High Temperature Properties of CdTe Crystals, Doped by Sb

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Abstract—Electrical properties of CdTe single crystals doped by Sb were studied *in situ* at high temperature point defect equilibrium under well defined Cd and Te vapour pressure. Up to ~ 700-800 K samples revealed p-type conductivity both under Cd and Te saturation. The position of the deep acceptor level $E_a = E_v + 0.28$ eV was determined using hole density temperature dependency at 350–650 K. The point defects responsible for hole density are supposed to be Sb_{Te} acceptors. The n-type conductivity above ~ 700-800 K is determined by intrinsic point defects: electrons under Te saturation due to native disorder and Cd interstitials under Cd saturation.

Index Terms—CdTe, Hall effect, high temperature, modeling, photoluminescence, point defects, Sb.

I. INTRODUCTION

BEING used for detector fabrication CdTe crystals have to possess high resistivity. Doping by shallow donors, usually In and Cl, allows to grow high resistive material due to the self-compensation phenomenon. Another way is using of Ge, V, Sn or other impurities as dopants, which produce deep levels in the middle of band gap. But in all cases precipitates (mainly Te) of different size are observed in as grown single crystals. Sb addition to CdTe essentially reduces or even eliminates the precipitation processes. When it is necessary to produce high resistive and simultaneously inclusion-free material, the Sb deep acceptor can be used in combination with other dopant.

For the first time the deep level of Sb in CdTe was defined by Iwamura by means of PICTS [1]. Sb content in the samples was determined by X-ray microanalyses as ~ 0.006 mole fractions. The band gap measured by optical investigations was found to be equal to 1.351 eV at 300 K. The samples were high resistive ($\rho = 4.5 \times 10^7 \, \Omega \times$ cm). The existence of 2 acceptor levels was

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reported: $E_{a1} = 0.068$ eV (up to 200 K) and $E_{a2} = 0.262$ eV (at $T > 200$ K). These values were determined from conductivity temperature dependency.

The results of electrical studies in CdTe crystals grown by the Bridgman-Stockbarger method and doped by Sb to concentrations of $10^{17} - 3 \times 10^{19}$ cm^{-3} (in the melt) were considered by Nikonyuk [2]. The analysis of Hall coefficient temperature dependencies, charge carrier mobility and photoconductivity values under intrinsic-absorption excitation in various parts of the ingots allowed to conclude that Sb_{Te}, Sb_{Cd} centers and probably $Sb_{Te}Sb_{Cd}$ associates are introduced in CdTe crystals by Sb doping. The hole conductivity at low temperatures in doped crystals was controlled by Sb_{Te} acceptors which concentration was no higher than 5×10^{16} cm^{-3} and was much lower than the real Sb content. The ionization energy of these acceptors was determined as 0.28 ± 0.01 eV.

Nair [3]–[5] and Picos-Vega [6] with coauthors published several papers about the properties of CdTe⟨Sb⟩ films doped during the growth run. The proton induced X-ray emission spectroscopy was used to prove dopant incorporation into the lattice. But Sb concentration in several obtained films was above 50% which looks unlikely.

CdTe layers, doped by Sb and grown by the epitaxy hot-wall method, were investigated using PL by Kanie [7]. The ionization energy of Sb acceptor center was estimated as 57 meV. Annealing showed the strong influence of Cd vacancies on the Sb electrical state. Soltani studied PL spectra of CdTe crystals doped by Sb [8]. A new line 1.54 eV with 2 peaks fine structure was found. Their intensity depended on temperature. The effective Bohr radius and ionization energy of Sb were determined: $a_{Sb} = (1.03 \pm 0.1)$ nm and $E_{Sb} = (61 \pm 2)$ meV.

The lack of information concerning the Sb behavior in CdTe crystals complicates its use in CdTe crystal manufacture. Therefore, the knowledge of the structure of point defects (PD) is important to determine electrical properties of these crystals.

The goal of this work is the investigation of the Sb influence on the CdTe properties at high temperature (HT) defect equilibrium in a wide temperature range both under Cd or Te vapor pressure.

A. Experiment

CdTe⟨Sb⟩ single crystals were grown by the Bridgman technique. The dopant was added to 6N purity elemental Cd and Te before synthesis and its concentration in the melt was $2 \times 10^{16} - 3 \times 10^{19}$ at cm^3. It was difficult to determine the Sb content in the crystal by direct analytical measurements. Therefore it was estimated using Pfann's equation, Sb segregation coefficient [9] and known values of C_{Sb}^o and "g" ("g" is the sample

location in the ingot). For the most representative sample Sb9 these values were: $C = 3 \times 10^{18}$ at cm^3, $g \approx 0.4$ and [Sb] in the sample was estimated as $(2\text{-}3) \times 10^{17}$ at cm^3. All samples $(2.5 \times 2.5 \times 15$ mm$^3)$ were grinded, polished and etched before measurements. Six welded (tungsten wire) or graphite contacts were used to provide HT Hall effect measurements under well defined Cd/Te vapour pressure up to 1200 K. Chemically deposited Cu contacts were used for Hall effect investigations under 400 K. The formulas 1 and 2 were applied for the calculation of charge carrier density and mobility. Hall factor r_H was taken equal to 1

$$n = \frac{r_H}{e R_H} \qquad (1)$$

$$\mu = \sigma \cdot R_H. \qquad (2)$$

In every moment of electrical investigations (when both the temperature and component pressure were constant) 7–10 measurements were performed. The inaccuracy of σ, μ and R_H determination was estimated as 5%–10%.

For PL measurements the samples were mounted into closed-cycle He-cryostat equipped with temperature controller that allowed to tune the temperature from 8 to 300 K. The samples were optically excited with the 441 nm He-Cd laser line with 40 mW maximum output power. The spectra were recorded via the 40 cm grating computerized monochromator system and detected with the Hamamatsu R-632 photomultiplier detector. The emission spectra were corrected according to the grating efficiency variations and the spectral response of detectors.

Cathodoluminescence (CL) analyses were performed by Gatan MonoCL system mounted on Cambridge S360 scanning electron microscope (SEM). The spectra as well the panchromatic and monochromatic images were acquired using serial dispersion system, equipped with two diffraction gratings and detection system combining photomultiplier and solid state (Ge) liquid nitrogen cooled detector.

II. RESULTS

Starting from 77 K and up to 400 K all CdTe⟨Sb⟩ samples revealed p-type conductivity. The hole mobility varied from $\sim$150-200 to $\sim$40-70 cm^2/Vs at 77 and 400 K correspondingly. The temperature dependence of Hall coefficient ($T = 77 \div 400$ K) of the samples cut from ingots with different Sb content in the melt allowed us to determine the ionization energy of acceptors responsible for the p-type conductivity. As it is apparent in Fig. 1, electrical properties of samples with low Sb content $C_{Sb}^o < 3 \times 10^{16}$ cm^{-3} are dominated by shallow acceptors ($E_V + 0.05$ and $E_V + 0.12$ eV), typical in undoped CdTe crystals. At higher Sb density with $C_{Sb}^o > 3 \times 10^{16}$ cm^{-3} shallow acceptors are fully compensated and deep Sb acceptors with the $E_a = E_V + 0.28$ eV level act as dominant PD. Only in several samples these acceptors are compensated and deeper acceptors with $E_V + (0.40 \div 0.45)$ eV level are revealed. The high temperature measurements were performed on the samples with $E_a = E_V + 0.28$ eV ionization level.

Several CdTe⟨Sb⟩ samples were investigated at high temperatures, but clear picture was observed only in the samples with high Sb concentration. In other cases the difference between CdTe⟨Sb⟩ and undoped CdTe behavior was not significant.
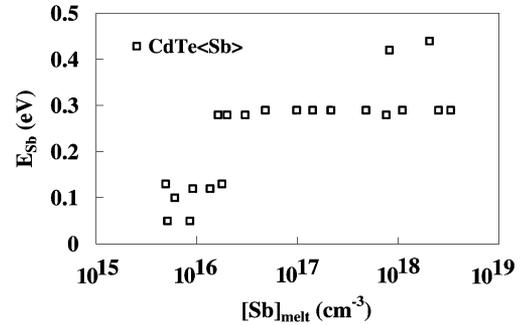

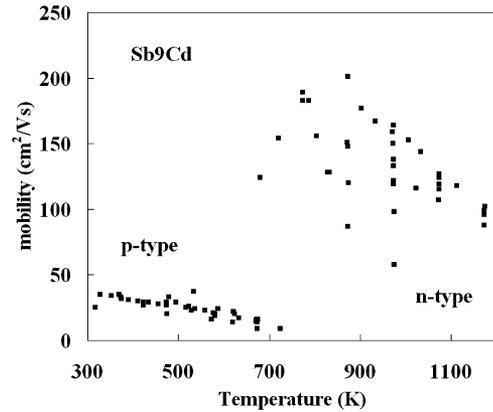Fig. 1. Acceptor ionization energy in CdTe⟨Sb⟩ crystals with different Sb content in the melt (cm^{-3}).

Fig. 2. Temperature dependency of charge carrier mobility in CdTe⟨Sb⟩ crystals under P_{Cd}.

Cd Saturation. At these conditions the samples possessed hole conductivity up to $\sim$730 K (Fig. 2). Above 870 K the electrons began to dominate and the conductivity became of n-type with reduced electron mobility from 200 to 100 cm^2/Vs at 1170 K. Cooling even after long-term high temperature measurements converted the conductivity back into p-type that is different from undoped CdTe [10].

At constant temperature at 870 and 970 K the calculated (2) electron Hall mobility reduced in 2–3 times during Cd vapor pressure decreasing (Fig. 2). Upper points at all temperatures correspond to maximal Cd vapor pressure; lower points were obtained at P_{Cd} decreasing (see explanation to Fig. 10).

The hole conductivity in temperature dependencies was clearly observed up to 630 K (Fig. 3). The acceptor ionization energy was determined from the temperature dependency and was equal to 0.28 eV. The same slope was found at low temperature Hall effect measurements in the 77–400 K temperature range. Above $\sim$630 K the conductivity was bipolar and above $\sim$870 K it became of n-type.

In the case of CdTe⟨Sb⟩ under Cd vapor pressure following regularities were observed (Fig. 4):
1) below 770 K the hole density did not depend on Cd vapor pressure;
2) at 770 K $< T <$ 970 K the calculated electron density $[e^-]$ decreased together with Cd vapor pressure reducing, but at low P_{Cd} it began to increase;
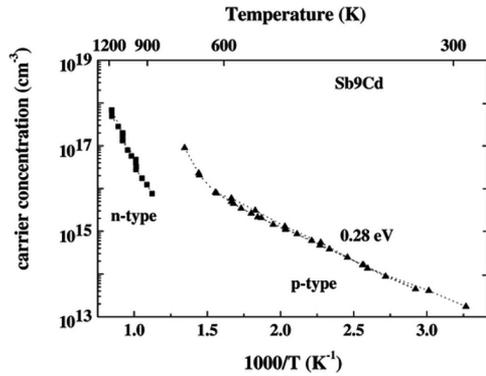
Fig. 3. Temperature dependency of charge carrier density in CdTe⟨Sb⟩ under Cd saturation. Triangles: holes; squares: electrons.
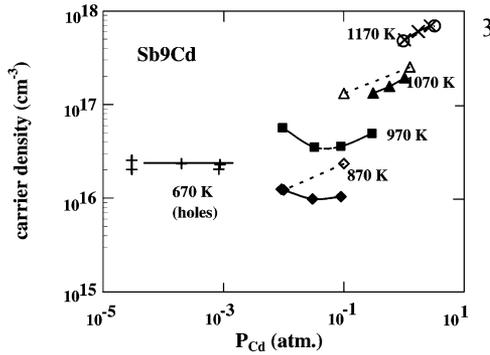

Fig. 4. Cd pressure dependency of charge carrier density in CdTe⟨Sb⟩ sample. Dashed lines – undoped CdTe at the same temperature.

Fig. 5. Temperature dependency of $[e^-]$ in CdTe⟨Sb⟩ at constant P_{Cd}:1–1 atm., 2 –0.5 atm., 3 –0.1 atm., 4 –0.01 atm.

Fig. 6. Temperature dependency of charge carriers' mobility in CdTe⟨Sb⟩ crystal under $P_{Te2,max}$.

3) at 1070 K the electron density decreased together with Cd vapor pressure reducing;

4) at 1170 K the electron density decreased with Cd vapor pressure reducing and was the same as in undoped CdTe.

The measurements of $[e^-]$ at constant P_{Cd} revealed a straight dependency upon temperature with the slopes 1 eV at $P_{Cd} = 0.1$-1 atm (Fig. 5), which are higher than those observed at the same conditions in undoped CdTe 0.8–0.85 eV [10]. This fact allows us to suppose that just foreign point defects were responsible for these slopes and determine the charge carrier density.

Te Saturation. Under Te saturation the hole mobility did not exhibit a monotonic reduction: between 570–700 K it almost did not change (Fig. 6). This peculiarity was reproduced in several runs. Above ~ 770 K the p-type conductivity was converted into bipolar type and at $T > \sim 820$ K it became of n-type.

The hole density during heating-cooling cycles demonstrated the tendency to increase. The slope of hole density at the temperature dependencies was 0.33 eV at the beginning and 0.21 eV at the end of the measurements. Between 770–830 K an inversion of conductivity type was observed. Above 870 K electrons became the major charge carriers. The Arrhenius activation energy in this region was reproducible and equal to $\Delta E \sim 0.8$ eV. Under Cd saturation the highest measured $[h^+]$ was equal to $\sim 2.5 \times 10^{16}$ cm^{-3}, under Te saturation—$\sim 4 \times 10^{16}$ cm^{-3}.

To investigate the influence of stoichiometry deviation on the CdTe⟨Sb⟩PD structure an annealing of 3 sets of samples under vacuum, maximal P_{Cd} and P_{Te2} was performed. All PL spectra showed exciton emission at about 1.59 eV indicating
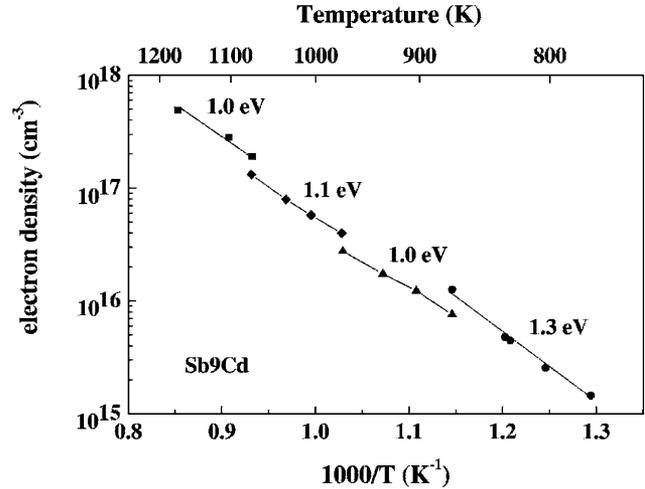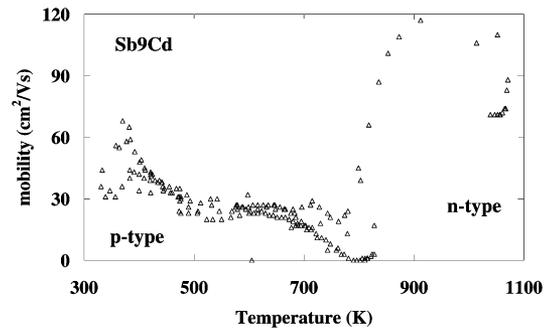
good quality of crystals (Fig. 7). However, the intensity of this emission varied at different samples. At the same time it was not possible to assign this exciton emission to Sb acceptor and it is probably related to excitons bound to intrinsic defects [14]. Sb-related excitons were proposed in [7] at 1.593 and 1.590 eV, but our results indicate that these lines can not be related to Sb acceptor.

Very intense PL band with phonon replicas at 1.55 eV appears in vacuum- and P_{Te2}-annealed samples, respectively (Fig. 7). This band is generally related to V_{Cd} and therefore it quenches in Cd-annealed sample. Instead a broad PL band at 1.42 eV becomes apparent in Cd-annealed samples.

The 1.42 eV band is usually attributed to $V_{Cd} - Cl_{Te}$ like defect and does not have any relation to Sb acceptor. At the same time this sample shows also an intense exciton emission at 1.595 eV and this peak is probably related to the exciton bound to the donor defect $-D^0X$. At this point it is too early to say anything about the nature of this donor defect, but it is not excluded that it can be Sb_{Cd} defect.

The comparisons between the CL spectra acquired from CdTe⟨Sb⟩ crystals with different Sb content in the melt are shown in Fig. 8. The same excitonic lines observed in the PL curves of Fig. 7, acceptor bound at 1.588 eV and donor bound at 1.595 eV, with its LO phonon replica at 1.574 eV, were present in both spectra. In addition a broad band centered at
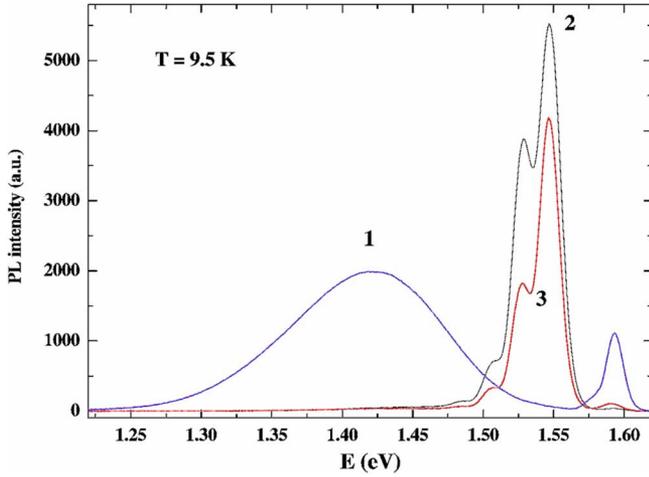
Fig. 7. Photoluminescence spectra of CdTe⟨Sb⟩ samples, measured at 9.5 K, annealed under: 1-$P_{Cd,max}$; 2- vacuum; 3-$P_{Te2,max}$.
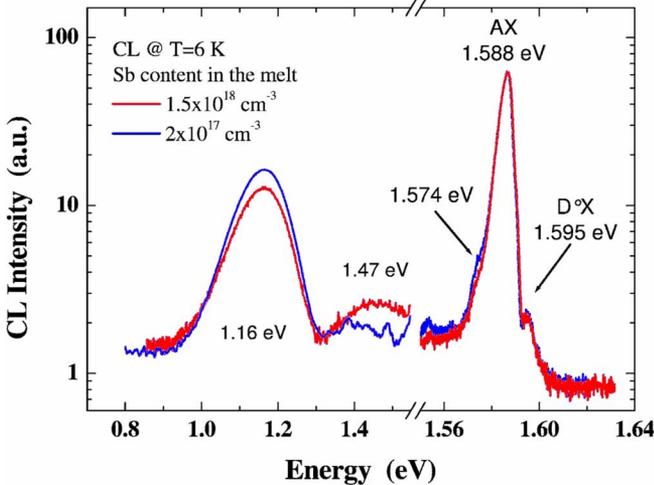

Fig. 8. Cathodoluminescence spectra of CdTe⟨Sb⟩ crystals with different Sb content in the melt. The NBE band is expanded for clarity.

1.16 eV with absolute intensity increasing by increasing the Sb content in the melt from 2×10^{17} to 1.5×10^{18} cm^{-3} was also observed. A deconvolution procedure revealed that in addition to the 1.16 eV emission another CL peak centered at about 1.08 eV was present. The nature of these bands was studied by temperature and injection density dependent CL analyses. The donor—acceptor (DAP) transition can be attributed to the 1.16 eV whilst the uncertainty in the fitting makes impossible an accurate attribution to the 1.08 eV one.

Available literature on PL studies of CdTe⟨Cl⟩ samples [15] shows that emissions centered at 1.08 eV and 1.17 eV have a DAP nature also involving a deep donor and a deep acceptor. The authors suggested PD related to the Te lattice as responsible for these transitions.

III. DISCUSSION

In the framework of the quasichemical defect reaction theory [13] the following point defect structure model in CdTe⟨Sb⟩ crystals can be proposed. The main dopant point defect, which insures hole conductivity of obtained ingots, is the acceptor center Sb on Te place

$$Sb(s) + V_{Te}^{o} \longleftrightarrow Sb_{Te}^{-} + h^{+}. \qquad (3)$$
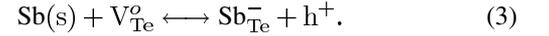
As follows from Fig. 3, this PD creates deep acceptor energy level $E_a = E_V + 0.28$ eV in CdTe. In Hall effect measurements it reveals itself in the temperature range from 77 up to 650 K. At higher temperatures the conductivity converts into n-type due to the increasing of electron density. Calculations show that the conductivity inversion occurs when the $[e^-]$ reaches ~ 0.01 of acceptor centers density ($[Sb_{Te}^{-}]$). This phenomenon is possible due to higher electron mobility comparing to hole one (approximately in 10 times). When electron density becomes higher than $[Sb_{Te}^{-}]$, the Hall electron mobility values become similar to those ones in pure CdTe (Fig. 2, upper points at the same temperature at $T > 800$ K).

However, the origin of the increase of electron density can be different in Cd and Te parts of phase diagram. Under Cd overpressure donor native PDs as Cd interstitials or Te vacancies act as a source of electrons [10]

$$Cd(g) \longleftrightarrow Cd_i^{2+} + 2e^- \qquad (4)$$

$$Cd(g) \longleftrightarrow Cd_{Cd} + V_{Te}^{2+} + 2e^-. \qquad (5)$$
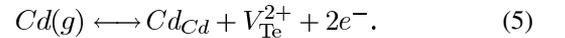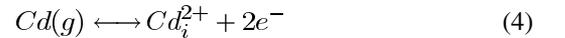
In Te vapor atmosphere at high temperatures electrons are supposed to appear due to intrinsic electronic disorder

$$0 \longleftrightarrow h^+ + e^-. \qquad (6)$$

Important question to be answered in CdTe⟨Sb⟩ is the effect consisting in the compensation of shallow acceptors with Sb doping acting as deep acceptor as it is apparent in Fig. 1. We propose two mechanisms, which could be responsible for this effect.

1) Though Sb occupies preferentially Te sublattice acting as acceptor, at high content [2] it can partly fill also Cd sites being donor there. Such donor states compensate preferentially shallow acceptors and deeper acceptor states then appear.
2) Due to self-compensation effect, which is strong in CdTe, each electrically active extrinsic defect incites the formation of native PD with electrical properties complementary to that defect. Consequently, high Sb doping results in enhanced formation of native donors (and/or in reduced formation of native acceptors), which results in the shallow acceptor compensation.

Both models might also act simultaneously at the compensation.

After long-time measurements at high temperatures ($T > 700$-900 K, depending on dopant nature and concentration) the experimentally measured electron density and its slope in temperature dependency both in undoped and doped by many impurities CdTe (Sb, In, Pb, etc.) is usually close to $\Delta E \sim 0.8$ eV (Fig. 9). Assuming that the band gap energy changes nearly linearly with temperature $E_g = E_{g0} - \alpha T$, the slope is equal to the zero temperature limit of the half-gap CdTe energy ($E_{g0} = 1.6$ eV) [16], which is in agreement with experiment [17]. If electron and hole effective masses $m_e = 0.096\, m_0$ and $m_h = 0.83\, m_0$ are used, experimental points are fitted with $\alpha = 6 \times$
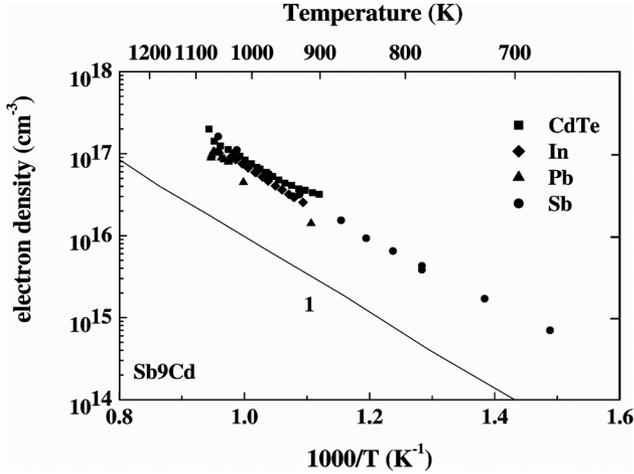
Fig. 9.   Temperature dependency of electron density in pure and doped CdTe crystals under Te saturation. 1: The intrinsic electron density [12].

Fig. 10.   Cd vapor pressure dependency of charge carrier mobility in CdTe⟨Sb⟩ crystals.

Fig. 11.   Temperature dependency of Hall coefficient in CdTe⟨Sb⟩ crystal. Experimental data are shown by crosses, full line plots the theoretical fit.

10^{-4} eV/K^{-1}. This α is larger than published data [15], which usually range between $(3 \div 5) \times 10^{-4}$ eV/K.

After cooling the native electron density of different origin (4)–(6) quickly reduces and at $T <\sim 600$ K the material restores its initial hole conductivity because the remaining acceptor centers Sb_{Te}^{-} become the dominant PD (Fig. 3).

In Cd vapor pressure dependencies below 1000 K in many CdTe⟨Sb⟩ samples the electron density and its line slope ($\gamma = 0.1$-0.25) were lower than in undoped material due to the presence of Sb_{Te}^{-} acceptors which suppress partly the electron density (Fig. 4). At higher temperatures (especially at 1170 K) this slope was close to $\gamma = 0.33$, which is typical in pure CdTe and $[e^{-}]$ was the same as in undoped crystals. It coincides with Smith's and Chern's results [11], [12] and allows us to suggest that Cd_i^{2+} is the main native donor PD at these conditions [10].

In the $\lg[e^{-}] - \lg P_{Cd}$ dependencies at 870–970 K plotted in Fig. 4 the carrier density rise is observed at low P_{Cd}. In this case the conductivity is not determined by electrons, but by holes. We used (1) for calculations, but at low P_{Cd} its application is not valid. The evidence of this assumption is the electron Hall mobility dependency upon P_{Cd} (Fig. 10). In fact the charge carrier mobility should not depend on the P_{Cd} value and is almost constant at the same temperature as it can be observed at 1170 K, for example. The reason of such electron mobility dependency vs P_{Cd} at 870–970 K is in comparable content of donor $\left(Cd_i^{2+}\right)$ and acceptor $\left(Sb_{Te}^{-}\right)$ point defects. In this case the equation for bipolar conductivity has to be used and the electron and hole concentrations should be calculated separately.

The modeling of temperature dependency of charge carrier density is shown in Fig. 11 with following model parameters:
— shallow donor density is 2×10^{15} cm^{-3};
— shallow acceptor density is 9×10^{15} cm^{-3}; $E_a = E_V + 0.26$ eV;
— deep acceptor density is 25×10^{15} cm^{-3}; $E_a = E_V + 0.5$ eV.

As one can see the agreement between experiment and calculations is good. The similar coincidence was obtained in modeling of charge carrier mobility and conductivity.
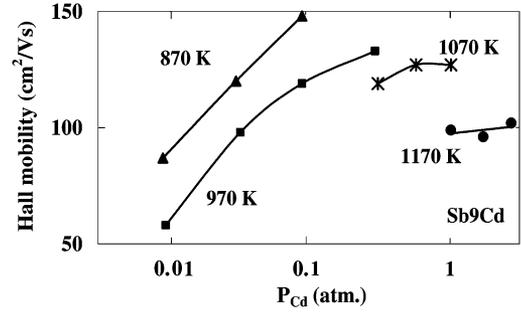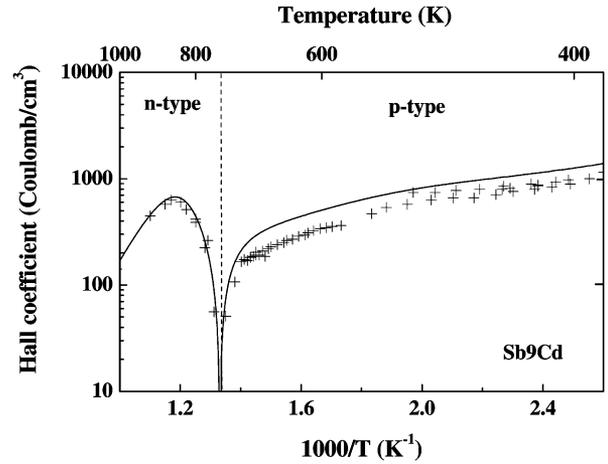
IV. CONCLUSION

Using high temperature Hall effect it was established that Sb in CdTe forms Sb_{Te}^{-} centers, which act as acceptors determining the hole conductivity up to $\sim$700-800 K both under Cd and Te vapor pressure. The Sb level location in the gap is $E_a = E_V + 0.28$ eV. At higher temperatures CdTe⟨Sb⟩ crystals convert into n-type conductivity due to the free electrons dominance incurred either by high native donor (Cd_i) content at Cd saturation or intrinsic electrons under Te saturation.

In comparison with undoped CdTe the electron density in CdTe⟨Sb⟩ crystals is lower under Cd vapor pressure up to 1100 K. It reduces with P_{Cd} decreasing with the 0.1–0.25 eV slope. At low P_{Cd} the Hall coefficient R_H begins to fall due to the mixed conductivity arranged by commensurable densities of Sb acceptors and native donors.

In CdTe⟨Sb⟩ crystals under Te saturation the hole mobility is not monotonous with temperature. The hole density grows with temperature more than in one order of magnitude. The activation energy changes from 0.33 eV at the beginning of measurements to 0.21 eV in the end of the measurements. At high temperatures the slope of electron density on temperature is equal to ~ 0.8 eV.

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