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Relaxation of electrical properties of n-type layers formed by ion milling in epitaxial HgCdTe doped with V-group acceptors

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Abstract
The relaxation of electrical properties of As- and Sb-doped HgCdTe epitaxial layers, which were converted into n-type by ion milling, is studied. It is shown that donor complexes formed under ion milling and responsible for p-to-n conductivity type conversion are not stable, and their concentration decreases upon storage even at room temperature. Increasing the temperature of the storage speeds up the relaxation process. It is demonstrated that the relaxation is caused by the disintegration of the donor complexes that starts right after the end of the milling process because of the decrease in the concentration of interstitial mercury atoms, which were generated during the milling. The results presented in the paper are important for the development of the technology of photodetectors based on HgCdTe doped with V-group acceptors.

1. Introduction

Ion milling (IM) of narrow-gap p-$Hg_{1-x}Cd_xTe$ (MCT) is currently used as a method of p–n junction formation in the technology of photodetectors based on this material [1, 2]. The method employs the effect of the formation of a thick (up to a few hundred μm depending on the IM mode and material parameters) layer converted into n-type conductivity as a result of IM of p-type MCT [3]. Initially, conductivity type conversion under IM was observed in vacancy-doped MCT, yet recently it was also reported for the material doped with I- and V-group acceptors (Cu, Au, As, Sb) [4–7]. The study of IM-induced conversion in MCT doped with As is particularly important. Arsenic is currently the most popular acceptor dopant in MCT epitaxial technology due to the low diffusivity of this species [8, 9]. Conductivity type conversion in HgCdTe:As subjected to IM was first reported in [4] and was studied in [10–14]. It is commonly accepted now that it takes place through the formation of donor complexes, which comprise an arsenic atom sited in tellurium sublattice As_{Te}, and a mercury interstitial Hg_i generated during the milling [10–12, 15, 16]. The validity of this model was also confirmed by the studies of reconversion of the IM-converted material back into p-type by means of annealing in Hg vapours [14, 16]. Currently the process of p–n junction formation by ion milling of HgCdTe:As is used in developing high-density vertical infrared photodetectors [16].

Recently, it has been found that the electrical properties of the IM-converted n-type layers in vacancy-doped MCT were not stable and changed upon storage even at the room temperature [15]. The authors of [15] suggested that such an instability was caused by the disintegration of

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Table 1. Parameters of epitaxial $Hg_{1-x}Cd_xTe$ layers used in this work.

						Before IM (77 K)			After IM (77 K)	
Sample	Dopant	x	$C_{As(Sb)} \times 10^{-16}$ (cm^{-3})	j (mA cm^{-2})	t (min)	$p \times 10^{16}$ (cm^{-3})	μ_p (cm^2 V^{-1} s^{-1})	d_j (μm)	n, right after IM $\times 10^{15}$ (cm^{-3})	n, after the relaxation $\times 10^{15}$ (cm^{-3})
B1-1	As	0.22	2 ± 1	0.1	5	1.9	410	10	16	2
B1-2	As	0.22	2 ± 1	0.15	10	2.9	170	16	30	2.5
B1-4	As	0.22	2 ± 1	0.15	10	3.1	270	16	40	2
B2-1	Sb	0.18	7 ± 3	0.15	5	1.8	630	21	3	1.5
B2-2	Sb	0.18	7 ± 3	0.1	10	1.8	550	23	3	1.5

donor complexes, which formed under IM and comprised atoms of residual acceptors (e.g., As). In [17] this suggestion was confirmed experimentally for samples doped with As intentionally; however, characteristic relaxation times observed in [17] greatly exceeded those reported in [15]. It is worth noting that all the studies of the IM-induced conversion in MCT doped with As (or Sb) performed so far have been done without taking into account relaxation processes. The non-uniformity of electrical properties of converted layers over their depth, and the dependence of the conversion depth on the alloy composition were not taken into consideration either. This created obstacles for the correct interpretation of the experimental data, as was the case, for example, for MCT layers doped with Sb [12].

In this work we report on the studies of the relaxation of electrical properties of the IM-converted epitaxial HgCdTe:As and HgCdTe:Sb, which were performed by taking into account the non-uniformity of the properties over the thickness of the layers. We demonstrate that the concentration of the donor complexes formed under IM decreases with time upon storage at the room temperature; this relaxation process is thermally activated. The results presented in this work confirm the validity of the existing model of conductivity type conversion under IM in MCT doped with V-group acceptors, and should be considered upon the development and employment of photodetectors based on this material.

2. Experiment

For the experiments, we used single-crystal varyband epitaxial p-HgCdTe layers grown by isothermal vapour phase epitaxy (ISOVPE) on CdTe:As and CdTe:Sb (1 1 1) substrates and autodoped with As and Sb, respectively [18]. After the growth the layers were annealed *in situ* at temperature $T \sim 300$ °C in Hg vapours to fill up the mercury vacancies. The thicknesses of HgCdTe:As and HgCdTe:Sb layers were $\sim$100 μm and 80 μm, respectively. According to the data of secondary ion mass-spectroscopy, the alloy composition x and dopant concentration $C_{As(Sb)}$ in the samples were constant throughout the $\sim$30 μm thick surface layer.

An IB-3 ion-milling system (EIKO, Japan) was used to perform IM with a 500 eV ion energy. The values of ion current density j and treatment duration t are listed in table 1; they were typical for the IM procedure. The sample temperature during IM was kept at $\sim$293 K by means of water cooling of the sample holder.

For the study of relaxation of electrical properties of the layers after the IM, the samples were subjected to isothermal and isochronous annealing in air. Isothermal annealing was performed at 293 ± 3 K. Isochronous annealing was performed in the temperature range of $T = 20$–140 °C with the step $\Delta T = 20$ °C and 30 min duration of every annealing stage.

The electrical properties of the samples were studied by measuring the Hall coefficient R_H and conductivity σ in the magnetic field B of 0.01 up to 1.5 T at $T = 77$ K. The R_H(B) and σ(B) dependences were analysed using discrete mobility spectrum analysis (DMSA) [7]. The profiles of the electron n and hole p concentrations, as well as the conversion depth d_j, were determined by measuring the magnetic field dependences of the integral R_H and σ with step-by-step chemical etching.

3. Results

The starting samples were of p-type conductivity at 77 K. The electrical activity of As atoms in HgCdTe:As layers was close to 100%, while in HgCdTe:Sb the electrical activity of the dopant was only $\sim$25% (table 1).

After the IM the integral values of R_H in both series of the samples were negative in the whole range of magnetic fields, which testified to the formation of n-type layers of a certain thickness.

The results of the study of the relaxation of electrical properties of the IM-converted n-type layers in typical HgCdTe:As and HgCdTe:Sb samples at the room temperature are presented in figure 1. It is seen there that both samples demonstrated substantial changes in integral R_H and σ values with time; in HgCdTe:Sb these values relaxed much quicker and changed less than those in HgCdTe:As. The field dependences of the integral R_H in the n-type converted layers pointed to the presence of electrons with both high and low mobilities, i.e., to the formation of an n^+-n-p structure with a radiation-damaged n^+-layer and a bulk n-layer. This is typical for narrow-gap MCT subjected to IM [19]. The formation of this type of structure was also confirmed by the analysis of the mobility spectra by DMSA (figure 2). The mobility spectra of the samples after the IM were dominated by peaks of electrons. Let us note that in contrast to vacancy-doped MCT, in the mobility spectra of the layers doped with As or Sb, the peaks of the electrons of the bulk n-layer (with high mobility) greatly exceeded those of the electrons of the n^+-layer (with low mobility). This can be explained by substantially greater concentration of electrons in the bulk of the converted n-layer in extrinsically doped samples as compared to vacancy-doped ones, i.e., by larger concentration of the donor centres formed under IM in the intentionally doped samples.

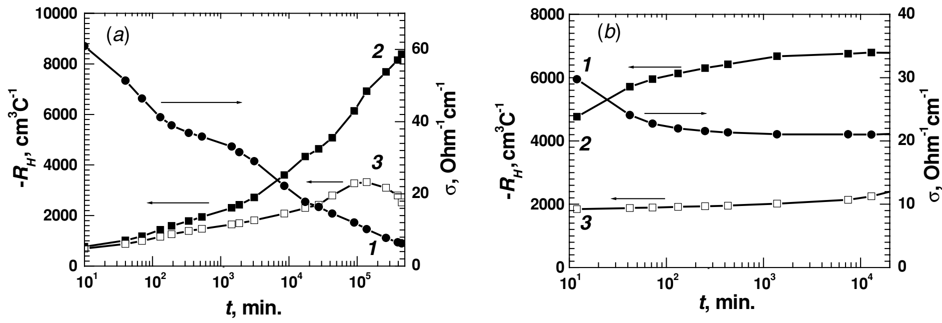

Figure 1. Time relaxation of integral values of conductivity (1) and the Hall coefficient ((2) 0.05 T, (3) 1.0 T) as measured at 77 K for IM-converted samples B1-4 (*a*) and B2-2 (*b*) during isothermal annealing at 293 K.

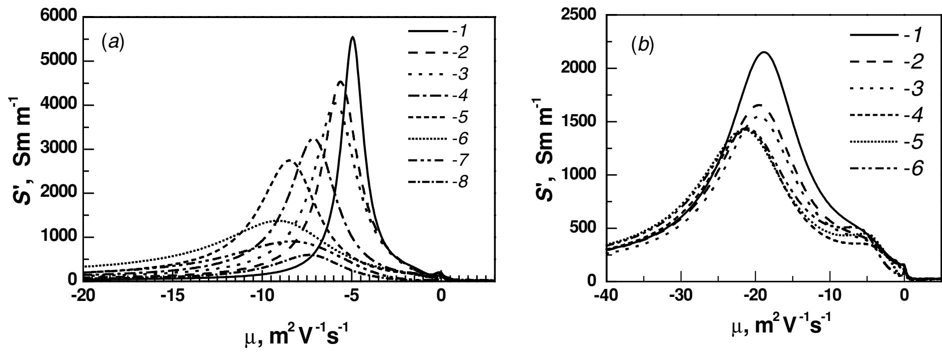

Figure 2. Mobility spectra envelopes for the samples B1-4 (*a*) and B2-2 (*b*), as measured after the IM upon storage at 293 K for (*a*) 10 (1), 40 (2), 70 (3), 340 (4), 1870 (5), 17370 (6), 90 660 (7) and 269 410 (8) min; (*b*) 12 (1), 42 (2), 72 (3), 7450 (4), 73520 (5) and 357 150 (6) min.

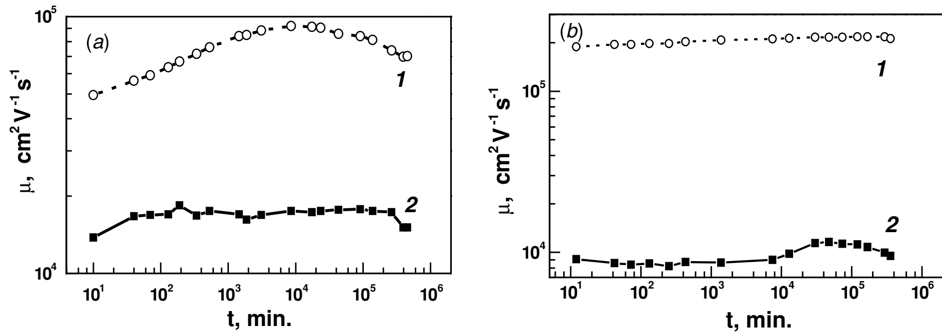

Figure 3. Time relaxation at 293 K of electron mobility (measured at 77 K) in the samples B1-4 (*a*) and B2-2 (*b*): (1) high-mobility electrons, (2) low-mobility electrons.

Upon storage of the samples at the room temperature the peak of the electrons with high mobility gradually decreased, pointing to the decrease in the concentration of the donor complexes.

The mobility and concentration of the electrons as determined by the use of DMSA on the basis of the data acquired in the process of isothermal annealing are given in figures 3 and 4. To calculate the carrier concentration in the bulk of the converted n-layer, we used the values of the thickness of the layer determined with the step-by-step etching. On the basis of the mobility values (see figure 3), it is safe to assume that the electrons with high mobility are the electrons of the bulk n-layer, while low-mobility electrons are those of the damaged n^+-layer. Therefore, these carriers are located in different sub-layers of the converted layer. Let us note that the electron mobility in the bulk of the converted layer represented a value, which was typical for n-HgCdTe with $x \approx 0.2$ and similar doping level.

It follows from figure 4 that upon isothermal annealing the concentration of electrons in the bulk of the converted layer decreased exponentially. The law of the relaxation of electron concentration for both series of samples corresponded to the first-order reaction and comprised three (for HgCdTe:As) and two (for HgCdTe:Sb) exponential stages with different characteristic times:

$$n = N_1 \exp(-t/{}_1) + N_2 \exp(-t/{}_2) + N_3 \exp(-t/{}_3) + N_0. \quad (1)$$

The parameters of relaxation curves obtained by fitting the experimental data are given in table 2. It is seen there that the characteristic relaxation times for HgCdTe:Sb are substantially smaller than those for HgCdTe:As. This points to the fact that

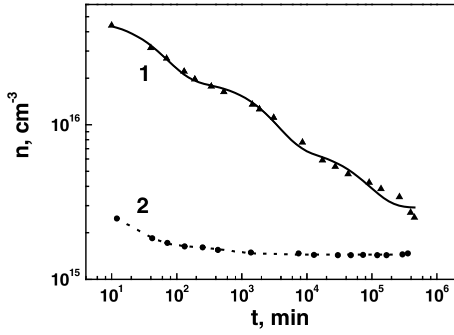

Figure 4. The dependence of the electron concentration (77 K) in the bulk n-layer on the duration of isothermal storage at 293 K for the samples B1-4 (1) and B2-2 (2).

Table 2. Fitting parameters of relaxation curves described by (1).

	Concentration $\times 10^{15}$ (cm^{-3})				Characteristic time (min)		
Sample	N_1	N_2	N_3	N_0	τ_1	τ_2	τ_3
B1-4 (*n*)	30	12	4.2	3	50	2500	70 000
B2-2 (*n*)	1.4	2.4	–	1.4	20	650	–

in the former case the donor complexes formed under IM are much less stable. This statement is also confirmed by the fact that the electron concentration in HgCdTe:Sb (3×10^{15} cm^{-3}) straight after the IM was much less than the hole concentration in the starting samples.

The time relaxation of the electron concentration in the damaged n^+-layers (not shown) was more complicated. It is known that such layers contain extended defects that capture Hg_i atoms, and create specific donor complexes, which relax in a different way than those formed in the bulk n-layer [20]. The analysis of relaxation of the defects in the damaged layers during isothermal storage will be presented in future publications.

Figure 5 shows a decrease in the electron concentration in both the bulk n-layer and the damaged n^+-layer upon isochronous annealing of HgCdTe:As sample B1-4, which had previously undergone isothermal storage. As is seen in the figure, upon isochronous annealing the electron concentration in the converted n-layer continued to decrease, which pointed to further relaxation of the donor complexes. Thus, full relaxation was not achieved during the isothermal storage.

The relaxation curve 1 in figure 5 was well described by the exponential law:

$$n(t) = n_0 \exp(-t/\tau), \quad (2)$$

where $\tau = \tau_0 \times \exp[E_{ann}/(k_B T)]$. The n_0 and τ_0 values were 2.5×10^{15} cm^{-3} and 7×10^{-7} s, respectively, with the energy of annealing activation $E_{ann} \approx 0.75$ eV. Let us note that at $T = 294$ K, $\tau \approx 7 \times 10^4$ min, which corresponds to the characteristic time of isothermal relaxation at the last stage τ_3.

Experimental data on the relaxation of the damaged n^+-layer (curve 2 in figure 5) were also well described by (2). There, the n_0 and τ_0 values were 2.6×10^{15} cm^{-3} and 7×10^{-7} s, respectively, with the energy of annealing activation equal to 0.79 eV.

To check the validity of the above analysis, which was performed under the assumption of the existence of the two

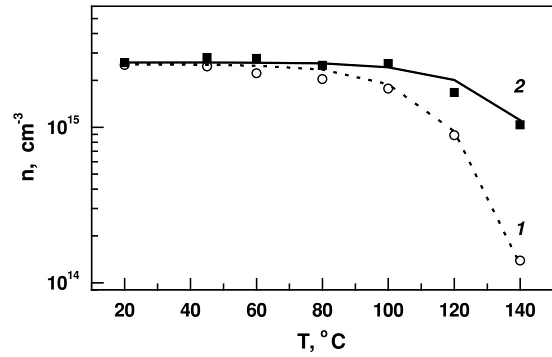

Figure 5. The dependence of the electron concentration (77 K) in the bulk n-layer (1) and the damaged n^+-layer (2) on the temperature of isochronous annealing for the sample B1-4.

sub-layers (the bulk n-layer and the damaged n^+-layer), we have also measured profiles of the carrier concentration in the samples subjected to IM. The results are presented in figure 6. Since the characteristic times of the electron concentration relaxation in the IM-converted HgCdTe:Sb were small, in reality the measurements of the carrier concentration profiles in these samples were performed after the relaxation. In samples B2-1 and B2-2 the profiles were measured after 2 and 250 days, respectively. The results presented in figure 6(*a*) clearly confirm the presence of two sub-layers in the converted layer. The electron concentration in the bulk n-layer in both samples was determined as $\sim 1.4 \times 10^{15}$ cm^{-3}; this value fitted well with that derived for sample B2-2 by DMSA (see figure 4, curve 1). The authors have to admit that in [12] such a low electron concentration and an assumption that the conversion depth did not depend on the MCT composition (which was later proven wrong [21]) made them confuse the damaged n^+-layer with the bulk n-layer.

In HgCdTe:As (figure 6(*b*)) the carrier concentration profiles were measured during the relaxation for sample B1-1 ([10, 12]), and after the relaxation for sample B1-4. Despite the large scatter of the experimental points in the profile measured during the relaxation of the sample B1-1 (figure 6(*b*), curve 1), we can see that the electron concentration in the bulk of the converted layer at the beginning of the relaxation process (with the mean value of $\sim 1.2 \times 10^{16}$ cm^{-3}) was close to the hole concentration (as well as C_{As} value) in the sample as measured before IM. This finding confirms the validity of the results of the analysis of $R_H(B)$ dependence (sample B1-2) as well as of DMSA (sample B1-4), listed in table 1 and displayed in figure 4. After the relaxation the electron concentration in the bulk of the converted layer ($\sim$2–3×10^{15} cm^{-3}) was much less than C_{As} and close to the concentration of residual donors.

The electron concentration in the damaged n^+-layers varied widely from sample to sample even after relaxation (figure 6(*b*), curves 2 and 3). This may be due to the presence of different kinds of donor centres formed in these layers under IM, and different relaxation mechanisms, correspondingly.

4. Discussion

The observed relaxation of the electrical properties of the IM-converted HgCdTe:As(Sb) layers points to the fact that

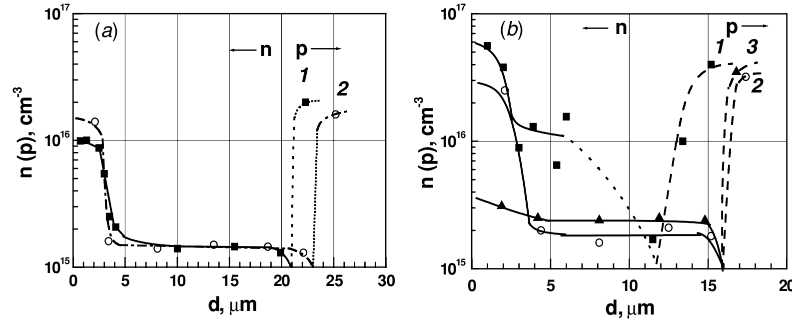

Figure 6. Carrier concentration profiles in n^{+}-n-p structures formed by the IM in (*a*) HgCdTe:Sb ((1): B2-1 [12], (2): B2-2), and (*b*) HgCdTe:As ((1): B1-1, (2): B1-2, (3): B1-4)).

donor centres formed under IM are not stable. This testifies to the validity of the concept, which implies the formation of donor complexes in extrinsically doped MCT under IM. Indeed, according to the model of [10] the complex $D^{\cdot}$ is formed by the reaction

$$\mathrm{Hg}_I^{\cdot\cdot} + X'_{\mathrm{Te}} \rightarrow D^{\cdot}, \tag{3}$$

where X'_{Te} designates a substitutional acceptor centre. The law of mass action, which corresponds to the reaction (3), is

$$[\mathrm{Hg}_I^{\cdot\cdot}] \times [X'_{\mathrm{Te}}] = K_X[D^{\cdot}], \tag{4}$$

where $K_X = K_0\,\exp(-\Delta H_{cX}/k_{\mathrm{B}}T)$ is the equilibrium constant ($K_0 \approx N_0$ is a node density in the metal sublattice), and ΔH_{cX} is the enthalpy of the complex formation. Equation (4) should be supplemented by a condition of the existence of a permanent amount of the V-group element $[X]_\Sigma$, dissolved in the crystal

$$[X'_{\mathrm{Te}}] + [D^{\cdot}] = [X]_\Sigma, \tag{5}$$

where we took into consideration that the amount of X in interstitial positions is negligibly small (which is indeed true for As atoms), and X'_{Te} centres are ionized at the room temperature.

From (4) and (5) we obtain

$$[D^{\cdot}] = \frac{[\mathrm{Hg}_I^{\cdot\cdot}]}{[Hg_I^{\cdot\cdot}] + K_X}[X]_\Sigma. \tag{6}$$

It follows from (6) that at $[\mathrm{Hg}_I^{\cdot\cdot}] \ll K_X$ isolated substitutional centres X'_{Te} prevail, while at $[\mathrm{Hg}_I^{\cdot\cdot}] \gg K_X$ the donor complexes $D^{\cdot}$ are prevalent. Thus, the dominant form of the V-group dopant is determined by the enthalpy of complex formation ΔH_{cX}. It was assessed in [10] that at the room temperature under typical IM conditions the V-group acceptor dopants should be bound in complexes with $\mathrm{Hg}_I^{\cdot\cdot}$ at $\Delta H_{cX} > 0.6$ eV. The values of ΔH_{cX} were assessed for HgCdTe:As and HgCdTe:Sb as 1 ± 0.1 eV and 0.8 ± 0.2 eV, respectively [10, 11]. These estimations mean a high probability of the complex formation under IM.

Upon completion of the IM process the concentration of non-equilibrium $\mathrm{Hg}_I^{\cdot\cdot}$ atoms in the converted layer quickly decreases because of their diffusion towards the surface (the characteristic time of this process d_j^2/D_I is about $\approx$10 s if the conversion depth equals 10 μm (D_I is the diffusion coefficient of $\mathrm{Hg}_I^{\cdot\cdot}$)). After $\mathrm{Hg}_I^{\cdot\cdot}$ concentration decreases down to a level, which corresponds to the condition $[\mathrm{Hg}_I^{\cdot\cdot}] \ll K_X$, according to (6), the donor complexes will begin to disintegrate. The kinetics of this process should correspond to the chemical kinetic equation of the first order that has a solution in the form of the exponential law, which describes the decrease in the donor complex concentration with characteristic relaxation time

$$\tau = \frac{1}{k_X K_X} = \frac{1}{k_{0X} N_0}\exp\left(\frac{E_{aX} + \Delta H_{cX}}{k_{\mathrm{B}} T_0}\right). \tag{7}$$

Here k_X is a constant of the rate of reaction of formation (or disintegration) of the complex, which is written in the following form [22]:

$$k_X = k_{0X}\exp(-E_{aX}/k_{\mathrm{B}}T), \tag{8}$$

where $k_{0X} = 4\pi r\,(D_I + D_{\mathrm{A}}) = 4\pi r_X D_I$, D_{A} is a diffusion coefficient of the acceptor dopant, which for As and Sb can be considered as being small ($D_{\mathrm{A}} = 0$), E_{aX} is the potential barrier, which hinders the formation (or breaking down) of a chemical bond in the complex and r_X is a capture radius, which for oppositely charged centres $r_X \approx r_D$, where r_D is a screening radius.

At the room temperature $D_I \sim 5 \times 10^{-4}$ cm^2 s^{-1} and $r \sim 10^{-6}$ cm, thus $k_{0X} \sim 6 \times 10^{-9}$ cm^3 s^{-1}. Estimations show that at $T = 300$ K and $E_{aX} = 0.2$ eV for $\Delta H_{c\mathrm{As}} = 1$ eV and $\Delta H_{c\mathrm{Sb}} = 0.8$ eV the disintegration time is $\tau \sim 3 \times 10^5$ min and $\tau \sim 150$ min for MCT doped with As and Sb, respectively. At $T = 500$ K the disintegration times become extremely small.

It is easy to see that the obtained estimations fit the experimental data quite well, even though the values of E_a and ΔH_{cX} were assessed very roughly. Therefore, it is the disintegration of the donor complexes that is responsible for the relaxation of the electron concentration in the converted layer after the IM. In these terms, the weaker stability of the complexes in MCT doped with Sb can be explained by the value of $\Delta H_{c\mathrm{Sb}}$ being close to the critical value of $\Delta H_{cX} = 0.6$ eV. This fact also explains the discrepancy between the electron concentration right after the IM and acceptor concentration in the starting HgCdTe:Sb samples.

However, such a relaxation mechanism should have resulted in the complete reconversion of the n-layer back into p-type, which we did not observe in the experiment even after having stored the samples for 1.5 years. This fact (as well as bi- and tri-exponential decay curves presented in figure 4) points to the fact that in reality the relaxation process occurs in a more complicated way. On one hand, it is easy to see that the concentration of electrons after the relaxation ($\sim 10^{15}$ cm^{-3}) in both HgCdTe:As and HgCdTe:Sb, irrespective of the $[D^{\cdot}]$ value right after the IM, is of the same

order as the concentration of residual donors in MCT grown by ISOVPE [18, 23]. In this connection one can assume that the disintegration of the donor complexes, which proceeds according to the reaction opposite to (3), continues only up to the moment when $[X'_{Te}]$ becomes equal to $[D^{\cdot}]$. Then mobile mercury interstitials, released during disintegration, leave the crystal, and donor complexes and acceptor substitutional centres compensate each other, so the electrical properties of the crystal are determined by the residual donors. Heating the crystal further activates the disintegration process, and the equilibrium shifts towards X'_{Te} centres. The alternative explanation is the disintegration of the charged (donor) complexes via electrically neutral ones. It was this concept that the authors of [15] used for explaining the relaxation process. However, in their model the mercury interstitials, which formed neutral complexes with X'_{Te}, were regarded as singly ionized donors. Since we presume that in the case under consideration mercury interstitial atoms are ionized doubly, the formation of neutral complexes, which comprise X'_{Te}, requires an assumption of the existence of a considerable amount ($\sim 10^{16}$ cm^{-3}) of other types of monovalent donors. These could be $X^{\bullet}_{Hg}$ or $Te^{\bullet}_{Hg}$ centres, especially since the data of table 1 clearly speak of the incorporation of Sb atoms in our samples in some electrically neutral form, possibly $Sb^{\bullet}_{Hg} - Sb'_{Te}$ pairs. However, the concentration of such defects, if any, in the samples under study is not known. Nevertheless, it should be noted that the results acquired during isochronous annealing, point out to the possibility of achieving the full reconversion. The effect of the full reconversion was observed by the authors of [14] in IM-converted HgCdTe:As after annealing the converted layer in Hg vapours at $T = 200$ °C during 17 h, and by the authors of [16] after annealing the material at $T = 120$ °C during 96 h. Even though it is obvious that establishing the exact mechanism(s) of the disintegration of the donor complexes in HgCdTe:As(Sb) requires further research, all these results confirm the validity of the model proposed in this paper.

5. Conclusions

We have studied the relaxation of electrical properties of HgCdTe:As and HgCdTe:Sb epitaxial layers, which were converted into n-type by ion milling. The studies were performed by taking into account the presence of a two-layer structure in the converted layers, which comprises the bulk n-layer and the radiation-damaged n^{+}-layer. We have shown that donor complexes, formed under ion milling and responsible for p-to-n conductivity type conversion, are not stable and disintegrate upon storage at the room temperature. Because of that the electron concentration in the bulk of the converted n-layer in HgCdTe:As equals the dopant concentration only just after the milling. In HgCdTe:Sb the electron concentration in the bulk of the n-layer is less than Sb concentration already right after the milling, which points to the disintegration of the complexes even during the ion milling process. In both cases the relaxation at the room temperature leads the electron concentration in the layers to become close to $\sim 10^{15}$ cm^{-3}. We have demonstrated that increasing the temperature of the storage can speed up the disintegration process. The data acquired under isochronous annealing of the converted layers confirm the possibility of achieving full reconversion as a result of thermal treatment. The results obtained should be considered in the development of photodetectors based on HgCdTe, doped with V-group acceptors, as well as in the studies of the properties of the IM-converted layers.

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