Features of the Electron Mobility in the n–InSe Layered Semiconductor

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ELECTRONIC PROPERTIES
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Features of the Electron Mobility in the *n*-InSe Layered Semiconductor

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Abstract—The dependences of the Hall electron mobility of *n*-InSe single crystals grown by the Bridgman method on a sample's technological history, temperature, electric field, doping, and illumination are experimentally investigated. It is established that at temperatures below room temperature, the dependences of the electron mobility on external factors, initial resistivity, and doping are anomalous, i.e., do not obey the theory of free carrier mobility in quasi-ordered crystalline semiconductors. The observed anomalies are attributed to partial disordering and fluctuation of the potential of free energy bands of the *n*-InSe single crystals and can be controlled by temperature, electric field, doping, and illumination.

1. INTRODUCTION

The layered semiconductor *n*-InSe has been known for a long time [1]. Due to its extraordinary crystal structure, unique electronic properties, surface stability against environmental conditions, appropriate band gap, and high electron mobility, single crystals of this material are highly promising for designing ultrathin-film devices and, therefore, generate interest among researchers working in different fields [2–13].

Pioneering studies on the electrical conductivity of this semiconductor appeared more than sixty years ago [14]. However, the carrier (electron) mobility in its single crystals has been understudied and the available works on this subject [15–18] often contain contradictory data and random dependences of these data on the synthesis methods used, fabrication techniques, prehistory, and conditions for long-term storage of investigated samples. In view of the great importance of the value and features of the free carrier mobility in a semiconductor for its fundamental research and application, in this work we experimentally explore the dependences of electron mobility in *n*-InSe single crystals on their technological origin and doping of the investigated samples, temperature (*T*), electric field (*E*), and the effect of light.

It is not excluded that the results obtained can be used not only to establish new properties of indium monoselinide, but also to elucidate the mechanisms of various electronic phenomena in other layered III–VI semiconductor compounds.

2. EXPERIMENTAL

The experimental measurements were performed on pure (not specially doped) *n*-InSe single-crystal samples and samples weakly doped ($N < 10^{-1}$ at %) with gadolinium (Gd), dysprosium (Dy), and holmium (Ho).

The above-mentioned chemical elements (lanthanides) were chosen as dopants from the following considerations. First, to date, there have been a great number of studies on different *n*-InSe doped with lanthanides [19–25], the results of which can help explain our experimental data. Second, the physicochemical properties [26–28] (electronic configuration, chemical activity, stability in air, melting point, magnetic properties, atomic and ionic radii, and electrochemical similarity to In) of these lanthanides make them the most suitable materials for use as ligators for InSe. Third, as was shown in earlier works [20, 23, 25], in InSe single crystals weakly doped ($N \leq 10^{-1}$ at %) with Gd, Dy, and Ho, the photoelectric, luminescent, and electrical characteristics can be purposefully controlled by changing the introduced impurity content *N*.

The InSe compound was synthesized by the co-melting of initial components, In (99.999%) and granular Se (99.999%), taken in the stoichiometric ratio. Doping was performed in two stages. At the first stage, before synthesis, a ground ligator (Gd, Dy, or Ho) was added in an oxygen-free charge placed in a special vacuum mini-chamber in the required amount to obtain doped InSe with the addition of 1 at % of impurity.

Table 1. Electronic parameters of the investigated samples

T, K	Sample no.	R_H, Kl^{-1} cm^{-3}	n, cm^{-3}	μ, cm^2/(V s)	σ, Ω^{-1} cm^{-1}	ρ_0, Ω cm
77	1	1.9×10^5	3.3×10^{13}	1.2×10^{-1}	6.0×10^{-7}	1.7×10^6
	2	1.9×10^5	3.3×10^{13}	1.8×10^2	6.0×10^{-4}	1.7×10^3
	3	1.9×10^5	3.3×10^{13}	9.0×10^2	2.9×10^{-3}	3.4×10^2
300	1	1.8×10^5	3.4×10^{13}	8.7×10^2	3.0×10^{-3}	3.3×10^2
	2	1.8×10^5	3.4×10^{13}	8.7×10^2	3.0×10^{-3}	3.3×10^2
	3	1.9×10^5	3.3×10^{13}	8.7×10^2	2.9×10^{-3}	3.4×10^2

At the second stage, to obtain samples with = lower impurity contents ($N = 10^{-5}$, 10^{-4}, 10^{-3}, 10^{-2}, and 10^{-1} at %), the substance obtained at the first stage was co-melted with the corresponding amount of pure InSe. This method allowed us, first, to obtain doped single crystals with a more accurate atomic percentage of the introduced impurity at $N \leq 10^{-1}$ at % and, second, to prevent additional contamination of the product. To avoid the formation of selenides of the ligators used and ensure a uniform impurity distribution over the ingot volume throughout the entire synthesis process (~14 h), the loading temperature was kept strictly within 960−1000 K and, at $T \geq 900$ K, the loaded ampoule was continuously shaken with a special mechanical vibrator.

The investigated single crystals of both (pure and doped) groups of materials were grown by the modified Bridgman method (slow cooling at a constant temperature gradient along the ingot [29]) according to the technological regime described in [30]. Analysis of the state diagrams of the impurities used with In and Se showed that at the temperatures ($T \approx 960−1000$ K) of synthesis and growth of the InSe crystals in the investigated amount ($N \leq 1.0$ at %), they easily dissolve in this substance and do not form compounds with Se [31, 32].

The structure and phase and elemental compositions of the obtained ingots, as well as the surface state along the plane of natural sample layers were determined via comprehensive X-ray diffraction, thermographic, X-ray spectroscopy, and microscopy investigations. With this aim, modern and rather reliable measuring facilities (DSC-910, ADVANCE-8D, SINTECP 21, and DRON-4-07, CuK_α radiation, a step of 0.050 in the angle range of 8°−135° and a Zeiss SEM with an energy-dispersive analyzer) were used.

It was established that the obtained materials of both groups are homogeneous and have to a great extent a single-crystal structure; their X-ray diffraction patterns are identified in the rhombohedral syngony (γ polytype) and belong to the R3m (C_{3v}^5) space group [33−35] with lattice constants of $a \approx 4.003$ Å and $c \approx 24.955$ Å; no selenide, oxide, or free lanthanide substitution phases were observed and no inho-

mogeneities or foreign phases were found on the surface.

The investigated samples were cleaved from different parts of the same ingot and from different single-crystal ingots. This mainly determined the difference between their technological origin, which was quantitatively estimated from the resistivity ρ_0 (at 77 K) or conductivity σ_0. The sample conductivity type was determined from the thermoelectric power and Hall coefficient signs and was found to be electronic at all experimental conditions.

Samples for studying the $I−V$ characteristics and Hall measurements were prepared. Due to the layered structure of the investigated material, its individual samples were easily obtained by cleaving from large single-crystal ingots and had an atomically specular (low-roughness) and chemically stable surface [36]. Therefore, there was no need for their additional processing. Samples of both groups had the form of plane-parallel wafers. In the first group of samples, the current flowed perpendicular to the layers and their lateral (along the natural crystal-layer plane) sizes and thickness (in the direction perpendicular to the natural crystal layers) were (3.00−5.00) × (3.00−5.00) mm and $d \leq 0.300$ mm, respectively. The length (distance between the current contacts) and lateral sizes (the thickness perpendicular to the natural crystal layers and the distance between the Hall contacts located in the plane of the natural crystal layers) were 6−8 mm and (0.300−1.00) × (2−3) mm, respectively. At the same time, both the current and probe contacts covered the entire thickness (all layers) of the sample under study.

In contrast to the free electron densities n or Hall coefficient R_H, the conductivity σ or resistivity ρ and electron mobility μ at 77 K for different samples significantly differed, depending on their technological origin. As the temperature increased, this difference decreased and, at $T \approx 300$ K, completely vanished (see Table 1).

The measurements were performed in wide ranges of temperature T, wavelength λ, and intensity Φ of monochromatic light incident onto the investigated sample (at $T = 77−500$ K, $\lambda = 0.30−3.00$ μm and

$\Phi \leq 5 \times 10^2$ L, respectively) in electric fields, from the weakest value to ~2.4×10^3 V/cm. The Hall measurements were performed by the conventional three-probe method in ac electric and magnetic fields. The current was directed along the sample layers and a magnetic field of 5×10^2–2×10^3 Oe, perpendicular to the natural crystal layers. Sometimes, the σ, n, and μ values were also estimated using the formulas of Lampert theory developed for space charge-limited currents [37]. Before each measurement, the samples were subjected to a special thermal procedure, which ensured the complete elimination of all possible sample history effects [38, 39] on the measurement results. For 1.5–2 years, no dependence of the μ value and characteristics on the time of storage in dry air at room temperature (300 K) was detected.

3. EXPERIMENTAL RESULTS

It was established that at 300 K in weak electric fields the σ, n, and μ values for the investigated n-InSe samples of both groups are almost identical, regardless of the fabrication technique used (Table 1). As the temperature decreases to the liquid-nitrogen boiling point (77 K), the n (R_H) value remains almost invariable and does not differ for different samples. In contrast to the n (R_H) value, the σ (ρ) and μ values in the high-resistance (with $\sigma_0 \leq 10^{-4}$ Ω^{-1} cm^{-1}) crystals change with temperature in an activation way, i.e., according to the law σ, $\mu \sim \exp(-\Delta\varepsilon/kT)$ (Fig. 1, curves 6 and 7). The latter indicates that the low-temperature $\sigma(T)$ dependence of the investigated n-InSe crystals has a drift character, i.e., is directly related to the $\mu(T)$ dependence and not caused by the $n(T)$ dependence. However, the experimental $\mu(T)$ dependence cannot be described by the existing theory of free carrier mobility in quasi-ordered crystalline semiconductors [40]. In addition, in the low-temperature region, features of the $\mu_0(\rho_0)$ dependences (Fig. 2, curves 1–3) and $\mu(N)$ dependences (Fig. 2, curves 4–6) and the dependences of μ on the effect of light (Fig. 4), which are atypical of quasi-ordered crystalline semiconductors, were observed. In particular, the μ value at 77 K in the dark in low-resistance ($\sigma_0 \geq 10^{-3}$ Ω^{-1} cm^{-1}) crystals barely differs from the value at 300 K and amounts to (850–1000) cm^2/(V s) and, with a decrease in the σ_0 value to 6×10^{-7} Ω^{-1} cm^{-1}, drops to fractions of units (down to 1.2×10^{-1} cm^2/(V s)).

In contrast to the low-temperature case ($T < 260$–280 K), in the high-temperature region ($T > 300$ K), the $\mu(T)$ dependence obeys the regularity characteristic of quasi-ordered crystalline semiconductors upon the dominance of free carrier scattering at acoustic lattice vibrations [40]. More accurately, in this case, instead of the exponential $\mu(T)$ dependence, we observe the dependence $\mu \sim T^{3/2}$ (Fig. 1, curves 5–7). At all other conditions being equal, with an increase
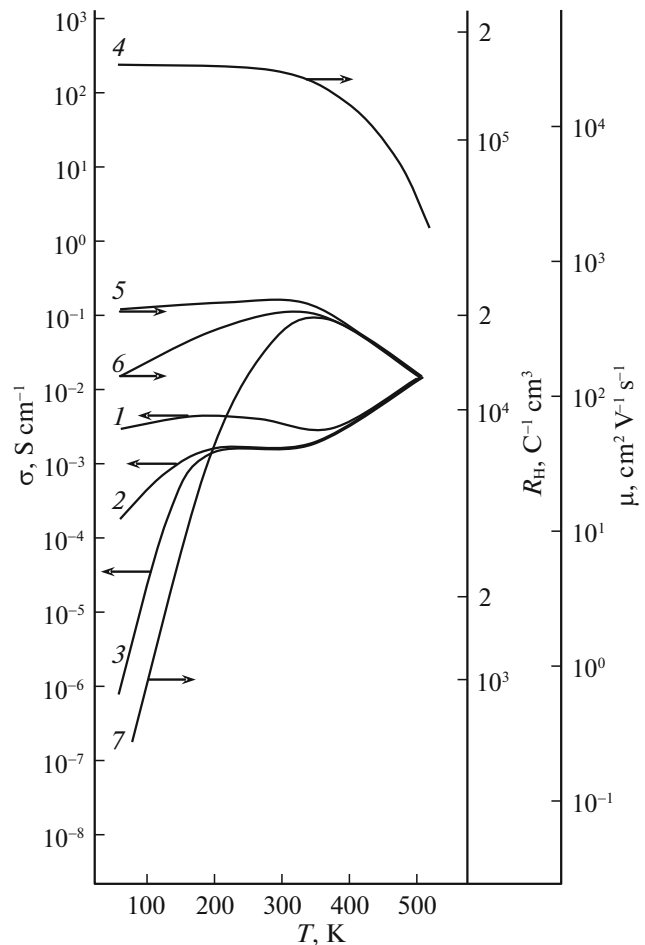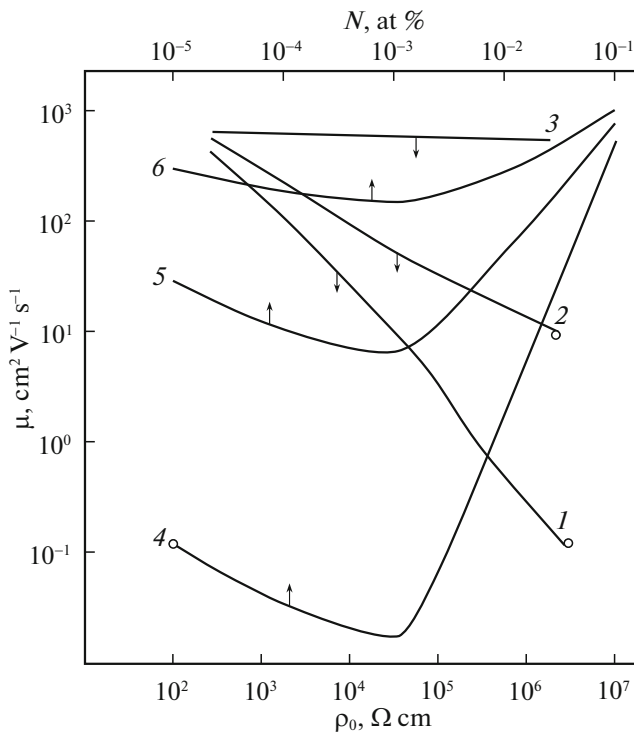

Fig. 1. Temperature dependences of the electrical conductivity σ (curves 1–3), Hall coefficient R_H (curve 4), and electron mobility μ (curves 5–7) for n-InSe single crystals with initial resistivities ρ_0 of (1, 4, 5) 3.4×10^2, (2, 4, 6) 1.7×10^3, and (3, 4, 7) 1.7×10^6 Ω cm.

in σ_0, the behavior of the $\mu(T)$ curves approaches that predicted by the theory of free carrier mobility in quasi-ordered crystalline semiconductors (Fig. 1, curve 5).

The effect of lanthanide doping on μ only manifests itself in the low-temperature region at $N \leq 10^{-1}$ at % and consists in a variation in both the absolute value and course of the dependences of ρ on different external factors with a change in the N value (Fig. 2, curves 4–6 and Fig. 3, curves 4–6). In the investigated n-InSe crystals, the dependence of μ on the chemical nature of the embedded impurity is not observed. At all other conditions and N values being equal, the μ value and its dependences on temperature, the effect of light, and electric field for the n-InSe:Gd, n-InSe:Dy, and n-InSe:Ho crystals are identical.

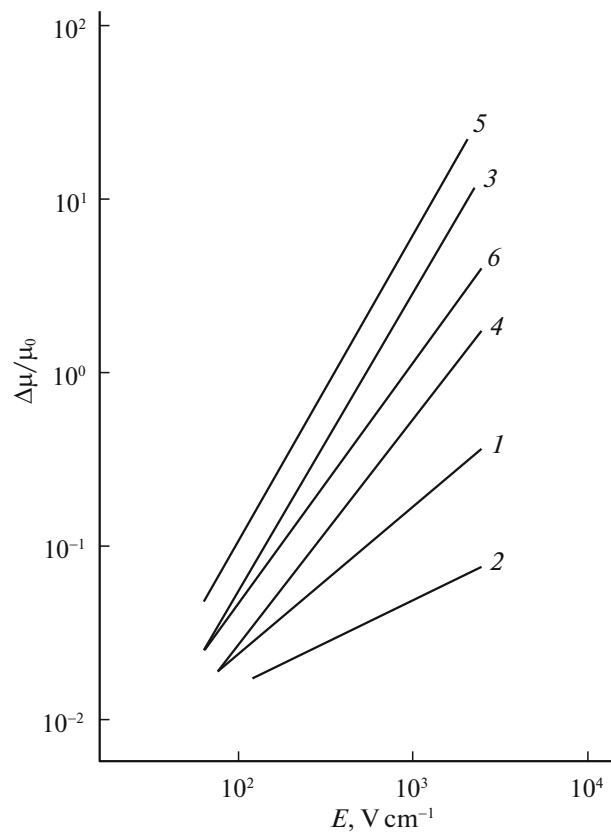In contrast to the case of free carrier heating in semiconductors by an electric field [41], in the n-InSe

Fig. 2. Dependence of the electron mobility μ on the initial resistivity ρ_0 (curves *1–3*) and content *N* of the embedded Gd impurity (curves *4–6*) for *n*-InSe single crystals at temperatures *T* of (*1, 4*) 77, (*2, 5*) 200, and (*3, 6*) 300 K.

Fig. 3. Dependence of the relative electron mobility variation $\Delta\mu/\mu_0$ on electric field *E* in pure (curves *1–4*) and Gd-doped (curves *5* and *6*) *n*-InSe single crystals at different temperatures. The ρ_0 values are (*1* and *2*) 1.7×10^3 and (*3, 4*) 1.7×10^6 Ω cm. The *N* values are (*1, 2, 3*, and *4*) 0 and (*5* and *6*) 10^{-3} at %. The temperature *T* is (*1, 3, 5*) 77 and (*2, 4*, and *6*) is 200 K.

single crystals (Fig. 3), the effect of *E* on μ is more pronounced in the samples with high ρ_0 (low μ_0) and with injecting electrical contacts; in addition, the μ(*E*) dependence is observed at relatively low *E* and has a completely different character: with increasing *E*, the μ value approaches its own value, which corresponds to *T* = 300 K, in weak electric fields.

However, in this case, the relative change in μ (the quantity $\Delta\mu/\mu_0$, where $\Delta\mu = \mu_E - \mu_0$ and μ_0 and μ_E are the μ values at the investigated *T* value in weak and strong electric fields, respectively) with *E* depends not only on *T*, but also on ρ_0 and *N*. The greatest $\Delta\mu/\mu_0$ value is observed in the sample with the highest ρ_0 value and, depending on *N*, the $\Delta\mu/\mu_0$ value changes nonmonotonically and attains its maximum at *N* = 10^{-3} at %.

The dependence of μ on the effect of light observed in the crystals with high ρ_0 also attracts attention (Fig. 4). In this case, first, the μ value in the illuminated sample (μ_c) differs significantly from that in the unilluminated sample (μ_T); second, the sign of the quantity $\Delta\mu = \mu_c - \mu_T$ appears dependent on the spectral composition of light incident onto the sample under study ($\mu_c > \mu_T$ when exposed to intrinsic light and $\mu_c < \mu_T$ when exposed to impurity light); third, when illumination ceases, the initial μ value is restored

much slower. At 77 K, in samples with a high ρ_0 value, the μ recovery time after stopping illumination reaches about $10^2–10^3$ s. In samples illuminated by light of moderate intensity from the intrinsic absorption region (intrinsic light), the μ_c value increases to the value at 300 K.

4. DISCUSSION

The established features of the electron mobility in the *n*-InSe single crystals (Section 3) cannot be explained only by the theoretical concepts of free carrier mobility in quasi-ordered crystalline semiconductors with different point lattice defects or local energy levels in the band gap.

Although some features of the μ(*T*) dependence can be attributed to the hopping-conductivity mechanism [15, 16], it cannot explain the observed dependence of μ on the effect of light, coincidence of the μ(λ) dependence with the negative photoconductivity

spectrum in the same sample [42], and the dependences of μ on E and N.

We attribute the features of the electron mobility in n-InSe single crystals unexplained by the theory of quasi-ordered crystalline semiconductors, first of all, to the presence of drift barriers for free carriers (current) in the free bands of this semiconductor due to potential fluctuations. Their energy height at 77 K is ~(0.05–0.20) eV for different samples. This assumption is also confirmed by the peculiar course of the temperature dependence of the current density and experimental results on space charge-limited currents [37] and isothermal relaxation of the dark conductivity (current) [23, 38] in the n-InSe single crystals.

Concerning the origin of the assumed drift barriers, the situation is as follows. The results of comprehensive investigations of the photoelectric, luminescent, and electrical properties of n-InSe single crystals [20, 22–25, 37–39] suggest that due to the layered structure, segregation of the component atoms during growth, and the presence of different polytypes in the samples of this semiconductor, random macroscopic defects occur [43]. Therefore, in the low-temperature region, n-InSe single crystals behave as partially disordered crystalline materials consisting generally of a low-resistance matrix with random high-resistance inclusions. At the interfaces between low-resistance matrices and high-resistance inclusions, recombination barriers for free carriers form and, at the interfaces between neighboring high-resistance inclusions, drift barriers occur [44].

Within this model, the observed discrepancies in the electron mobility in n-InSe single crystals, which occur in quasi-ordered crystalline semiconductors, can be explained from a single viewpoint by partial disordering of the investigated semiconductor. The existing recombination barriers, in this case, spatially separate excess electron-hole pairs and drift barriers, reducing the density of free carriers involved in the current (conductivity), and reduce also the Hall mobility of excess carriers. As was shown in [45], the measured Hall mobility of free carriers (μ_H) does not correspond to their drift mobility (μ_0) and is related to it as

$$\mu_H \sim \mu_0(n_0/\langle n \rangle),$$

where $n_0 \sim \exp((\varepsilon_F - \varepsilon_{nk})/kT)$ is the free carrier density at the percolation level ε_{nk}, $\langle n \rangle$ is the average free carrier density, and μ_0 is the true (drift) mobility. The sharp change in μ_H at low T is mainly due to a change in n_0. This question was theoretically discussed in detail in [45] for the general case. We assume that with increasing temperature, under illumination from the intrinsic absorption region and/or injecting electric voltage [37], the potential relief of empty energy bands in the semiconductor is smoothed. This results in the removal of existing drift and recombination barriers
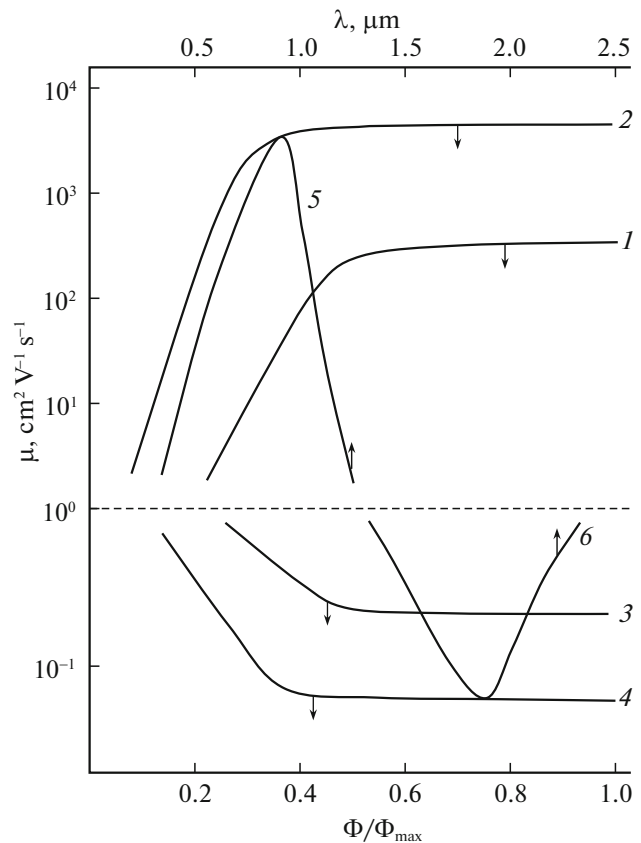
Fig. 4. Dependence of the electron mobility μ_c in illuminated n-InSe single crystals on the light intensity Φ/Φ_m (curves *1–4*) and wavelength λ (curves *5* and *6*). $T = 77$ K, $\rho_0 = 1.7 \times 10^6\ \Omega$ cm, and the Φ/Φ_m values are (*1* and *3*) 0.5 and (*2*, *4*, *5*, *6*) 1.0. The λ values are (*1* and *2*) 0.95 and (*3* and *4*) 1.60 μm.

for free carriers, which, in turn, leads to an increase in the μ value in the sample under study. Upon doping, first (at $N \leq 10^{-3}$ at %), embedded impurity ions are accumulated at macroscopic defects and grow in size under the action of an internal electric field induced at the interfaces between these defects and the low-resistance matrix. Therefore, at such N values, with an increase in this field, the degree of partial disordering of the crystal increases. Then, due to a decrease in the distances between neighboring defects, as in [46], the defects gradually agglomerate and the degree of partial disordering of the crystal lowers. As a result, at $N = 10^{-1}$ at %, the electron mobility in lanthanide-doped n-InSe single crystals at low T also obeys the theory of free carrier mobility in quasi-ordered crystalline semiconductors. In view of the aforesaid, the more pronounced manifestation of electron-mobility anomalies in high-resistance n-InSe single crystals is explained by a high degree of their partial disordering as compared with low-resistance ones. This, in turn, can explain the large ρ_0 value in them.

5. CONCLUSIONS

The low-temperature dependence of the electrical conductivity of *n*-InSe single crystals on the technological origin of a sample, doping, temperature, electric field, and illumination has a drift rather than concentration character, or, more exactly, is caused by changes in the free carrier mobility rather than in the concentration. The experimentally established deviation of the electron-mobility behavior from the presented theory of free carrier mobility in quasi-ordered crystalline semiconductors, i.e., the theoretically explained dependences of the electron mobility on the sample resistivity, temperature, electric field, illumination, and doping, are directly related to partial disordering of the *n*-InSe single crystals, which is caused by the presence of random macroscopic defects in them. The degree of disordering of the *n*-InSe single crystals, along with the variation in temperature, effect of light, and injecting electric field, can be controlled by changing the embedded impurity (Gd, Dy, and Ho) content.

At low impurity contents, due to an increase in the size of random macroscopic defects, the effect of the latter on the electron mobility becomes stronger. With a further increase in N, due to a decrease in the distance between neighboring macroscopic defects as compared with the mean free path and electron diffusion length, their influence on the electron mobility gradually weakens and, finally, completely vanishes.

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