The dependence of the electrical properties of silicon on the plastic deformation and annealing temperatures

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The influence of dislocations on the spectrum of electronic states in silicon single crystals has been investigated. It was found that the characteristics of the dislocation p - n junctions, the photoconductivity spectrum, and the type and concentration of the majority carriers in samples with the same dislocation density depend in a fundamental way on the temperature and duration of plastic deformation or on the subsequent heat treatment. It was shown that the results cannot be explained within the framework of traditional ideas and the existing theories, which only take account of the interaction of the broken bonds in the dislocation core, and of the microstress field of the dislocation, with elementary excitations of the electron subsystem of the crystal. The possible causes of the observed effects were analyzed, and their dependence on the impurity concentration was studied. Direct evidence was obtained of the need to take account of indirect mechanisms by which dislocations influence the carrier-energy spectrum; these are due to the formation of new centers when dislocations interact with point defects.

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The problem of the influence of dislocations on the electrical properties of semiconductors has attracted ever increasing attention from researchers for more than twenty years.^[1-5] This interest is stimulated not only by the possibilities of altering the spectrum of electronic states in a crystal appreciably by plastically deforming it, but also by the prospects of using dislocations to study the characteristics of the electrical and magnetic phenomena occurring in one-dimensional systems.^[6-8] In the overwhelming majority of studies carried out to date the effects observed have been explained on the basis of an analysis of the possible changes in the carrierenergy spectrum in a peculiar distortion field. The character of the microdistortions in the dislocation cores was only taken into account in terms of the existence of broken bonds at the edge of the extra half-plane, and the compressive stresses were assumed equal to those calculated from the known value of the Burgers vector in the framework of the theory of elasticity. It was assumed that dislocations did not interact with other defects in the crystal lattice (impurities, vacancies, microsegregations of new phases), and that the change recorded in the carrier density was due in the main to their capture by the broken bonds at dislocation cores.

In the general case, however, this assumption is incorrect. A dislocation cannot exist in isolation not only from the elementary excitations in the electron subsystem of the crystal, but also from the broad range of structural defects which arise under the influence of the thermal fluctuations and nonideal conditions of the growth, of the processing and even of the storage of the sample. This means that along with the discussed direct effect of dislocations on the carrier-energy spectrum, various indirect-action mechanisms must manifest themselves; this indirect action arises from a change in the state of any defects capable of capturing or releasing electrons in the microstress fields of dislocations. This makes it necessary to take account of the dislocation-stimulated redistribution and charge exchange of existing centers in the crystal, of the formation of new complexes, and

of the consequences of possible changes in the dislocation distortion fields themselves (partial stress relaxation, splitting of dislocations with the formation of stacking faults, etc.).

The observed change in the spectrum of electronic states as a function of the conditions in which dislocations are introduced into the sample and of its subsequent processing will be influenced by these indirect defects to varying degrees. In some cases they may even completely mask the contribution of the proper deformation and electrostatic potentials of the dislocations. Indeed, in a number of $papers^{[9-12]}$ it was found that the influence of dislocations on the electrical properties of silicon depends on the temperature at which the crystal is deformed and annealed, and on the method of cooling it, and this cannot be understood in the framework of the existing theories of the direct influence of dislocations on the carrier-energy spectrum.

To explain the physical nature of the processes that determines the electrical activity of dislocations in a real crystal, and to create the conditions for a more distinct separation of the manifestations of the qualitatively different mechanisms noted, it was essential to carry out a detailed study of the dependence of the electrical properties of plastically deformed samples on the deformation and annealing temperatures and on the impurity content of the crystal. In the present paper the results of such experiments on single crystals of silicon are described. The carrier-energy spectrum was analyzed using the results of measurements of the Hall effect, the electric conductivity, the photoconductivity, and the characteristics of the dislocation p-n junctions.

PROCEDURE

In crystals containing few dislocations, in which the cylinders screening the dislocation charge do not overlap, the positions of the dislocation levels are determined to an appreciable extent by the electrostatic interaction of the trapped electrons. Allowing for the effect of this interaction is quite complicated and can be done in various approximations,^[1] and this may be responsible, albeit partially, for the contradictory results obtained by different authors. To eliminate this possibility of treating the experimental data ambiguously, we have carried out our investigations on silicon samples with a high dislocation density in which the Read cylinders overlap and a situation is easily achieved in which the interaction of the carriers captured by a dislocation can be neglected. In this case no knowledge is required of the dependence of their interaction energy on the degree of filling of the dislocation levels, and the position of shallower unfilled levels can be determined directly from the temperature dependence of the carrier density. Moreover, for the situation described it is possible to effect a comparison with the data from a more direct method of investigating unpaired electrons, viz., electron paramagnetic resonance, [6,10] as the sensitivity of the latter is only adequate for studying samples with a high dislocation density.

To obtain fuller information on all the levels, including deep ones, which arise when silicon is deformed, use was made of measurements of photoconductivity^[13] and the characteristics of the dislocation p-n junctions^[111] in addition to the methods of investigation traditionally employed (electric conductivity and Hall effect).

The initial, dislocation-free silicon ingots were doped with phosphorus or boron. Dislocations were introduced into the crystal by plastically deforming it in the creep mode at stresses of $10-14 \text{ kgf/mm}^2$ or with a constant loading rate of 20 μ m/min. The samples, in the form of prisms $8 \times 3 \times 3$ mm in size, were compressed 4-5%along the long $\langle 110 \, \rangle$ axis at temperatures of 650–700 $^\circ C$ in the first case and at 700-900 °C in the second. Deformation took place for 10-30 minutes, after which the load was removed from the crystals and they were taken out of the furnace. The average density of dislocations found by electron microscopy in these samples was (2- $6) \times 10^9$ cm⁻². Annealing was carried out in the temperature range 500-1000 $^\circ C$ in quartz ampules in a vacuum of less than 5×10^{-5} mm Hg. The heat treatment conditions employed did not change the electrical properties of undeformed control crystals significantly. After deformation and annealing the samples were cooled to room temperature in a time of several minutes. None of the effects caused by quenching^[12] were observed when this was done.

Dislocation p-n junctions were produced by locally deforming *n*-Si by pressing a four-face sapphire indentor into the {111} surface under a load of 2 kg at temperatures of 300-1000 °C or by three-point bending.^[11]

The electrical contacts were made by electrostatic welding using gold wire 0.1 mm in diameter. After each treatment the crystals were chemically polished in a mixture of HF and HNO₃ (1:7). The measurements of the electric conductivity and of the Hall effect were carried out in the temperature range 80-480 K and magnetic fields of 3-10 kOe with direct current, using an electrometer. The Hall factor was taken as equal to 1. The current-voltage characteristics of the dislocation p-n junctions were plotted point by point.

To study the photoconductivity, plates $3 \times 3 \times 0.4$ mm in size were cut from n-Si crystals deformed by compression. Indium contacts were attached to the large faces on both sides. A linear and a quadratic portion were observed in the current-voltage characteristics of these samples, corresponding to a space-charge-limited current flow.^[14] Measurement of the photoconductivity was carried out in the region where the current depends linearly on voltage, i.e., in conditions of low-level injection from the contacts. The sample was placed in a cryostat with a CaF2 window, cooled to 80 K, and illuminated from an end-face by a type SPM-2 mirror monochromator with an LiF prism. The light source was a globar. The photocurrent was read from a 10 m Ω resistor in series with the sample. At the used light intensity, the lux-ampere characteristic was practically linear.

EXPERIMENTAL RESULTS

1. The investigation of the influence of annealing on the energy spectrum of levels associated with dislocations was carried out on n- and p-Si single crystals deformed at temperatures for which the existence of broken bonds can be confirmed by the EPR method with certainty (650-700 °C).

The conductivity type of *n*-Si samples with a chemical donor density of $N_d = 4 \times 10^{13}$ cm⁻³ underwent inversion as a result of deformation. The temperature dependence of the hole density in them was described by the expression $p \propto \exp(\Delta_1/kT)$, where $\Delta_1 = 0.44 \pm 0.04$ eV (Fig. 1, curve 1), i.e., the shallowest unfilled level in these samples is $E_1 = E_v + 0.44$ eV. The most effective generation-recombination centers (gr-centers), which determine the temperature dependence of the reverse current of the dislocation p-n junctions formed at the boundary of the deformed and dislocation-free regions, ^[111] have a level situated at the same distance from the top of the valence band E_v .

In investigating photoconductivity in these samples we succeeded in finding one more level situated at a distance of 0.27 eV from one of the bands. Figure 2 (curve 1) shows the spectral dependence of the photoconductivity normalized to the number of quanta of incident light. The red photoconductivity threshold is situated at $h\nu$

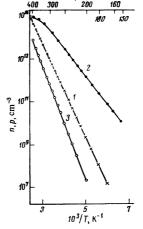


FIG. 1. The influence of annealing on the charge-carrier density in phosphorus-doped ($N_d = 4 \times 10^{13}$ cm⁻³) silicon crystals plastically deformed at 700 °C: 1—after deformation (*p*-type); 2, 3—after annealing for 60 minutes at 750 °C (*p*-type) and 950 °C (*n*-type) respectively.

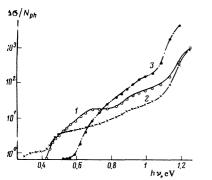


FIG. 2. The spectral dependence of the photoconductivity of Si samples deformed at 700 °C (curve 1) and of the change in it after on subsequently annealing for 60 minutes: 2—at 750 °C; 3—at 850 °C. The initial crystals were doped with phosphorus $(N_d = 4 \times 10^{13} \text{ cm}^{-3})$.

≈0.44 eV. Comparison with the results presented in Fig. 1 shows that in this spectral region the photoconductivity is due to the transition of electrons from the valence band to the level E_1 . The presence of a second level is evident from the increase in photoconductivity at $h\nu \ge 0.85$ eV. Since a level at $E_2 = E_{\nu} + 0.27$ eV is observed under some conditions in deformed silicon,^[11, 15, 16] the increase in photocurrent observed at $h\nu \ge 0.85$ eV can be linked with the transition of electrons from the filled E_2 level to the conduction band.

Study of plastically deformed *p*-type crystals reveals not only a more extensive set of acceptor levels, but also donor ones. In samples with an excess boron content $N_a = 3 \times 10^{12}$ cm⁻³ the temperature dependence curves of the hole density in the deformed and starting material intersect at $T_0 \approx 290$ K (Fig. 3, curves 1 and 1'). At T $>T_0$ the hole density increases as the temperature rises and the trend of the p(T) curve is determined by the acceptor level $E_1 = E_v + 0.44$ eV. At $T < T_0$ donor action is evident (the hole density decreases). The slope of the p(T) curve does not change on passing through $T = T_0$. This attests to the presence either of two closely spaced donor and acceptor levels (or of a one-dimensional dislocation band) in silicon at a distance of 0.44 eV from the top of the valence band, or to the fact that the donor level is higher than E_1 . The latter supposition must

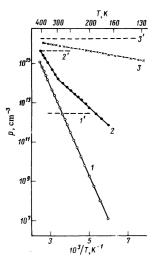


FIG. 3. The temperature dependence of the hole density in crystals plastically deformed at 700 °C (curves 1-3) and in the initial boron-doped crystals (curves 1'-3'): 1, 1' $-N_a = 3$ ×10¹² cm⁻³; 2, 2' -5×10^{15} cm⁻³; 3, 3' -2×10^{16} cm⁻³. also not be excluded since the centers may be of a different kind. Donor-level electrons fill the $E_2 = E_v + 0.27$ eV acceptor level in lightly doped crystals, and the electrical properties of such samples are determined by the thermally activated ejection of electrons from the valence band to the level E_1 .

When the density of chemical acceptors in the original silicon is increased, electrons fill the shallower boron acceptor level, and in this process the level E_2 is partially emptied and begins to manifest itself in the temperature dependence of the hole density (Fig. 3, curve 2 for T > 290 K). Even shallower levels, e.g., $E_v + 0.17$ eV and others, which arise during deformation, are observed in heavily doped crystals.

Comparison of the results presented in Fig. 3 (curves 3 and 3') shows that the total donor density introduced by deformation exceeds 10^{16} cm⁻³. Similar values are obtained by estimating the density of centers with the E_1 and E_2 levels from the experimental data (Figs. 1 and 3) using the formula for a compensated semiconductor.^[17]

Figure 4 shows the temperature dependence of the hole mobility in deformed crystals. It is evident that the carrier mobility decreases as a result of deformation, and that it is at the same time weakly dependent on the carrier density in the initial sample. The latter result seems quite strange since in the studied crystals with different dopant contents the filling coefficient f varied in the range $0 \le |f| \le 0.2$, and according to the existing concepts^[18] the scattering of carriers by a charged dislocation is proportional to the square of its charge, i.e., to $|f|^2$.

2. Isochronous 60-minute annealing of crystals deformed at 700 °C changed the carrier energy spectrum radically. Thus, for example, keeping samples at T = 750 °C led to an increase in the hole density in all the types of crystal investigated. In these circumstances the slope of the p(T) curve in Si samples doped with phosphorus was observed to decrease, and it began to be determined by the level $E_2 = E_v + 0.27$ (Fig. 1, curve 2). The red photoconductivity threshold in such samples also shifted into the short-wave region (Fig. 2, curve 2).

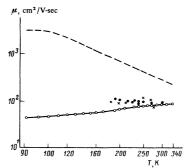


FIG. 4. The temperature dependence of the hole mobility in boron-doped (o: $N_a = 2 \times 10^{16} \text{ cm}^{-3}$; •: $N_a = 3 \times 10^{12} \text{ cm}^{-3}$) or phosphosphorus-doped (×: $N_d = 4 \times 10^{13} \text{ cm}^{-3}$) silicon crystals deformed at 700 °C. The broken line shows the change in mobility in undeformed *p*-Si crystals with a boron concentration of N_a = $2 \times 10^{16} \text{ cm}^{-3}$ with temperature.

On raising the annealing temperature T_{ann} further, the crystals began to regain their properties. In samples heavily doped with boron ($N_a = 5 \times 10^{15} - 2 \times 10^{16}$ cm⁻³) the hole density increased and at $T_{ann} \approx 850-900$ °C it returned to its initial value. At the same time the hole mobility increased somewhat, but remained lower than in the initial samples.

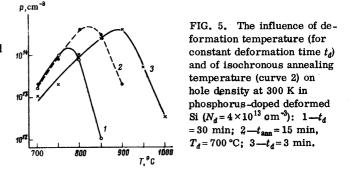
In crystals doped with phosphorus $(N_d = 4 \times 10^{13} \text{ cm}^{-3})$ the hole density reached a maximum at $T_{\text{man}} = 750$ °C, and on raising the annealing temperature further it began to fall, while the slope of the p(T) curve increased. At $T_{\text{man}} \ge 850$ °C the conductivity type of the samples was restored. However, after annealing at 1000 °C the electron density in such crystals varied with temperature according to $n \propto \exp(\Delta_3 / kT)$, where $\Delta_3 = 0.52$ eV. Centers with the same level were also revealed in the photoconductivity study (Fig. 2, curve 3). It should be noted that heat treatment of samples with a high phosphorus content ($N_d = 10^{15} \text{ cm}^{-3}$) at 1000 °C also virtually restored the carrier density.

Annealing at temperatures below the deformation temperature hardly changed the electrical properties of deformed crystals.

3. Variation of the plastic deformation temperature also causes appreciable changes in the spectrum of electron states associated with dislocations. The nature of these changes (Fig. 5) is analogous to that observed for annealing. It should be noted that the method of obtaining a specified dislocation density (deformation under creep conditions or at constant speed) had no influence on the electrical properties of the deformed samples.

4. The duration of the heat treatment had as decisive an effect on the energy spectrum of a crystal as a change in the temperature (Fig. 5); to obtain the same effect by plastic deformation or annealing of a sample deformed at a lower temperature it was necessary for the duration of both processes to be the same. Thus, in crystals doped with phosphorus the p(T) dependence determined by the E_2 level can be obtained not only by annealing, but also by deformation at 750 °C for 40 minutes or at 800 °C for 10 minutes. If deformation was effected in one or two minutes as, for example, by applying a concentrated load, inversion of the conductivity type could be achieved even at 1000 °C. On the other hand, the inversion disappeared even at 800 °C if the deformed sample was annealed for 5 hours.

When the deformation temperature was increased (T_d >1000 °C) inversion of the conductivity type was not observed in *n*-Si samples even after rapid loading with an indentor, and the diode effect was absent. Investigations over a wide temperature range showed that the latter disappears also at $T_d < 500$ °C. This may be due to the shunting action of intermediate layers of a new phase that appears in Si under the action of a concentrated load, ^[10] since the diode effect^[20] was preserved at isolated dislocations introduced at $T_d < 500$ °C. To elucidate the cause of this "low-temperature" anomaly, additional studies are needed on samples homogeneously deformed to high dislocation densities (~10⁹ cm⁻²) at temperatures near the transition to the brittle state. It has



not yet been possible to do such experiments. They require too great a plastic deformation time at stresses often resulting in destruction.

To conclude this section we note that by varying the deformation temperature it is possible to alter the dependence of the reverse current I_r both on temperature (for $T_d > 850$ °C the reverse current is determined by the gr-centers with the level $E_3 = E_c - 0.52$ eV), and on voltage. A smooth junction $(I_r \propto U^{1/3})$ is formed with $T \approx 800-850$ °C, and a sharp one $(I_r \propto U^{1/2})$ at the remaining temperatures of the deformation that causes inversion of the conductivity type in *n*-Si.

DISCUSSION OF RESULTS

According to the generally accepted views a whole series of levels (or even bands) due to the broken bonds in the core or the distortion field surrounding the core may be associated with dislocations.^[2] If the experimental data obtained for only one deformation temperature (700 °C) are examined, then qualitative agreement with these concepts can be perceived. Indeed, the study of crystals (differing in carrier density in their initial state) by several methods that give information both about filled and about partly empty local levels has shown that plastic deformation leads to the appearance of a whole series of donor and acceptor levels in the forbidden band, most of which have already been mentioned disjointedly in a number of papers.^[11,21-23] Since the EPR spectrum of Si crystals deformed in conditions similar to ours reveals evidence of the existence of paramagnetic centers associated with the uncompensated spins of "dangling" bonds, ^[6] some of the levels found may belong to these centers. It is only the very high density of all the centers discovered in the experiments which is surprising.

However, it is impossible to explain the experimental data on the influence of the temperature and duration of deformation and annealing solely on the basis of the influence of dislocations on the carrier-energy spectrum. Electron microscope studies have shown that at the used heat treatment times the character of the dislocation structure and the dislocation density in the samples hardly changes. The possible slight decrease in the average dislocation density (by 2–3 times) cannot possibly decrease the number of broken bonds by two orders of magnitude during annealing at 800–1000 $^{\circ}$ C, and is even less likely to increase the hole density as a result

of heat treatment at 750-800 °C. It should be noted that the latter effect is also not in accord with the decrease in the density of uncompensated spins in the dislocation core by annealing, as observed in studies of the EPR signal.^[6,10]

The observed dependence of the electrical properties of deformed silicon on the temperature and time of deformation can be explained in principle by taking account of the interaction of dislocations with other defects in the crystal lattice, or attributed to a change in the structure of the dislocation core through splitting (or contraction) above a certain critical temperature.

If the second of these possibilities were to occur, then it would inevitably be manifest in studies of the mobility of individual dislocations, i.e., in the characteristic which is perhaps the most sensitive to the state of the fine structure of the dislocation cores. In that case an abrupt change would be observed in the activation energy of the dislocation motion on increasing the deformation temperature. Moreover, as follows from the observed dependence of the electrical properties on the deformation time, the velocity of the dislocation would depend quite strongly and in a unique way on the path traversed. However, no such effects have been observed in studies of the mobility of dislocations.^[24,25] It therefore seems improbable that the effects under discussion are due to dislocation splitting.

On the other hand, allowance for the indirect influence of the dislocations on the carrier-energy spectrum in semiconductors because of their interaction with point defects permits a consistent qualitative interpretation of many phenomena not described by the traditional concepts that only take account of the direct action of the dislocations. Even in crystals obtained by modern growing techniques, with an infinitesimally low content $(10^{11}-10^{12} \text{ cm}^{-3})$ of electrically active impurities, the concentration of oxygen (and apparently, of carbon) varies from 10^{15} to 10^{17} cm⁻³. It is easier for all impurities and inherent point defects to diffuse to dislocations when displaced and subjected to high-temperature heat treatment. When this happens, a region with a high concentration of impurity atoms is formed near the dislocation; these may form complexes with one another or with the Si atoms at the edge of the extra half-plane in the dislocation core. As a result new electronic states arise and the density of the dislocation centers themselves decreases because of neutralization of the broken bonds and the partial relaxation of the compressive stresses. Evidence that such processes occurred in the experiments described above is provided by the strong dependence of the equalization of the contribution of the centers producing deep levels to the electric conductivity on the high-temperature heat-treatment time. As the annealing temperature and time are increased, the diffusion processes are accelerated and this speeds up the changes in the carrier-energy spectrum. Evidence of the appreciable influence of impurity atoms on the energy spectrum of plastically deformed crystals is provided by the fact that the nature of the p(T) dependence is determined not only by the conditions of deformation or annealing, but also by the content of electrically inactive impurities in the initial samples. Thus, the changes in the electrical properties of deformed n-Si crystals with the same phosphorus content, but with different oxygen concentrations, during annealing are not identical. In particular, after annealing at 750 °C the slope of the p(T) curve for crystals with low oxygen content (grown by skull melting) does not change.

Our investigations showed that the heat treatment also has an appreciable influence on the magnitude of the starting stresses for dislocation movement. On raising the annealing temperature, just as on increasing the concentration of dopant, they may increase by more than an order. This effect is the obvious result of increasing the number of barriers associated with point defects in the path of an expanding double kink. Moreover, electron microscope studies of plastically deformed Si crystals with different dopant contents (Sb, Al, Ga) have enabled direct evidence to be obtained of the enrichment of a moving dislocation by point defects (Fig. 6). In moving through a crystal, a dislocation collects the latter from quite a significant volume adjacent to the slip plane. When it intersects the surface, channel diffusion, which may exceed volume diffusion by many orders, transports some of them to the bottom of the oxide film, where they become fixed and lead to the appearance of tracks behind a moving dislocation. In weakly doped crystals the latter are seen as a thin, continuous blackand-white line,^[26] but the concentration of point defects on the surface is insufficient for stable segregation of a new phase, and the tracks are rapidly resorbed on irradiation by the electron beam in the microscope. On the other hand, in strongly doped crystals the concentration of impurities is sufficient for the formation of a stable phase, and a chain of discrete point segregations is left behind the dislocation (Fig. 6).

The most striking evidence, in our view, for the reality of the mechanism under discussion is revealed in the results of the following experiment. Copper impurity atoms, which give rise to deep acceptor and donor levels in a sample in the lower half of the forbidden band^[27] were specially introduced into *n*-Si single crystals by diffusion. The copper concentration reached 10^{17} cm⁻³, and during this process the conductivity type of the *n*-Si underwent inversion. As a result of subsequent deformation at 650-800 °C the Si crystal again acquired *n*type conductivity. Since doping with copper and introducing dislocations spearately invert the conductivity type of *n*-Si, it must be assumed that in the presence of dis-



FIG. 6. Tracks behind dislocations in a heavily doped Si crystal ($N_{\rm Sb}=5\times10^{18}~{\rm cm}^{-3}$); $T_d=700~{\rm ^{\circ}C}$.

locations copper atoms form donor centers with other impurities or with broken bonds. This experiment provides unambiguous evidence that even at relatively low deformation temperatures the interaction of dislocations with impurities present in a concentration typical of pure Si crystals appreciably alters the charge-carrier energy spectrum of deformed crystals.

When complexes involving impurity atoms are formed near a dislocation, a certain delocalization ("smearing") of the dislocation charge may be expected, and this decreases the energy of interaction between carriers of like charges trapped at the dislocation. This permits one to explain the anomalous behavior of potential around a dislocation found in studies of the diode effect at a single dislocation^[20] and the high filling coefficient observed by us (0.4) in Si crystals deformed at 700 °C with a dislocation density of 3×10^{6} cm⁻³ and also in^[28]. The existence of a large number of centers that contain impurity atoms near a dislocation also enables one to understand the weak dependence of carrier mobility on the dislocation charge, since in that case scattering is due to the cloud of charged complexes near the dislocation.

CONCLUSION

Consequently, the experiments presented provide convincing evidence that the influence of dislocations on the electrical properties of Si is determined not only by their direct interaction with carriers. An appreciable contribution is made by the change in the state of point defects in the dislocation microstress field. Only by taking account of the relative role of these processes can a qualitative explanation be given of the dependence of the energy spectrum of crystals with a high dislocation density on the temperature and time of deformation or annealing of the samples, and also on the method by which they are cooled.^[12] This fact opens up further prospects for bringing about the necessary changes in the properties of semiconductor material with a view to creating devices out of it through mechanical and heat treatment, and it also enhances the possibilities of studying the characteristics of processes occurring in one-dimensional systems using dislocations as an example.

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