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Dynamics of native point defects in H₂ and Ar plasma-etched narrow gap (HgCd)Te

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Abstract

We report on a long-run evolution of electrical conductivity $\sigma(77\text{ K})$ of n-Hg_{1-x}Cd_xTe ($x=0.21$) samples which were created by reactive ion-etching process (RIE) in hydrogen and argon plasma. We show that after storing at room temperature $\sigma(77\text{ K})$ decreases to less than one half of its initial value in a time interval $\sim 2 \times 10^5$ s. The time of the relaxation is about 1000 times longer than the etching time. The increase of the storing temperature to 323 K results to about 5 times faster rate. In the coincidence with our previous model we interpret the effects as a result of release and diffusion out of donor-like mercury interstitials (Hg_I) captured on defects inside the sample during RIE. The numerical solution of the diffusion equation allows to estimate the diffusion constant of Hg_I at room temperature $> 10^{-8}$ cm² s⁻¹, concentrations of two trap levels $\sim 10^{16}$ and $\sim 10^{15}$ cm⁻³ and respective formation free energies ~ -500 and ~ -600 meV. The traps are interpreted as complexes which are formed by extrinsic acceptors and bound Hg interstitials. The method is proposed to be a proper tool to study the defect structure also in other semiconductors. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

The possibility to change the type of conductivity by dry etching is a special feature of narrow gap p-(HgCd)Te [1,2]. It is widely accepted [3–7] that

the p-to-n conversion occurs due to ultrafast diffusion of mercury interstitial atoms (Hg_I) which are created near the surface of the sample by bombarding of etching atoms. The Hg_I diffuse into the sample and recombine with the p-type Hg vacancies (V_{Hg}). When the Hg vacancies are filled the residual donors dominate resulting in p-to-n conversion. Such a model was successfully used for an evaluation of the migration energy of Hg_I being

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$E_M = 120 \pm 30$ meV [8]. Recently the p-to-n conversion in extrinsic p-doped(As) samples was reported [9,10] as well.

In this paper we establish that the electrical properties of n-type layer created by RIE subsequently develop. The electron concentration of this converted part decreases significantly within several days. For such a behavior we suggest a model based on traps (X) present in the sample. The Hg_I are captured in traps during RIE and form donor-like complexes XHg_I with them



After RIE Hg_I successively escape from the traps and diffuse to the surface and to the p–n junction. The concentration of the donor-like XHg_I decreases which results in decreasing of the electron concentration and the conductivity as well. We show that such a view of the role of Hg_I during and after RIE coincide with all experimental data which are known about this topic.

The measurement of the conductivity $\sigma(77\text{ K})$ is used to evaluate the density and the formation energy of the XHg_I complex. The model description is similar to the analysis in Ref. [11] where the traps are assumed to be located only in the damage region near the surface of the sample.

2. Experiment

The $Hg_{1-x}Cd_xTe$ (MCT) single crystals ($x \approx 0.21$) were grown by the diffusion-controlled Bridgman growth [12]. The samples were undoped p-type with the hole concentration $p(77\text{ K}) = 1\text{--}4 \times 10^{16} \text{ cm}^{-3}$ and hole mobility $\mu_h(77\text{ K}) = 500\text{--}700 \text{ cm}^2 \text{ Vs}^{-1}$. All experiments were performed in a standard parallel-plate RIE reactor operating at 13.56 MHz in H_2 or Ar plasma [2] and the etching time t_E was between $3 < t_E < 20$ min. The depth of the p–n junction below the etched surface was established by electron-beam induced current method (EBIC) and the thickness of n-type layer (L) reached up to 80 μm . The time dependence of the conductivity $\sigma(77\text{ K})$ was measured by the van der Pauw method (Fig. 1). The samples were stored after RIE in a liquid nitrogen (77 K) or in open air at 295 K (room temperature) or 323 K,

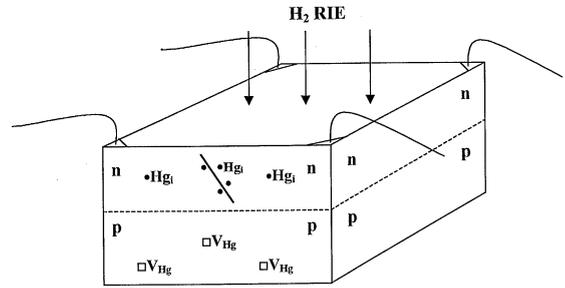


Fig. 1. Schematic diagram of measured samples. The dashed line is the p–n junction and the long arrows indicate the direction of impacting hydrogen or argon atoms. Four contacts on the surface were used for van der Pauw measurement. The p-type substrate contains Hg vacancies V_{Hg} and the trapping sites, possibly extrinsic acceptors A. Based on the presented model it is supposed that the converted n-type layer contains free and trapped Hg_I . After etching the Hg_I quickly diffuse to the surface and to the p–n junction. Trapped Hg atoms are gradually liberated due to thermal activation and the relaxation of $\sigma(77\text{ K})$ is observed by electrical measurements.

alternatively. The temperature changes ($295, 9323\text{ K} \rightleftharpoons 77\text{ K}$) were done as fast as possible to avoid indeterminate periods which took less than 1 min. Some samples were slightly chemically etched after the RIE or after the relaxation to estimate the surface conductivity. These processes had no significant effect on $\sigma(77\text{ K})$. In all cases the relaxation was dominantly affected by the t_E .

Due to the p-to-n conversion, $\sigma(77\text{ K})$ significantly increases during the RIE and the Hall coefficient changes its sign. After the RIE no evolution was observed at samples kept at 77 K during several hours. At room or higher storing temperature $\sigma(77\text{ K})$ gradually decreases approaching the equilibrium value within several days. The increase of the storing temperature from 295 K to 323 K results to about 5 times faster relaxation. The differential Hall measurements which were performed after the relaxation have shown that the equilibrium value of $\sigma(77\text{ K})$ is uniform in the whole n-type region [6].

3. Theory

The theory to explain the effects connected with the type conversion of (HgCd)Te must describe

several features which are typical for the experimental results:

(i) The thickness of the converted layer and its conductivity is proportional to the square root of the etching time t_E and the former reaches $\sim 100 \mu\text{m}$ within $\sim 10^3 \text{ s}$ [1,4]. (ii) The $\sigma(77 \text{ K})$ of converted n-type layer is uniform after relaxation [6]. (iii) The p-to-n conversion is finished shortly after etching [8]. (iv) If a part of the etched surface is shielded by a mask, the p–n conversion extends also below the mask. The p–n junction line shape agrees well with the results of two- or three-dimensional numerical simulations of diffusion from the surface source limited in space [5,6]. (v) The process of the type conversion is temperature-activated [8]. (vi) The type conversion occurs both in intrinsic-doped (V_{Hg}) and in extrinsic-doped (As) [9,10] p-type samples. (vii) The concentration of the converted area relaxes in time about 1000 times longer than t_E . (viii) The converted area is long-time stable below 70°C after the end of relaxation [13]. (ix) The relaxation is a temperature-activated process with the activation energy about 3 times higher than the type conversion. (x) The $\sigma(77 \text{ K})$ is not influenced by weak chemical etching neither during nor after the relaxation which implies that the relaxation of the surface damage region is not responsible for the effect.

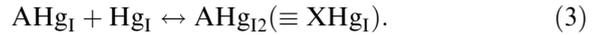
In this section we propose a model which fulfills all the given demands. In accord with our previous papers [2,6–8] we expect that Hg_I are created at the surface during RIE and diffuse fast into the sample filling the p-type Hg vacancies. Further we introduce a set of weak binding sites (traps, X) in the model where Hg_I can be captured. We use unspecified traps which do not require detailed information about their nature. The low binding energy is not sufficient to hold Hg_I in traps and they can leave them and continue to diffuse. Hg_I can be captured many times if the traps are mostly empty but they are practically free if the traps are filled. Though the charge states of Hg_I are not well understood yet, there is a prevailing opinion that Hg_I are donors (monovalent or divalent) with the ionization energy above the bottom of the conduction band [14,15]. In our model we assume that V_{Hg} (Hg_I) are monovalent acceptors (donors) and traps (X) are neutral for simplicity. The XHg_I

complexes are supposed to be monovalent donors. Using double-ionized XHg_I in the model has no substantial effect on the results, only the concentration of traps results about one-half of the values presented in this paper.

If extrinsic acceptors (A) are present (As, e.g.) we suppose, that the Hg_I neutralize them forming a complex



which is stable at room temperature [9,10]. In view of the evaluated trap concentration ($\sim 10^{16} \text{ cm}^{-3}$) the complex AHg_I could be one of the possible species, which originate the trap ($\text{AHg}_\text{I} \equiv \text{X}$). The diffusing Hg_I are temporarily captured by these traps and form the donor-like XHg_I complex due to defect reaction



This idea cannot, however, be proven from $\sigma(77 \text{ K})$ experimental data because of unknown ratio of intrinsic and extrinsic acceptors in our samples.

The set of coupled one-dimensional equations describing diffusion of Hg_I , their recombination with V_{Hg} and generation or escape at the surface during both RIE and the relaxation processes reads

$$\frac{\partial c_{\text{IT}}}{\partial t} = D_1 \frac{\partial^2 c_1}{\partial y^2} - R c_1 c_v + s \delta(y), \quad (4)$$

$$\frac{\partial c_v}{\partial t} = -R c_1 c_v, \quad (5)$$

where $c_{\text{IT}} \equiv c_{\text{IT}}(y, t)$, $c_1 \equiv c_1(y, t)$ and $c_v \equiv c_v(y, t)$ are the total Hg_I concentration (both trapped and free), the concentration of free diffusing Hg_I and concentration of V_{Hg} , respectively. The initial conditions are $c_1(y, 0) = c_{\text{IT}}(y, 0) = 0$, $c_v(y, 0) = c_0$. The Hg_I diffusion coefficient D_1 is in the form $D_1 = D_0 \exp(-E_M/k_B T)$, where $E_M = 120 \text{ meV}$ [8]. $s = s_1 - s_{21} c_1(0, t)$ defines the Hg_I surface creation and destruction, s_1 and s_{21} being temperature-independent constants [8]. The s_1 is zero after the RIE. The interstitial-vacancy recombination R and surface recombination s_{21} after the RIE are so high that c_{IT} decreases strongly to zero at the p–n junction and at the surface [6,7]. In view of the fact that the samples are long time stable below

70°C after relaxation we neglect both Hg_I and V_{Hg} generation terms and V_{Hg} diffusion term in our model. The kinetics of Hg_I is described by Eq. (4). The time evolution of the total Hg_I concentration c_{IT} is determined only by the mobile species c_I and therefore only c_I appears on the right-hand side of Eq. (4). The capture in the trap or the escape from it do not change the total Hg_I concentration and do not manifest in Eq. (4).

To simplify following numerical procedure we shall expect from now that the coupling between free and trapped Hg_I is high enough so that the c_I and c_{IT} are in a local equilibrium. Such an expectation can be accepted if the mean free path of free diffusing Hg_I between single escape and capture process l is much less than the thickness of the converted layer L . In case of the recombination with V_{Hg} , the l was estimated to be significantly less than 0.1 μm [6]. We suppose that in the case of trap recombination the situation is similar. In the opposite case if $l \geq L$, most of the Hg_I would leave the converted layer just after the first escape from the trap. Such mechanism would product the exponential relaxation of the conductivity with the time of the relaxation independent of the t_E . That result contradicts to our experimental observations. The effect of the tight coupling of c_I with c_{IT} to the simplification for the numerical treatment is obvious. Without the local equilibrium an additional equation describing trapped Hg_I density should be simultaneously solved where several unknown parameters are to be supplied as well.

We characterize the traps by the densities C_j and formation free energies F_j ; $j = 1, 2, \dots$, respectively. The $F_j = U_j - TS_j$, where energy U_j includes the trap energy and energy part of the vibration free energy, and S_j comprises the configuration and vibration entropy of the respective defect. We found two trap levels in our analysis ($j = 1, 2$). With respect to $c_I \ll C_{Hg}$, where Hg density $C_{Hg} = 1.185 \times 10^{22} \text{ cm}^{-3}$ in $Hg_{0.8}Cd_{0.2}Te$ corresponds to the Hg_I site concentration, the total Hg_I concentration is written in the form

$$c_{IT} = c_I \left[1 + \sum_j \frac{C_j}{c_I + C_{Hg} \exp(U_j/k_B T - S_j/k_B)} \right]. \quad (6)$$

The set of coupled equations (4), (5) and (6) is solved numerically optimizing model parameters to experimental data. The nine parameters list: diffusion parameter D_0 , surface generation rate s_1/s_{21} , background donor concentration N_{Dres} , and 6 parameters of two trap levels. The n-type MCT is degenerate and the donor levels are expected to lie high above the bottom of the conduction band. In case of the trapped as well as free Hg_I we expect the situation is the same being all the charged monovalent donors. The $c_{IT}(y, t)$ and $c_v(y, t)$ profiles form a body of homogeneous parallel layers which allow to express the $\sigma(77 \text{ K})$ integrating across the sample thickness d

$$\sigma(77 \text{ K}) = \frac{e}{d} \left(\mu_e \int_0^L [c_{IT}(y) + N_{Dres} - c_v(y)] dy + \mu_h \int_L^d [c_v(y) - c_{IT}(y) - N_{Dres}] dy \right), \quad (7)$$

where the thickness of the converted layer L is defined by change of the sign of the integrated function. At impurity concentration below 10^{16} cm^{-3} the electron scattering at 77 K is dominated by the optical phonons [16,17] and therefore the electron mobility $\mu_e = 10^5 \text{ cm}^2 \text{ V s}^{-1}$ [6], which is independent of impurity concentration, is used overall.

The experimental data to be fit are: the time evolution of $\sigma(77 \text{ K})$ of the sample stored alternatively at various temperatures and the thickness L of the n-type layer after the relaxation. In the case of relaxation at constant temperature only the total F_j instead of U_j and S_j can be established.

4. Results and discussion

The time dependence of $\sigma(77 \text{ K})$ was measured on samples which were etched in H_2 or Ar plasma for various etching time. The results with etching conditions are shown in Table 1. In case of the samples which have relaxed both at 295 K and 323 K, the time of the relaxation was modified according to our model as if the samples relax at 295 K all the time. Preliminary experiments were done on three other samples subjected to Ar ion

Table 1

The electrical conductivity $\sigma(77\text{ K})$, the etching time t_E , storing temperature T_S and time of relaxation t_R

Sample no.	$\sigma(77\text{ K})^a$ ($\Omega^{-1}\text{ cm}^{-1}$)	t_E (min)	$\sigma(77\text{ K})^b$ ($\Omega^{-1}\text{ cm}^{-1}$)	T_S (K)	t_R (h)	$\sigma(77\text{ K})^c$ ($\Omega^{-1}\text{ cm}^{-1}$)
6404A ^d	1.35	15 H ₂	23	295	200	8.5
50127B ^d	2.7	20 H ₂	30	295; 323	160	7
50127C	1.93	3 H ₂	10.5	295; 323	60	3.5
50127D	3.5	20 Ar	13	295	200	7.5

^a σ before etching.^b σ after etching.^c σ after time relaxation.^d The model fit is shown for labeled samples.

milling [6]. All these samples exhibit about twofold decrease of the $\sigma(77\text{ K})$ after 1 week of the relaxation in the comparison with the value just after milling.

The detailed time relaxation of $\sigma(77\text{ K})$ on two samples which were subjected to RIE in H₂ plasma was measured and fit. Though the number of nine parameters seems to be rather high, it is not easy to hit all features which are observed at the experimental data. Especially in the case of relaxation at varying temperature the complicated course of $\sigma(77\text{ K})$ asks to proceed carefully in the optimization. In the case of $D_I < 10^{-8}\text{ cm}^2\text{ s}^{-1}$ the $\sigma(77\text{ K})$ cannot be fit within our model. If $D_I > 10^{-8}\text{ cm}^2\text{ s}^{-1}$, a good fit can be obtained. However, increasing D_I results in the increase of entropy terms S as well. In the numerical calculations we have chosen $D_0 = 4.4 \times 10^{-6}\text{ cm}^2\text{ s}^{-1}$ which corresponds to $D_I = 4 \times 10^{-8}\text{ cm}^2\text{ s}^{-1}$ at room temperature and fulfills the experimental fact, that the process of p-to-n conversion is finished shortly after the end of RIE. If the Stanford $D_I = 2.35 \times 10^{-3} \exp(-0.15\text{ eV}/k_B T)\text{ cm}^2\text{ s}^{-1}$ [18] ($D_I = 6.4 \times 10^{-6}\text{ cm}^2\text{ s}^{-1}$ at room temperature) is used, the fit of the same quality as presented here can be obtained. Just the entropy terms increase by about $5k_B$ comparing to S shown in Table 2 and consequently the free energy F is modified by that, too. Such S looks however rather high because it approaches to S which is connected with a formation of a new point defect in MCT [14]. In our case the S corresponds only to the defect reaction without creation of a new defect and the S should be lower.

Table 2

The sample and trap parameters including estimated deviations for two samples. The sample 6404A was stored only in the room temperature and U_j and S_j cannot be established

Sample no.	6404A	50127B
μ_h ($\text{cm}^2\text{ Vs}^{-1}$)	640	560
$p(77\text{ K})$ (cm^{-3})	1.3×10^{16}	3×10^{16}
N_{Dres} (cm^{-3})	3×10^{15}	1×10^{15}
L (μm)	75	65
C_1 (cm^{-3})	1.4×10^{16}	1.2×10^{16}
F_1 (meV)	-480 ± 50	-500 ± 50
U_1 (meV)		-380 ± 80
S_1 (k_B)		5.3 ± 2.7
C_2 (cm^{-3})	1.4×10^{15}	1.9×10^{15}
F_2 (meV)	-610 ± 50	-620 ± 50
U_2 (meV)		-430 ± 100
S_2 (k_B)		7.6 ± 5

The presence of two dominant trap levels is demonstrated in Fig. 2 which shows the relaxation of $\sigma(77\text{ K})$ at room temperature. We observe two dominant relaxation rates at the time less and greater than $5 \times 10^4\text{ s}$, respectively. The sample parameters and the fit are collected in Table 2. The deviations depict the interval in which the fit of a good quality can be obtained. The trap energies cannot be established in the experiment at the constant relaxation temperature.

The relaxation of $\sigma(77\text{ K})$ at various temperatures allows to distinguish the energy and entropy terms in the free energy. The experimental data and the fit are shown in Fig. 3. The results are summarized in Table 2. The increase of the temperature from 295 K to 323 K results to about 5 times faster relaxation rate which is caused by

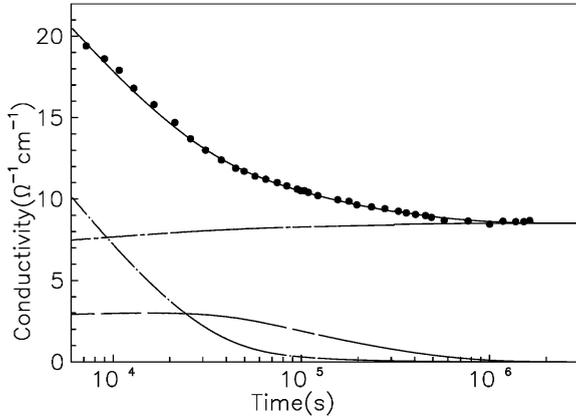


Fig. 2. The experimental (●) and theoretical (solid line) time dependence of conductivity $\sigma(77\text{ K})$ of the sample 6404A which was stored after RIE at the room temperature. The dashed and dash-dotted lines correspond to σ due to the electrons donated by the first and the second trap levels (see Table 2), respectively. The long-short dashed line corresponds to the p-type bulk + residual donors σ .

higher D_I and by higher free Hg_I concentration due to activation from the traps. With respect only to the small temperature variation ($\Delta T = 28\text{ K}$) there is relatively large estimated deviation of the trap parameters. The larger deviation in case of the second trap level is caused by the interference with the high-concentration first level.

The time evolution of thickness of the n-type layer L was also calculated during both RIE and the relaxation. We established that it reaches about 70% of the final thickness at the moment of the finishing of RIE and 15 min later 80% of that. This finding agrees well with the statement (iii) in the theory.

In view of the possibility to convert extrinsic p-doped (As) sample into stable n-type [9,10], the fitted trap concentrations allow to interpret the traps to be connected with extrinsic acceptors which are neutralized by the first Hg_I according to reaction (2). The capture of Hg_I within the trapping effect can be then depicted by reaction (3). The traps of another nature are however possible as well, e.g. inherent neutral binding sites or deep acceptors.

The presented model describes all experimental data which are known about the effects connected

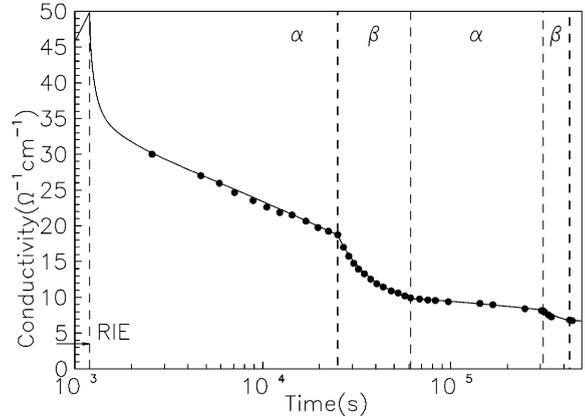


Fig. 3. The time dependence of $\sigma(77\text{ K})$ (●) of the sample 50127B which was stored after RIE at the room temperature (part α) and at 323 K (part β), alternatively. The dashed labels separate the regimes.

with the type conversion in MCT. The advantage also consists in the fact that only one diffusing species is necessary to describe the RIE and the relaxation simultaneously. All other models must work with several evolving species describing partial effects by distinct processes. One of such perspective mechanisms could be a possible diffusion of some residual donors which are liberated from their lattice sites by the exchange with Hg_I . The decision if this process occurs in MCT after RIE could be done by the analysis of the impurity profile at the border of the converted region. The concentration of such diffusing atoms would be significantly increased there.

5. Conclusions

The evolution of the conductivity $\sigma(77\text{ K})$ of n-type $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ ($x = 0.21$) after H_2 or Ar reactive ion etching was investigated. The experimental data are described by the model of two trap levels. This analysis presents a novel view on a defect structure in semiconductors. Though the physical nature of the trap states is not set definitely, we suggest that the appropriate object can be formed as the complex of extrinsic acceptor and Hg interstitial. The method can be a perspective tool to study point defects also in other II–VI or III–V

semiconductors. A rough estimation shows that in case of GaAs and similar semiconductors the RIE as well as the relaxation should happen at about 400°C. The fact that the (HgCd)Te subsequently develops after RIE is the main result reported in this paper and it was never reported before.

Acknowledgements

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