Electric conductivity and Hall effect in silicon single crystals with dislocations

V. A. Grazhulis, V. Yu. Mukhina, Yu. A. Osip'yan, and S. A. Shevchenko

Institute of Solid State Physics, USSR Academy of Sciences
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The conductivity and Hall effect of plastically deformed n-type silicon single crystals containing 5×10^{16} cm^{-3} phosphorus atoms were investigated experimentally in the 100–4.2 K temperature range. The following anomalies in the temperature dependence of the free-electron mobility μ were observed in crystals with dislocations: 1) In the region $T<20$°K, an exponential decrease of the mobility with decreasing temperature was observed, with an activation energy $E_\mu\simeq0.016$ eV; 2) in the vicinity of $T=50$°K, a mininum was observed in the mobility temperature dependence, with the position of minimum and shape of the $\mu(1/T)$ curve depending on the dislocation density. It is suggested that the minimum in the $\mu(1/T)$ curve is due to the scattering of the carriers by the dislocations. The crystal conductivity in the low temperature region $T<20$ K is of the hopping type. The temperature dependence of the sample resistance is of the form $\rho=\rho_3\exp(E_3/kT)$. The results are interpreted on the basis of a model according to which there exists in deformed crystals at low temperatures, besides the hopping conductivity along the donor impurities, also a dislocation conductivity that is predominant at high dislocation densities.

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INTRODUCTION

It can now be regarded as established that the presence of dislocations in semiconductor and dielectric crystals produces in the forbidden band of the energy spectrum new electronic states that are connected with the dislocations themselves, but the details of the structure of the spectrum of these states are still unclear. In particular, there is no answer as yet to the fundamental question whether the dislocation-level system should be regarded as a system of local levels or as a one-dimensional dislocation band (or several bands).[1, 2] The choice of any particular model depends, of course, on the degree of overlap of the wave functions of the electrons in the dislocation cores. If this overlap of the wave functions is appreciable, then it should lead, generally speaking, to a conductivity along the dislocations; this conductivity can be of the hopping type in the presence of localized states and of the metallic type in the case of a band. The possibility of experimentally observing conductivity along dislocations is quite enticing and there is no doubt that a study of this question is quite timely.[3, 4]

It should also be noted here that the question of scattering of free carriers by dislocations is also worthy of considerable attention. The study of carrier mobility can yield valuable information not only on the interaction of the carriers with the elastic-stress fields and the electric fields around the dislocations, but also on the spin state of the dislocations as scattering centers.[5] The latter is of particular interest, since it has been suggested earlier[6] that silicon has a magnetic phase transition at T ≈ 50°K in a dislocation spin system.

The foregoing considerations have induced us to investigate the electric conductivity and the Hall effect in dislocation-containing single crystals of silicon. We present below the experimental results and their discussion.

SAMPLES AND EXPERIMENTAL PROCEDURE

We have investigated n-type single crystals of silicon grown by crucibleless zone melting, doped with phosphorus, and having an excess donor concentration 5×10^{16} cm^{-3}. The choice of such crystals was dictated mainly by the difficulty of preparing satisfactory ohmic contacts (for low-temperature research) on the surface of weakly-doped silicon crystals.[7] The degree of compensation of the initial crystals was $K\ll1$, and the concentration of the compensating acceptors was $10^{12}-10^{13}$ cm^{-3}. The initial samples were parallelepipeds measuring $4\times4\times10$ mm. The long edge coincided with the [110] direction. The dislocations were introduced into the crystal by plastic deformation due to compression of the samples along this direction. The deformation was effected at temperatures 700 and 750° in an inert medium. During the course of the deformation we plotted the $\epsilon(t)$ curve, where ϵ is the deformation and t is the deformation time.

Two batches of samples were prepared. In the first the samples were deformed at 700°C and at a normal stress ≈ 12 kgf/mm². The deformation was stopped in the different samples at the points c, d, e, f, g, and h, respectively (Fig. 1). In addition to these samples, we prepared an initial sample a and a control sample b. The latter was heat treated in the same manner as sample h. After the deformation, the samples were spontaneously cooled (together with the holders) for several minutes to a temperature close to room temperature.

The samples of the second batch were deformed at 750°C until the knee of the $\epsilon(t)$ curve was reached, under various external stresses in the range from 1 to 12 kgf/mm² (in the first batch, the knee is designated by the letter f). The samples and the corresponding deformation curves were designated by the letters C, D, E, and F (see Fig. 1).

For both batches, we measured the Hall effect and

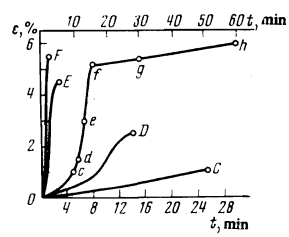

FIG. 1. Dependence of the deformation (ϵ) on the time (t) for samples of the first batch (c, d, e, f, g, h) and samples of the second batch (C, D, E, F). The upper t axis pertains to the first batch.

the electric conductivity in the temperature interval 100–4.2°K. The Hall effect was measured by a null method in a constant magnetic field ≈ 6 kG and with direct current flowing through the sample. The required sample temperature was maintained constant in the entire range within ~2%.

The samples on which the measurements were performed measured 1.5 × 1.5 × 8 mm. All the samples were mechanically polished and then chemically polished in a solution HF:7HNO$_3$ prior to the deformation. Additional information on the procedure used to prepare the samples can be found in the earlier papers.[8, 9]

EXPERIMENTAL RESULTS

We note first that in spite of the different crystal deformation conditions, all the results obtained for both sample batches agree qualitatively. We shall therefore present henceforth, by way of illustration, only the results obtained for the first batch of samples.

Figures 2 and 3 show plots of the sample resistivity against the temperature. One can see clearly two temperature regions characterized by different slopes of the linear sections of the curves relative to the coordinate axes (in logarithmic scale, the $\rho(1/T)$ can be extrapolated by means of straight lines with an error $\lesssim 15\%$ in both temperature intervals).

The slope of the log $\rho(1/T)$ line in the high-temperature region, $T > 20°K$ (or the activation energy E_1) is determined mainly by the activation energy of the donor impurity (phosphorus). The difference between the slope angles for samples with different dislocation densities is small, so that E_1 can approximately be regarded as independent of the dislocation density.

The increase of the resistivity with increasing dislocation density (see Fig. 2, the region $T > 20°K$) is due primarily to the decrease of the concentration of the free electron as a result of the acceptor action of the dislocations. As seen from Fig. 4, in the region $T > 20°K$ the electron concentration is smaller the higher the degree of deformation on the first stage of the plastic deformation (samples e and f), i.e., the higher the dislocation density.[9] During the second stage of plastic deformation (samples g and h), a certain decrease of the average dislocation density takes place during the course of polygonization[9], and accordingly the concentration of the free electrons increases (Fig. 4) and the resistivity of the samples decreases (Fig. 3). We note that curve g of Fig. 3 and curve f of Fig. 2 practically coincide in position.

It must be emphasized here that the electron concentration (as well as their mobility, which will be discussed later) was determined under the assumption that the Hall constant R is connected with the carrier density n by the very simple relation $R = (ne)^{-1}$, and the Hall factor

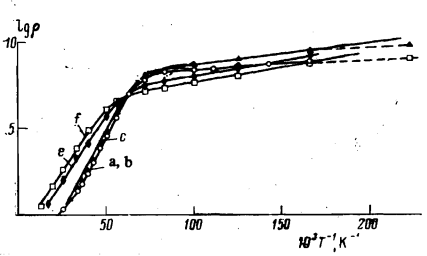

FIG. 2. Dependence of the bulk resistivity $\rho(\Omega\text{-cm})$ on the reciprocal of the temperature (T, °K); a–initial sample, b–control sample.

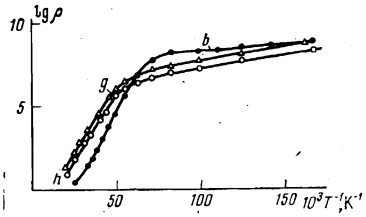

FIG. 3

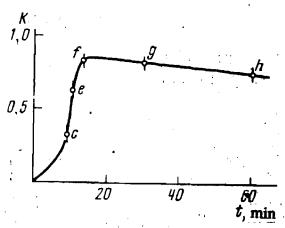

FIG. 5

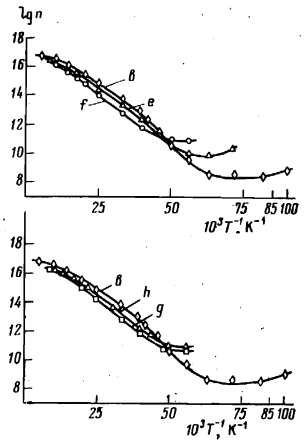

FIG. 4

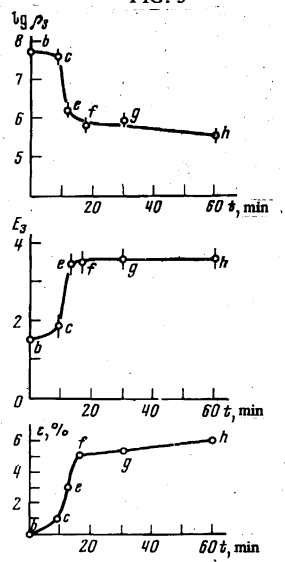

FIG. 6

FIG. 3. Plot of the bulk resistivity $\rho(\Omega\text{-cm})$ of samples deformed during the second stage (see Fig. 1).

FIG. 4. Dependence of the electron density (cm^{-3}), defined as $n = (Re)^{-1}$, on the reciprocal temperature. The upper and lower curves correspond to the first and second stages of the deformation (see Fig. 1).

FIG. 5. Dependence of the compensation coefficient K on the deformation time.

FIG. 6. Dependence of the parameters E_3 and ρ_3 and also of ϵ on the deformation time (E_3 is in milli-electron volts and ρ_3 in Ω-cm).

was assumed equal to unity in the entire temperature interval. Thus, Fig. 4 shows plots of $\log n = \log(Re) = f(1/T)$. As seen from this figure, the $\log n(1/T)$ curves flatten out at $T < 20°K$. In the vicinity of $T \sim 20°K$ the Hall emf decreases strongly with decreasing T, and at $T < 10°K$ its value becomes vanishingly small. We therefore measured the Hall effect for all the samples only to $T \approx 15°K$, whereas the electric conductivity was measured all the way to 4.2°K. Knowing the behavior of the electron density in the conduction band in the $T > 20°K$ region, we can determine the coefficient of compensation of the donor impurity (K) by the dislocations, which act as acceptors. We define K as $K = \Delta n/n_0$, where Δn is the decrease of the density of the free electrons as a result of the dislocations, and n_0 is the initial electron density in the conduction band. Using the data of Fig. 4, we can determine $K = f(1/T)$ for different samples. It turns out that at $T < 50°K$ the value of K is practically independent of the temperature, but in the region $T > 50°K$ the value of K decreases substantially with increasing T.

Figure 5 shows plots of K against the deformation time for various samples. The values of K were determined in the region $T \approx 30°K$. As seen from the figure, K increases with increasing dislocation density during

the first stage of the deformation (samples c, e, and f) and decreases somewhat during the second stage of deformation (samples g and h).

As already noted earlier, at low temperatures, $T < 20°K$, the slope of $\log \rho(1/T)$ changes abruptly in comparison with the high-temperature region and depends significantly on the deformation (see Figs. 2 and 3). In the vicinity of ~5°K, there is observed also a certain deviation from a linear dependence of the function $\log \rho(1/T)$. This is clearly seen from curves c and f of Fig. 2 (for samples c and f the measurements were carried out to 4.2°K). Extrapolating the plots of $\log \rho(1/T)$ by straight lines for all the samples (in the interval $10^3/T = 80$ to 170) and introducing the activation energy for the low-temperature conductivity, E_3, we can represent $\rho(T)$ in the form

$$\rho^{-1}(T) = \rho_1^{-1} \exp(-E_1/kT) + \rho_3^{-1} \exp(-E_3/kT)$$

(we follow here the notation employed in the theory of hopping conductivity, since E_3 for the initial and control samples corresponds to the activation energy of the hopping conductivity along the donor impurity[10]).

Using the curves shown in Figs. 2 and 3, we can readily determine the parameters of E_3 and ρ_3, which are mainly of interest. The dependences of these parameters on the deformation are shown in Fig. 6. As seen from this figure, in the deformation region $\epsilon < 3\%$ the value of E_3 increases rapidly with increasing degree of deformation (dislocation density), while ρ_3 decreases. At $\epsilon > 3\%$, further deformation of the crystal, i.e., a growth of the dislocation density (samples e and f) or else a decrease of this density (samples g and h), leads to no change of E_3' the change of the resistivity ρ_3 is likewise insignificant. Thus, at $\epsilon_3 < 3\%$ the values of E_3 and ρ_3 depend strongly on the deformation, and at $\epsilon > 3\%$ the energy E_3 does not depend on the deformation, while ρ_3 depends on it weakly.

We proceed to report the results on the Hall mobility of the electrons.

Figures 7 and 8 show the temperature dependences of the Hall mobility of the carriers, $\mu = R\sigma$, where σ is the bulk conductivity of the crystals. As seen from Fig. 7, the behavior of μ is qualitatively the same for the initial sample a and for the control sample b. For deformed samples, the following attributes are typical: first, a general decrease of the mobility with increasing dislocation density (see curves d, e, and f) in the entire temperature range, and second, the presence of a minimum of the mobility in the region 40–50°K. The minimum becomes smeared out with increasing dislocation density and degenerates practically into a step for strongly deformed crystals (curve f). During the second stage of the deformation, the mobility is partially restored (see Fig. 8, curves g and h).

The described peculiarities in the behavior of μ pertain to the high-temperature region $T > 20°K$. In the region $T < 20°K$ the value of μ decreases exponentially with decreasing temperature (see Figs. 7 and 8), i.e., $\mu = \mu_0 \exp(-E_\mu/kT)$, where $E_\mu \approx 1.6 \times 10^{-2}$ eV for all samples.

For the samples of the second batch, the minimum on the $\mu(T)$ curves is less pronounced and practically all the $\mu(T)$ curves for the samples of this batch are similar to curves f, g, and h in Fig. 8. In concluding the experimental part we note that we did not investigate the

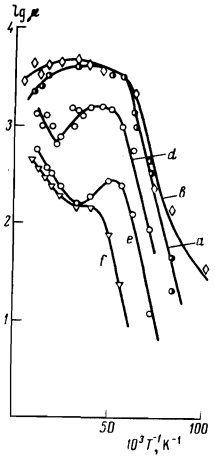

FIG. 7

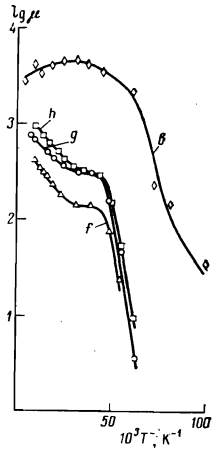

FIG. 8

FIG. 7. Mobility vs. the reciprocal temperature for samples of the first deformation stage.

FIG. 8..Mobility vs. the reciprocal temperature for samples of the second deformation stage.

annealing processes. It was shown earlier[8, 9] that under the chosen crystal deformation and cooling conditions the overwhelming majority of the point defects become annealed.

DISCUSSION OF RESULTS

We note first that in an earlier[8] study of the EPR spectra of silicon containing phosphorus atoms and dislocations we have shown that the diffusion of the phosphorus atoms towards the dislocations cannot be completed within the time of plastic deformation of the crystals. This manifests itself in the EPR spectra by the fact that no significant intensity of the main lines of phosphorus is transferred to the lines due to clusters of phosphorus atoms. The ratio of the intensities of these lines is the same for both the control samples and the deformed samples, including the samples deformed for a long time during the second deformation stage. During this stage, the dislocation density changes slowly with time, so that the conditions for diffusion are most favorable. This conclusion is confirmed also by data obtained in the present study, namely, as already noted, there is a qualitative agreement between the results for both sample batches (in spite of the considerable differences between the deformation rates and the temperatures).

Thus, there are grounds for assuming that the spatial distribution of the impurities is not significantly altered during the course of the deformation of the crystals.

As already mentioned, the initial crystals are partially compensated, and therefore hopping conductivity along the donor impurities is observed in the region $T < 20°K$ ($\rho = \rho_3 \exp[E_3/kT]$) with activation energy $E_3 \approx 1.5 \times 10^{-3}$ eV-cm (Figs. 2, 3, 6). In the case of deformed crystals, as seen from Figs. 2 and 3, there is qualitatively the same $\rho(1/T)$ dependence at $T < 20°K$, but certain peculiarities are also observed. The latter are connected with the fact that these crystals are compensated not by a chemical impurity but by dislocations that serve as acceptors. To assess the distinguishing feature of the influence of the dislocations on the low-temperature conductivity of the investigated samples, it is expedient to compare the properties of crystals com-

pensated with chemical acceptors and crystals compensated by dislocations.

1. In the case of hopping conductivity the donor impurities partially compensated by chemical acceptors, the activation energy E_3 has a minimum at compensation coefficients $K \approx 0.4-0.5$, and increases quite rapidly as $k \to 1$ (approximately in proportion to $(1-K)^{1/3}$ [9]). A comparison of the plots of $E_3(t)$ and $K(t)$ on Figs. 5 and 6 shows that in our case, first, there is no minimum and, second, at $K > 0.6$ the energy E_3 does not depend at all on K. For the initial crystals (a, b) the energy is $E_3 = 1.5 \times 10^{-3}$ eV, and for strongly deformed crystals $E_3 \approx 3.6 \times 10^{-3}$ eV.

2. A comparison of the $\rho_3(t)$ and $K(t)$ curves for samples of both series shows that at $K > 0.6$ the quantity ρ_3 is likewise independent of the value of K (see Figs. 5 and 6), and moreover there is no one-to-one correlation whatever between ρ_3 and K at $K > 0.6$. In fact, on going from sample e to sample f, the parameter K increases while ρ_3 decreases. If we assume a unique relation between ρ_3 and K, then on going from sample f to sample K, they there should be an increase of ρ_3 (by virtue of the decrease of K), but we observe a decrease of ρ_3. Thus, ρ_3 is not a function of K (at $K > 0.6$) but depends uniquely on the deformation time. We note that in the case of an impurity compensation $K > 0.5$ the value of ρ_3 increases monotonically with increasing K, owing to the fact that the Fermi level E_F is located at $K > 0.5$ in the lower tail of the impurity states, and with increasing K the E_F level drops lower, so that the density of states responsible for the hopping conductivity decreases in the vicinity of E_F.[10, 11]

Thus, a comparison of our results with the data available on impurity hopping conductivity shows that the behavior of dislocation-compensated crystals cannot be described by the standard theory of hopping conductivity. The behavior of E_3 and ρ_3 as described by us can be explained by assuming that crystals with dislocations have, besides conductivity along the donor impurities, also conductivity along the dislocations, and the latter predominates in strongly deformed crystals.

We note that when crystals are compensated by a chemical acceptor impurity, there can be no conductivity along the acceptor impurity at low temperatures, since all the acceptors are occupied by electrons. When the crystals are compensated by dislocations, the role of acceptors is assumed by the broken bonds in the cores of the dislocations, and even at low temperatures not all the bonds are occupied, but only 0.1–0.2 of their total number.[1, 2, 9] One can therefore not exclude the possibility of deformed crystals having an n-type conductivity along the acceptor "impurity," namely the dislocations.

In such a model, the quantity $E_3 = 1.5 \times 10^{-3}$ corresponds to a "pure impurity" hopping conductivity, and $E_3 = 3.6 \times 10^{-3}$ corresponds to "pure dislocation" conductivity. The intermediate values of E_3 (see Fig. 6) correspond to a transition region, in which a change takes place in the conductivity mechanism).

The decrease of the activation temperature E_3 with decreasing temperature in the low-temperature region (as is usually the case with impurity conductivity[10, 11]), and also the independence of E_3 of the dislocation density (Figs. 2 and 6) can be explained by assuming that the electrons contributing to the dislocation hopping conductivity overcome potential barriers due principally to fluctuations of the linear charge density on the dislocations themselves. It is obvious that with decreasing temperature the fluctuations in the distribution of the electrons captured by the dislocations will decrease, and this will lead to a decrease of E_3. On the other hand, since the population of the dislocations by electron in n-type crystals at $N_d > N_D$ (N_d is the donor density and N_D is the density of the broken dislocation bonds) at low temperatures is independent of the dislocation density,[1, 2] it is clear that E_3 should likewise be independent of the dislocation density.

The value of ρ_3 in the model of hopping conductivity along dislocations will be determined mainly by the degree of overlap of the wave functions for states localized on neighboring broken bonds.[10] It is obvious that this overlap will depend only on the type of the dislocations. Generally speaking, the value of ρ_3 should also depend on the dislocation density, or more accurately both on the dislocation density and on the form of the dislocation structure. Indeed, one can conceive of a crystal in which there is a high dislocation density, but nonetheless the conductivity along the dislocations is low. This is a crystal containing a system of intersecting dislocation loops. It is clear therefore that the growth in the number of dislocation intersections should greatly increase the effective conductivity along the dislocations. Therefore the decrease of ρ_3 in the course of deformation (see Figs. 2, 3, 6) can be attributed to the increase of the dislocation density and to the increase in the number of dislocation intersection. The latter is particularly appreciable during the second deformation stage, when polygonization processes occur.

The absence of the Hall effect at low temperatures can be explained by assuming that the electrons localized on the dislocations and impurities make no appreciable contribution to the Hall effect, for example by virtue of the one-dimensional character of the dislocations or the low mobility of the electrons localized on the dislocations and impurities.

We note that the Hall mobility $\mu = R\sigma$ shown in Figs. 7 and 8 has values $\mu \gg 1$ cm^2/V-sec and corresponds to the mobility of free electrons in the conduction band, i.e., it is not connected with hopping mobility along the impurities or dislocations. In fact, in the region of hopping impurity conductivity with activation energy E_3 we have $\mu \sim \exp[-E_3'/kT]$, where $E_3' \lesssim E_3$.[11] However, for the crystals investigated by us (Figs. 7 and 8) at $T < 20°K$ we have $\mu \sim \exp[-E_\mu/kT]$ and $E_\mu \approx 1.6 \times 10^{-2}$ eV, and consequently $E_\mu \gg E_3 \approx 1.5 \times 10^{-3}$ eV. For dislocation conductivity we have similarly $E_\mu \gg E_3$. It is advisable to note here also that in the case of impurity hopping conductivity[11] that value of μ is usually much less than 1 cm^2/V-sec.

We thus assume that in the entire investigated temperature range the Hall effect is determined entirely by the free carriers, i.e., $R \approx R_{free} \gg R_{loc}$, where R_{free} is the Hall constant for free carriers, R_{loc}, where the constant for carriers localized on the donor impurity or dislocations, and $\mu = R\sigma$ corresponds to the mobility of the free electrons in the conduction band. The "vanishing" of the Hall effect can be attributed to the exponential decrease of μ of the free carriers with decreasing T. We note that the quantity μ_0 in the expression $\mu = \mu_0 \exp[-E_\mu/kT]$ correlates with the change of the dislocation density (see Figs 7, 8). However, E_μ

does not depend on the density. The cause of the exponential $\mu(T)$ dependence in the region $T < 20°K$ is not clear. It is difficult to attribute this dependence to screening of the scattering centers, for at $T < 20°K$ we have $r_D \gg R_D$, where r_D is the Debye screening radius and R_D is the average distance between the dislocations. This question calls for additional research.

Let us examine the behavior of the crystals in the region $T > 20°K$. Consider first the mobility μ. As seen from Figs. 7 and 8, in the region $T = 40$ to $50°K$ there is a small minimum whose position and shape change with increasing dislocation density. The causes of this minimum are not fully clear, but we wish to note that its position on the temperature scale practically coincides with the temperature of the proposed magnetic phase transition in the dislocation spin system of the broken bonds.[6] This allows us to assume that the minimum is caused by the anomalous spin scattering of the electrons by the dislocations in the vicinity of the magnetic phase transition. It should be noted here that the influence of the plastic deformation of the silicon crystals on the carrier mobility calls for independent investigations, since the observed anomalies (see also the paper by Milevskiĭ and Zolotukhin[12]) are worthy of attention and have not yet been completely interpreted.

As already indicated, the dislocations in n-type silicon act as acceptors (see Figs. 4 and 5). This is confirmed by numerous results obtained for germanium, and also obtained earlier for silicon by the EPR method.[8] What is remarkable is the fact that the compensation coefficient K depends little on the temperature: the latter indicates that the dislocation acceptor levels in the investigated crystals lie deeply in the forbidden band, and therefore to determine their positions research must be carried out at high temperatures.[13] The behavior of the log n(1/t) curve in the region $T > 20°K$, namely, the flattening of the curve, can be easily attributed to the onset of the hopping mechanism of conductivity (along impurities or dislocations), and is not connected with an increase of the free-electron concentration in the conduction band.

CONCLUSION

The experimental results obtained here for the $T < 20°K$ region cannot be explained in principle, from our point of view, as being due only to the influence of dislocations on the hopping conductivity along the impurities, without introducing the concept of dislocation conductivity.

We suggest therefore that in the general case we have $\sigma = \sigma_{free} + \sigma_d + \sigma_D$, where σ_{free} is the conductivity due to the free electrons, σ_d is the conductivity of the electrons localized on the donor impurity, and σ_D is the conductivity of the electrons on the dislocations. In the entire temperature region the Hall effect is determined by the free electrons, so that $R \approx R_{free} \gg R_d, R_D$. In strongly deformed crystals in the region $T > 20°K$ we have $\sigma \approx \sigma_D$, since $\sigma_D \gg \sigma_{free}$ and $\sigma_D \gg \sigma_d$, i.e., the crystal conductivity is determined entirely by the dislocations and increases with increasing density of the latter. In the region $T > 20°K$ we have $\sigma \approx \sigma_{free}$, and the conductivity of the crystals decreases with increasing dislocation density because of the following: 1) the acceptor action of the dislocations and 2) the scattering of the free electrons by the dislocations. The Hall mobility $\mu = R\sigma$ of the free electrons decreases in the entire temperature region with increasing dislocation density, and the $\mu(1/T)$ curves then have two anomalies: 1) in the region $T < 20°K$ (where $\mu \sim \exp[-E_\mu/kT]$) and 2) in the region $T = 40$ to $50°K$ (where there is a small minimum). It is assumed that the minimum is connected with the anomalous spin scattering by dislocations.

We note in conclusion that investigations of germanium crystals with dislocations have enabled Osip'yan and Shevchenko[14] to conclude that conductivity along dislocations exists also in germanium crystals. This gives grounds for hoping that the phenomenon of conductivity along dislocations is general in character and should be observed also in other semiconducting crystals.

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230