersion signal $(T = 1.3^{\circ}K)$ in AFP, and then the magnitude of the field



FIG. 1. EPR spectrum recorded for a sample $\rho \sim 3 \times 10^4$ ohm cm at various temperatures. The dc magnetic field H₀ was parallel to [111].

FIG. 2. Dependence of the static magnetic susceptibility on inverse temperature ($^{\circ}$ K).



FIG. 3. Dispersion signal in AFP at 1.3° K. Orientation of H₀ approximately the same as in Fig. 1. The signal is between the lines a and b of the Mn⁺⁺ spectrum. The dashed curve is a Gaussian of approximately the same area. The width of this curve is taken equal to the width of the spectrum at half height.

 H_0 was set, for example, to correspond to the maximum of the extreme right line of the fine structure (Fig. 3). If it is supposed that the spectral lines are ideally inhomogeneously broadened and consequently consist of isolated spin packets, then for a small amplitude of modulation of the magnetic field $H_M \ll \Delta H$, where ΔH is the width of this line, repeated passages through resonance leads to destruction of the magnetization of spin packets lying only in a narrow interval $H_0 \pm H_M$. Hence, upon repeated recording of the spectrum in a time interval short compared to the spin-lattice relaxation time (see below), it can be expected that a dip will be observed at the place of saturation^[3]. However, in our case, the spectral line was saturated practically as a whole and there was no dip. At the same time the ratio of the intensities of the other lines was almost unaffected. If a similar experiment is carried out with any of the central lines of the spectrum, a small dip appears at its position and the signal amplitude is decreased since the spectrum becomes weakly resolved.

3. We measured the spin-lattice relaxation time only at helium temperatures. The spectrum was saturated by repeated passages through resonance in AFP and after a certain time t recorded again. The population difference of the energy levels was thereby determined. By repeating such measurements, it is possible to obtain the dependence of the population difference n on time. As is seen in Fig. 4, the systems tends to equilibrium exponentially: $n(t) = n(\infty) + [n(0) - n(\infty)]exp(-t/\tau_1)$, where τ_1 is the spin-lattice relaxation time. For $T = 1.3^{\circ}$ K, we find $\tau_1 \approx 70$ sec. Determination of the temperature dependence showed that in the helium region it is of the type $\tau_1^{-1} \sim T$.

In measuring the time τ_1 by the process of saturation we observed a phenomenon of "anomalous" loss of sample magnetization upon repeated passages of the spin system through resonance under AFP conditions. This effect is illustrated in Fig. 5. As is seen from this figure, the time between two successive passages $\Delta t \approx 3$ sec and consequently we have $\Delta t \ll \tau_1$. Hence we would expect a signal decrease (for double passage) due



FIG. 4. Change of population difference of energy levels with time after saturation (T = 1.3° K).

FIG. 5. Decrease of signal upon repeated passage through resonance in AFP. The spectrum was recorded only with the field changing in one direction. The time between consecutive passages $\Delta T \approx 3 \sec (T = 1.3^{\circ} \text{K})$.

to spin-lattice relaxation (at least in order of magnitude) equal to $\Delta t/\tau_1 \sim 5\%$.^[3] However, the actual signal decrease turned out to much greater, about 40%. Because of this phenomenon, in order to avoid a large error in determining τ_1 , we made only one measurement of intensity in a certain interval of time after saturation of the spectrum. To determine the intensity after some other time interval the spectrum was saturated again.

4. The formation of D-centers in doped Si crystals is of interest from two points of view. First, one would like to know the effect, if any, of electrically active impurities on the processes of formation and disappearance of D-centers. Second, we need to see if there is any correspondence between the D-center concentration and the magnitude of the relative deformation in different stages.

We investigated a broad class of n- and p-type monocrystals with impurity (P, B) content in the range from 10^{11} to 10^{16} cm⁻³. The investigation showed that D-centers arise upon plastic deformation of all monocrystals regardless of the type and concentration of dopant and of the method of crystal growth. Certain quantitative results that reflect the kinetics of formation of D-centers in Si crystals during deformation are presented in Fig. 6. The upper curves give the dependence of the magnitude of deformation $\epsilon(\%)$ on time t (min) with constant tangential pressures acting on glide planes of the type $\{111\}$. The points on the curves correspond to the moments the deformation was terminated. For the selected sample sizes the reproducibility of the $\epsilon(t)$ curves for crystals of one type was not worse than 5-10%. The lower curves reflect the dependence of the integrated intensities (ND of the D-center spectra on time. The graphs of $N_{D}(t)$ are normalized, so that $N_{D \max} = 1$. The experimental values of $N_{D \max}$ for samples with different dopants are of the same order. In individual cases there were significant departures in the smallest values, which may be due to fluctuations in the mechanical properties of these crystals.

As is seen from Fig. 6, the concentration of D-centers at constant pressure and temperature is a function



FIG. 6. The upper curves represent the dependence of magnitude of deformation $\epsilon(\%)$ on time t(min). The time scale is nonlinear. The samples were deformed at constant values of tangential pressures ~4.5 kg/mm² acting in glide planes of the type {111}. The lower curves reflect the dependence of the integrated intensities of the D-center spectra: 1-for samples with $\rho \sim 3 \times 10^4$ ohm cm; 2-for samples with phosphorous concentration ~10¹⁵ cm⁻³; 3-for samples with boron concentration ~10¹⁵ cm⁻³.

of many parameters: $\epsilon, \dot{\epsilon}, t$. In the first stage of deformation the magnitude of $\dot{\epsilon}$ is large, since the process of generation of D-centers is going on. In the second stage it is small; a process of decreasing concentration with time is going on. In this the presence of donor impurity is practically immaterial, whereas in samples with acceptor impurity there is a markedly different behavior of $N_D(t)$ compared to undoped crystals.

5. In view of the prior assumptions [1,2] about the dislocational nature of the D-centers and the fact that dislocations are electrically active defects, it is of great interest to see how the presence of D-centers in Si monocrystals affects the charge state of donor impurities at low temperatures. To this end we conducted experiments on the plastic deformation of Si crystals with a phosphorus concentration $\gtrsim 10^{15}$ cm⁻³. Figure 7 is the EPR spectrum of the dispersion signal in AFP at 1.3° K. Curve 1 is that of the control sample; one sees two intense lines from phosphorus and a very weak central line. Under plastic deformation D-centers arise and the intensity of the phosphorus line is diminished (curve 2). Decrease of the D-center concentration in the second stage of deformation leads to reestablishment of the intensity of the phosphorus lines (curve 3).

Results of quantitative measurements of the integrated intensities of these spectra for different deformations are shown in Fig. 8. As is seen from this figure, curve 1, which is the function $N_{\rm P}(t)/N_{\rm P}(0)$ (here $N_{\rm P}(t)$ is the concentration of paramagnetic phosphorus atoms), is, in the first approximation, the ''mirror'' image of curve 2; the latter corresponds to the function $N_{\rm D}(t)/N_{\rm D}\,{\rm max}$. The lower curve in Fig. 8 reflects the dependence of the magnitude of deformation of the investigated crystals on time at constant temperature and constant pressure acting on the sample.



FIG. 7. Dispersion signals recorded in AFP at T = 1.3° K: 1-for initial sample with phosphorus content ~ 10^{15} cm⁻³ which underwent thermal treatment, but not deformation; 2, 3-for plastically deformed samples.

FIG. 8. Upper curves: 1-dependence of integrated intensity of P signal on time; 2-dependence of integrated intensity of D-center spectrum on time. The time scale is nonlinear. Below is the dependence of the magnitude of deformation $\epsilon(\%)$ on time. The deformation was produced by tangential pressures in {111} slip planes, equal to ~9.5 kg/mm².

DISCUSSION

The results show that D-centers arise in all crystals regardless of how they are grown (zone melting without crucible or Czochralski technique; the methods introduce differing amounts of oxygen, 10^{16} to 10^{18} cm⁻³) and regardless of the type and concentration of doping agent (P, B). It follows that impurities have no direct effect on the formation of D-centers, and consequently the centers are due to lattice defects, i.e., first to valence bonds broken in the deformation process, and second. the paramagnetism of these defects is not due to the capture or loss of electrons, since the form of the spectrum, as well as the process of formation of D-centers, is the same in n- and p-type crystals. This means that the lattice defects are paramagnetic and electrically neutral. They can only be "large" defects caused by agglomeration of broken bonds, since elementary defects in an electrically neutral state are not paramagnetic, as a rule; i.e., the broken valence bonds form, by overlapping of wave functions, a configuration the total spin of which is zero.^[5]

Since the electrons of the broken bonds are strongly localized, all lattice defects have, as a rule, deep energy levels^[5,6]. In the case of D-centers the ionization energy $E_i \gg kT$ up to room temperature. This follows from the fact that the centers remain un-ionized and signal is observed even at $T \approx 300^{\circ}$ K (Fig. 1).

In view of these remarks, we can draw the following conclusions from our experimental results:

1. The distance between the interacting spins (in spite of the fact that the maximum attained value of

D-center concentration is small and does not exceed 5×10^{16} cm⁻³) is comparable with the lattice constant. In fact, since the paramagnetic electrons of the D-centers are strongly localized, the overlap of their wave functions and consequently the presence of exchange interaction is possible only when the spacing between the neighboring spins is of the order of the lattice constant. And from this it then follows that we actually have to do with groups of closely arranged centers.

2. With a distance between spins of the order of the lattice constant a, the line broadening on account of dipole interactions only will be of the order μ_0/a^3 (μ_0 the Bohr magneton), which for a ~ 4 Å gives ~ 100 Oe. Since experimentally one observes a fine-structure line width equal to several oersteds, we can conclude that the exchange interaction strongly narrows the line in the central portion, leaving long wings of the Lorentzian type.

3. The saturation experiments show that each line of the fine structure corresponds to a group of spins with closely spaced resonance fields. Because of spin-spin interaction there is a transfer of excitation among spins at least within a single group, since the line saturates as a whole. Groups of spins whose resonance fields lie within the limits of different lines are "isolated" from one another, since saturation of one fine-structure line does not produce saturation of the other lines. Dislocations can have such properties. In fact, the distance between broken bonds in dislocation nuclei with edge components can be of the order of the lattice constant, and therefore interaction between paramagnetic electrons of adjacent bonds is possible. On the other hand, since the distance between dislocations is large on the average (even with a density of $\sim 10^8$ cm⁻²), spins located on different dislocations are practically isolated from each other. In this case equivalent dislocations will indeed give the groups of spins mentioned above. The presence of several lines in the spectrum can be attributed to some anisotropy and thus to preferred crystallographic directions along which the dislocations of the same type are oriented. Obviously, increasing the temperature of deformation will produce a more isotropic distribution of dislocations in the slip planes, and hence we might expect in this case a gradual disappearance of fine structure in the spectrum.

4. The presence of exchange interactions, generally speaking, can lead to ordering in a spin system, even a linear chain^[7,8]. Pokrovskii^[8], in discussing our results^{(2]}, assumed that the presence of fine structure in the D-center spectrum may be connected with excitation of spin waves in chains of finite length caused by the dislocations. However, subsequent interpretation of all the experimental results on the basis of this model is beset with a number of difficulties. First, there is no important effect of an electrically active impurity on the form of the spectrum, as one would expect in the case of spin waves on chains of approximately equal length^[8]. Further, $\chi_0(1/T)$ is linear (Fig. 2), which is hard to explain if there is an ordered spin system. Nonetheless, this interpretation is attractive; we obviously need more investigation before final conclusions are made.

5. The phenomenon of "anomalous" magnetization loss upon passage of the spin system through resonance

in AFP can be explained by the presence of spin-spin interactions. (Similar effects were observed in an investigation of paramagnetic impurities in $silicon^{(3)}$ and explained by discrete spin diffusion due to hyperfine interactions of electrons with Si^{29} nuclei.) However, the specific mechanism of the interaction between spins responsible for the loss is not established.

6. It is seen from Fig. 1 that the fine-structure line width is practically independent of temperature. This is convincing evidence that the spin-lattice interactions are weaker than the spin-spin interactions ($\tau_1 \gg \tau_2$, where τ_2 is the spin-spin relaxation time), and hence spin-lattice interactions do not contribute significantly to the line width. Thus we can estimate τ_2 from the width of the fine-structure lines. We find $\tau_2 \sim 10^{-7}$ sec.

7. The measurements of the spin-lattice relaxation time showed that the relaxation process is exponential (Fig. 4). This means that the spin system can be thought of as a two-level system. When more levels participate, one usually observes departures from the exponential law^[9]. In the helium region we find $\tau_1^{-1} \sim T$. This means that the relaxation process at these temperatures is accompanied mainly by direct transitions of the spins with participation of only one phonon. In the case of multi-phonon processes one usually observes a power dependence on $T^{[4]}$.

8. The investigations of the kinetics of D-center formation in the plastic deformation showed (Fig. 6) that the concentration of centers is not an unambiguous function of time (magnitude of deformation). Introduction of some dopant or other leads to certain changes in the mechanical properties of the crystal. In the final analysis this affects the process of generation and disappearance of D-centers. In the first stage, when there is an intense generation of dislocations, the concentration of the D-centers grows almost proportionately with ϵ . In the second stage the concentration of centers decreases gradually. The latter can be explained by different phenomena: decrease in the average density of dislocations due to coagulation of small dislocation meshes into larger ones, reduction of the number of kinks in the dislocations, change of dislocation type, etc. The fastest drop in concentration occurs in pure crystals. This means that the reduction in the number of paramagnetic centers is not associated with processes of diffusion of an electrically active impurity to the broken bonds. In the presence of diffusion, the concentration in pure crystals decreases more slowly than in impure ones. Thus we conclude that a chemical impurity influences the formation of D-centers only so much as it affects the plastic properties of the crystal, i.e., the motion and generation of dislocations. Note that in^[1] we observed a change in the form of the spectrum when the crystal was plastically deformed. The investigations of^[2], as well as those of the present paper, show that a change in the form of the spectrum takes place only in the region of large deformations, when the sample shape is highly distorted and g-factor anisotropy affects the width and even the position of the fine-structure lines. For $\epsilon \lesssim 5-6\%$ this effect, as a rule, is unimportant.

9. In n-type crystals formation of D-centers leads to a reduction in the number of paramagnetic donor atoms (Fig. 7). Generally speaking, one would try to explain this effect, not by the capture of donor electrons, but by the diffusion of impurity atoms, in this case P, to dislocation sites, the formation of Si-P bonds, and the transition of P to a nonparamagnetic state as a result. However, as has already been mentioned, diffusion processes have low probability. This is also confirmed by the fact that the central line in the P spectrum, which is sensitive to the spatial distribution of P atoms, does not undergo any anomalous changes in either the initial or final stages of the deformation. Considering that the curve $N_{\mathbf{p}}(t)$ is the "mirror" image of the curve $N_D(t)$, we conclude that the D-centers themselves are responsible for the decrease in the intensity of the P lines, and consequently they behave like acceptors in n-type crystals- some of the electrons go from phosphorus levels to D-center levels which are lower in the forbidden band. However, it is important that the number of D-centers capable of capturing electrons is much less than their total number, i.e., the filling factor for electron capture of D-centers $f \ll 1$. If we realize that a D-center that has captured one electron ceases to be paramagnetic and consequently does not contribute to the integrated intensity of the spectrum, it is easy to show that f is given by

$$f = \frac{N_{\rm P}(0) - N_{\rm P}(t)}{N_{\rm D}(t) + N_{\rm P}(0) - N_{\rm P}(t)}.$$

The notation is the same as before. By determining the integrated intensities of the spectra of D-centers and phosphorus corresponding to identical deformations, we find from this expression that $f \sim 0.2$.

We should say a few words about the error in determining f. To construct the graphs of $N_{D}(t)$ and $N_{D}(t)$ in relative units (Fig. 8) it suffices to carry out all measurements under identical conditions, and the mode of recording the spectra is generally speaking unimportant. However, in determining the value of f it is necessary to compare integrated intensities of P and D-center spectra, hence it is necessary that the conditions of passage through resonance of P and D-centers be the same. Otherwise, the effects of the passage can lead to significant error, since the spectrum is recorded under AFP conditions, and the spin-lattice relaxation times at 1.3° K for P and the D-centers differ by more than an order of magnitude.^[9] In our case the spectra were recorded in such a way that the AFP conditions were fulfilled sufficiently rigorously both for P and for the D-centers. Nevertheless, the effect of "clipping" the wings in the D-center spectrum leads to a value of ND that is too low compared to N_P, and this in turn, makes the value of f too high. Estimates show that in our worst case this error is still not greater than 30%.

Numerous experimental investigations of the electrical properties of plastically deformed Ge and Si monocrystals^[10,11] show that dislocations with edge components possess acceptor properties of this type. The broken bonds in the dislocation nuclei turn out to be capable of trapping an electron and thus become negatively charged. Since the distance between bonds is of the order of the lattice constant, a Coulomb interaction arises between the electrons, so that even at low temperatures the electrons are trapped only by bonds that are sufficiently far from one another. As a result of this the filling coefficient of broken bonds is much less than unity. As follows from the thermodynamic calculations of Read⁽⁶⁾ under the assumption that the acceptor dislocation level is located 0.2 eV from the bottom of the conduction band, the value of $f \sim 0.1$ for a pure edge dislocation. Note that the presence in the crystal of dislocations with smaller edge components can lead to a certain increase in the average value of f. Thus, we conclude that our value of f agrees qualitatively with theoretical predictions.

CONCLUSION

All of our information about the properties of D-center spectra (see $also^{(2)}$) suggests that D-centers are due to the electrons of "dangling" bonds in the nuclei of dislocations with edge components and consequently are linear chains of spins that are about a lattice constant apart from one another. In this connection it should be noted that an EPR investigation of plastically deformed Si crystals was also carried out in^[12,13]; however, they did not get a signal of purely "deformational" origin. This may have been due to the high temperature of the deformation process ($\gtrsim 900^{\circ}$ C), at which it is usually not possible to obtain in the samples a sufficient concentration of any kind of paramagnetic centers because of annealing.

In^[14,15] an EPR signal was observed in deformed Si crystals, and the authors attributed it to dislocations. However, unlike our results, the spectrum consisted of a single line of width ~ 11 Oe with an almost isotropic g factor that varied from 2.0032 to 2.0053 in different samples; the energy of activation for the annealing process was about 1.36 eV (instead of about 2 eV in our case^[2]); exchange interactions were absent; from the line width Wöhler et al.^[15] concluded that the paramagnetic electrons were quite far apart and probably localized on kinks of the dislocations. The nature of these centers was not finally established, but it is clear that dislocations play an important role, since a certain correlation was observed between a change in the signal and a change in the dislocation structure during annealing of the samples. [15]

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