Low-temperature occupation of a donor state resonant with the conduction band in $Al_{0.35}Ga_{0.65}As$

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n_H (Hall) and n_{CV} (capacitance-voltage) electron density data are compared in a Si-doped $Al_{0.35}Ga_{0.65}As$ sample having an $N_d - N_a = 2.8 \times 10^{18}$ cm^{-3} net donor density. Data taken during low-temperature photoionization of DX centers and during temperature variations in the persistent photoconductivity regime ($T < 50$ K) suggest that as the density of photoexcited electrons increases the Fermi energy rises until a Si-related D^o donor bound state, resonant in energy with the Γ conduction-band valley, becomes populated. During the initial stage of isothermal capture transients, n_H is found to decrease at a slower time rate than n_{CV}, since electrons are continuously supplied in the Γ valley by the depopulation of the D^o level. During the final stage of the capture, when D^o is empty, n_{CV} and n_H are found to coincide. The analysis based on an equilibrium distribution of the photoexcited electrons between the Γ valley and the D^o level gives for this latter an energy of 40.6 ± 0.5 meV above the Γ minimum ($T = 0$). The possibility that the D^o state is linked to the X secondary minima of the conduction band is discussed.

I. INTRODUCTION

Isolated donor impurities in $Al_xGa_{1-x}As$ and other III-V alloys are believed to have a bistable behavior between the simple substitutional configuration and a lattice-distorted one, thus resulting in two distinct spectra of electronic bound states. The states of the substitutional configuration are expected to be hydrogenic states, while the ground state of the distorted configuration is the so-called DX center.[1–3] The DX state is characterized by a thermally activated cross section for electron capture,[4] leading to exceedingly small capture rates at low temperatures. In this way, photoexcited electrons cannot be recaptured by the DX centers and the well-known phenomenon of persistent photoconductivity (PPC) takes place.[5,6] In GaAs the DX level is resonant with the conduction band (CB), but it can be brought into the gap under hydrostatic pressure or, equivalently, by substituting a sufficiently high x fraction of Ga with Al atoms ($x \approx 0.22$ in Si-doped samples).

The coexistence of DX and hydrogenic states has been shown in different ways in $Al_xGa_{1-x}As$. In the direct gap material, electrons generated by low-temperature photoionization of DX centers can be recaptured by the ground hydrogenic state linked to the Γ minimum. Thus, in lightly doped samples a far-infrared absorption due to $1s$-$2p$ hydrogenic transitions has been observed.[3,7] In samples with doping levels in the 10^{18}-cm^{-3} range the photoexcited electron density near saturated PPC exceeds the estimated critical n_c density for the Mott transition ($n_c \approx 5 \times 10^{16}$ cm^{-3}), but under weak persistent photoexcitation mixed conduction between free electrons and electrons in the impurity band originated by the shallow state linked to Γ could be argued.[8] In indirect gap material ($x \gtrsim 0.40$), evidence that at low temperature photoexcited electrons can populate a hydrogenic level linked to the X CB minima has been found by various groups using electron paramagnetic resonance (EPR),[9–12] optically detected magnetic resonance,[13] and far- and mid-infrared absorption measurements.[14,15] A similar conclusion has also been supported by low temperature photo-Hall measurements.[16–18]

In this work we compare Hall and capacitance-voltage (C-V) density data in a highly doped ($N_d - N_a = 2.8 \times 10^{18}$ cm^{-3}) $x = 0.35$ sample. The measurements were taken in saturated PPC condition (steady state) as well as during isothermal slow capture transients. The comparison suggests that, as the density of low-temperature photoexcited electrons increases, the Fermi energy rises until a Si-related donor bound state, resonant in energy with the Γ valley of the CB, becomes populated. This level is 40.6 ± 0.5 meV above the Γ minimum: the possibility that it is linked to the X secondary minima of the CB is discussed.

II. EXPERIMENTAL RESULTS AND DISCUSSION

Hall effect measurements and standard C-V profiling in Schottky barriers give identical electron density data if the following conditions are satisfied. (i) The samples are uniform. (ii) The DX center charge state is frozen so that it cannot follow the quasistatic bias variations during C-V measurements. (iii) The electron gas is strongly degenerate so that the Hall r_H factor is one. (iv) Complications due to mixed conduction are avoided; they could be due to many-valley population (Γ,L,X) of the con-

duction band or to a partial filling of an impurity band. Finally, (v) bias variations do not induce variations in the occupancy of bound states. All these requirements are completely fulfilled, for example, in $x = 0.25$ Si-doped molecular-beam epitaxy (MBE) samples with doping levels in the 10^{18}-cm^{-3} range, under saturated PPC conditions. As pointed out in Ref. 19, the r_H factor is one and the hydrogenic level coupled to Γ is ineffective owing to the fact that the electron density overcomes the critical one for the Mott transition. Some of us have in fact verified that Hall and C-V density data agree in this case within $\pm 7\%$.[20] However, as the x AlAs molar fraction increases, the n_{CV} density derived by C-V profiling becomes significantly larger than the n_H Hall density when both data are taken under saturated PPC conditions. This is clearly seen in Fig. 1, where C-V and Hall photoionization transients taken at $T = 33$ K are compared for the same $x = 0.35$ sample.

The sample was a Si-doped layer, 5.5 μm thick grown by MBE at a temperature of 600 °C. The x AlAs molar fraction was $x = 0.35 \pm 0.018$, as deduced through growth rates measured by reflection high-energy electron diffraction oscillation periods. Between the (001) undoped semi-insulating GaAs substrate and the doped $Al_xGa_{1-x}As$ layer, a 0.5-μm-thick GaAs undoped buffer layer and a 0.2-μm-thick $Al_xGa_{1-x}As$ one were interposed. This was done to improve the quality of the sample as well as to avoid the formation of a two-dimensional electron gas at the interface between the n-doped $Al_xGa_{1-x}As$ and the undoped GaAs. The sample was finally passivated by a 200-Å-thick undoped GaAs cap. Two distinct pieces of the same sample were used for C-V and Hall measurements, respectively. Schottky diodes having a coplanar structure were fabricated for C-V measurements. Ohmic contacts were achieved by a large area evaporation of a $Au_{0.88}Ge_{0.12}$ alloy followed by annealing at 400 °C for 1 min in forming gas atmosphere. The Schottky contacts were made by evaporating Au dots having a 0.26-mm^2 area. Before the Au-Ge and Au evaporation, the surface was cleaned by etching in HCl, rinsing in methylene chloride and in iso-propyl alcohol, and, finally, dried in a nitrogen flux. The capacitance was measured by a HP impedance analyzer 4192A with a 1-MHz ac signal of 15-mV amplitude. A series-equivalent circuit was employed to account for series resistence effects. Hall measurements were taken in a 0.5-T magnetic field using a standard van der Pauw method. A GaAs light-emitting diode (LED) was used as a light source for DX center photoionization: in this way saturated PPC was easily reached.

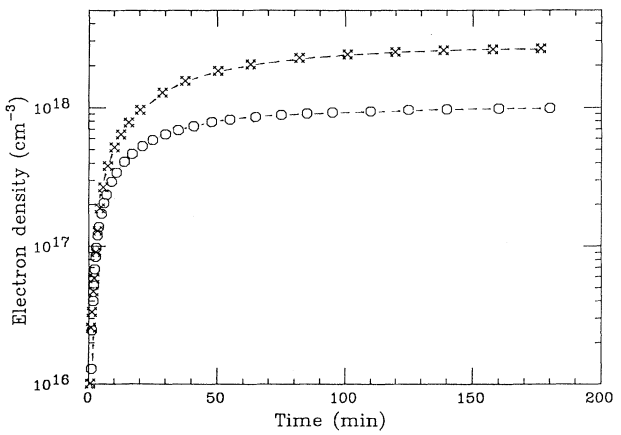

FIG. 1. Density data obtained by C-V (n_{CV}, crosses) and Hall (n_H, circles) measurements during photoionization transients. $x = 0.35$, $T = 33$ K. Each datum was taken at the end of a photoionization step with the LED switched off. The time scales were normalized to make n_{CV} and n_H coincident at the initial stages of the transient. Dashed lines are guides for the eyes.

As shown in Fig. 1, the saturation value of n_{CV} is 2.5 times larger than that of n_H. This can be readily explained if condition (v) is relaxed, all the other requirements being satisfied. With reference to Fig. 2, let us suppose that a bound donor state D^o, resonant in energy with the CB, has an energy E_d above the Γ minimum. Then, if the doping level is sufficiently high, the Fermi energy will rise until very close to E_d at the final stage of the photoionization process: a significant fraction of photoexcited electrons will then populate E_d and a fraction of donors will be in the neutral D^o charge state. Under these conditions n_H will only give the density of electrons in the extended CB states. On the other hand, the occupancy of the D^o level is expected to follow the quasistatic variations of the V bias near the edge of the space charge region of a Schottky barrier. By following the line sketched in Ref. 20 it is easy to show that, if V is larger than the V_o bias applied during cooling, n_{CV} gives approximately $n + n_{D^o}$, where $n = n_H$ and n_{D^o} are the free electron density and the electron density in the bound D^o state, in the flatband region, respectively. The data in Fig. 1 thus indicate that nearly 60% of the photoexcited electrons populate the bound state under saturated persistent photoconductivity (SPPC).

The temperature dependences of n_H and n_{CV} are reported in Fig. 3. The data were taken in the dark, after the SPPC condition was reached. They refer to a temperature range where recapture by DX centers is unobservable, as was also ascertained by checking that the curves were reversible. $n_{CV} = n_{CV}^{\mathrm{SPPC}}$ is thus constant, as expected, while n_H^{SPPC} slightly increases as the temperature decreases, thus indicating a depopulation of the bound state. The solid line gives the calculated $n_H^{\mathrm{SPPC}}(T)$ curve according to

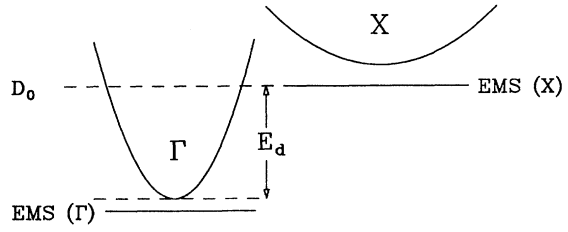

FIG. 2. Sketch of bound and band states for the examined case (not to scale).

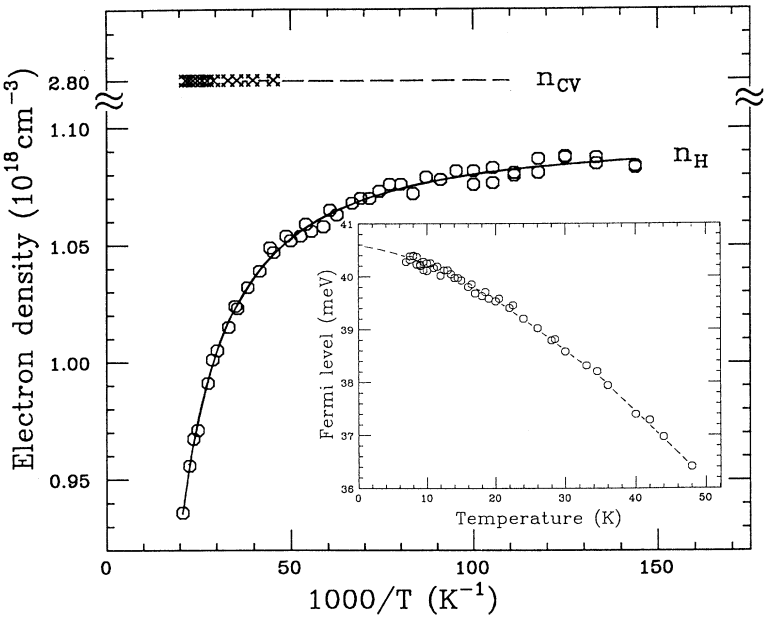

FIG. 3. Temperature dependences of n_{CV} (crosses) and n_H (circles) under saturated persistent photoconductivity (SPPC). The solid line gives the $n_H(T)$ calculated data according to Eq. (1). $E_d(0) = 40.6$ meV, $m_1 = 0.157$ meV/K, $m_2 = -1.63 \times 10^{-3}$ meV/K^2. The inset gives the temperature dependence of the Fermi energy (see text). Dashed lines are guides for the eyes.

$$n_H^{\mathrm{SPPC}}(T) = n_{CV}^{\mathrm{SPPC}} - \frac{N_d}{\frac{1}{6}\exp\left(\frac{E_d - E_F}{K_B T}\right) + 1}, \qquad (1)$$

where the N_d donor density is $N_d = n_{CV}^{\mathrm{SPPC}}/(1-\alpha)$, $\alpha = N_a/N_d$ (N_a is the acceptor density) is the compensation ratio, and a degeneracy factor $g = 6$ was included as for a hydrogenic level linked to X. However, the analysis resulted to be rather insensitive to the chosen value for g. The second term in the right-hand side of Eq. (1) represents the density of electrons trapped at the E_d donor state. Here the density of E_d states was taken equal to the N_d total donor density, since all the Si impurities are expected to be in the substitutional configuration under SPPC condition. α was chosen as equal to 1/3, as a result of analysis performed in MBE GaAs samples under similar growth conditions.[21] E_d was allowed to vary with temperature as

$$E_d(T) = E_d(0) + m_1 T + m_2 T^2 \,. \qquad (2)$$

By relating n_H^{SPPC} to E_F, Eq. (1) was self-consistently solved for $E_F = E_F(T)$ until a satisfactory agreement with the experimental n_H data was found. The best fitting procedure gave $E_d(0) = 40.6 \pm 0.5$ meV in good agreement with the $T = 0$ extrapolated value of the Fermi energy, $E_F(0) = 40.6 \pm 0.2$ meV, as shown in the inset of Fig. 3. These latter E_F data were directly obtained by the experimental n_H values through inversion of the Fermi-Dirac integral. The estimated values of m_1 and m_2 are also indicated in the figure.

Further evidence for the role of the D^o bound state can be obtained by comparing $n_{CV}(t)$ and $n_H(t)$ during isothermal capture transients. What is expected is that during the initial stage of the capture process of electrons by DX centers, as long as the fraction of neutral D^o donor is large, n_{CV} should decrease faster than n_H. In fact, the decreasing rate of $n_{CV}(t)$ is univocally determined by the rate at which the electrons are captured by DX centers, while the free electron density in the CB is expected to decrease at a slower rate since electrons are continuously supplied in the Γ valley by the depopulation of the D^o level. On the other hand, during the final stage of the capture, when D^o is empty, $n_{CV}(t)$ and $n_H(t)$ should practically coincide. This was indeed observed, as shown in Fig. 4. Only slow transients could be investigated, of course, in order that variations during the duration of a single measurement (approximately 1 min and 5 min for C-V and Hall measurements, respectively) did not introduce large errors. At $T = 60$ K [Fig. 4(a)] the $t = 0$ values of n_{CV} and n_H were close to the SPPC values and the electron capture by DX centers proceeded slowly after the LED was switched off. 300 min later, the fraction of photoexcited electrons in the D^o state was still as high as 30%. Figure 4(a) thus refers to the initial stage of the capture process, and the expected slower behavior of $n_H(t)$ compared to $n_{CV}(t)$ is clearly shown. On the other hand, at $T = 75$ K [Fig. 4(b)], the capture is considerably faster. At $t \approx 100$ min the number of photoexcited electrons is less than one order of magnitude lower than the SPPC value: $n_{CV}(t)$ and $n_H(t)$ coincide, as expected, thus indicating that the D^o state is practically empty. It is worth noting that under these circumstances, the requirements (i)–(v) for having $n_{CV} = n_H$ are all fulfilled. In particular the free electron density is still large enough to have $r_H = 1$, although not so large as to have significant population of the X and L valleys, despite the relatively high temperature. Condition (ii) is practically satisfied if only slow transients are considered, as in the present case. The fact that $n_{CV}(t)$ and $n_H(t)$ coincide during the final stages of the 75-K transient [Fig. 4(b)] can also be considered as a proof that the C-V measurements are not affected by important contributions due to bias-induced variations in the DX charge state.

The data of Fig. 4(a) can be analyzed as follows. Since the actual density of substitutional Si impurities is $N_d - N_{DX}(t)$, $N_{DX}(t)$ being the DX center density, n_{CV} and n_H are related through

$$n_{CV}(t) = n_H(t) + \frac{N_d - N_{DX}(t)}{\frac{1}{6}\exp\left(\frac{E_d - E_F(t)}{K_B T}\right) + 1} . \quad (3)$$

In the negative correlation energy ($U < 0$) approach for the DX center, $N_{DX}(t)$ is equal to one-half the density of electrons captured at the time t:

$$N_{DX}(t) = \frac{n_{CV}^{\mathrm{SPPC}} - n_{CV}(t)}{2} . \quad (4)$$

In Eq. (3) the $E_F(T)$ values were directly obtained by the experimental n_H data through inversion of the Fermi-Dirac integral. The solid line in Fig. 4(a) gives the $n_{CV}(t)$ values calculated through Eqs. (3) and (4) using the experimental $n_H(t)$ data and $E_d(T = 60\ \mathrm{K})$=49.3 meV. In this case the agreement is only fair. This last value of E_d is 12% higher than the one (44.2 meV) extrapolated at $T = 60$ K through Eq. (2) using for $E_d(0)$, m_1, and m_2 the values obtained by fitting the $n_H(T)$ data of Fig. 3. A slightly better agreement is found in the positive-U approach, where $N_{DX}(t)$ is given by twice the expression (4). The fitting procedure gave $E_d(T = 60$ K)=47.3 meV in this case. Positive- and negative-U approaches lead to small differences, the analysis being scarcely influenced by a factor of 2 in Eq. (4). Thus, the slightly better agreement of the $U > 0$ model is not significant. Like the above-mentioned discrepancies, they could easily be justified. In fact, besides the uncertainty of extrapolation, we observed that m_1 and m_2 values of Eq. (2) are somewhat dependent on the chosen value of α, while $E_d(0)$ is not. Unfortunately, the derivation of the α compensation ratio in $Al_xGa_{1-x}As$ through mobility data is rather uncertain, while the extrapolation of GaAs data to $Al_xGa_{1-x}As$ could be a point of discussion. Finally, a few simplifications introduced in the present model also have to be considered, as discussed below.

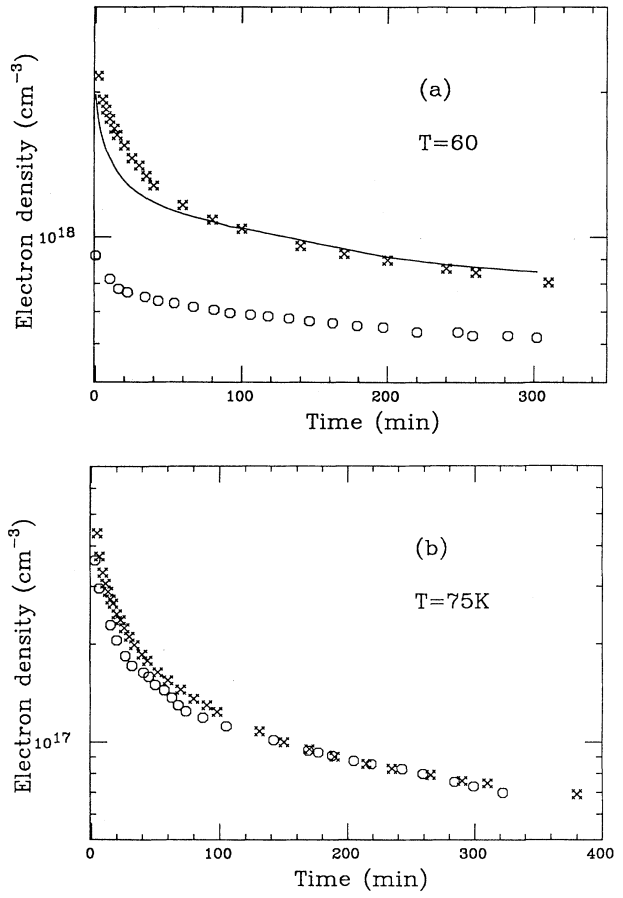

FIG. 4. Time dependences of n_{CV} (crosses) and n_H (circles) during isothermal capture transients at $T = 60$ K (a) and $T = 75$ K (b). The solid line in (a) gives the calculated n_{CV} curve according to Eqs. (3) and (4) (see text).

As indicated in Fig. 2, a bound effective-mass state EMS(X) linked to the X minima is expected to be resonant in energy with the lowest CB Γ valley in direct gap samples having x AlAs molar fractions not far from the one ($x \approx 40$) where Γ-X crossing is expected. Under these circumstances, electrons produced by low-temperature DX center photoionization will persistently populate excited bound or extended states according to the following scheme. At low electron densities (weak photoexcitation), electrons will occupy a few band states at the Γ minimum together with a fraction of bound Γ-like effective-mass states EMS(Γ). Ln(n_H) vs T^{-1} curves have negative slopes in this case, thus indicating a low-temperature freezing at a discrete EMS(Γ) level in lightly doped samples.[3] In samples with doping levels in the 10^{18}-cm^{-3} range, the analysis is complicated by mixed conduction through an impurity band originated by EMS(Γ) itself.[8,19] When the electron density exceeds the $n_c \approx 5 \times 10^{16}$ cm^{-3} critical one for the Mott transition related to the Γ-like level, a degenerate free electron gas occupies a number of band states in the Γ valley according to the Fermi-Dirac statistics: n_H is constant with temperature in this case.[8,19] At n_c the Fermi energy lies ≈ 5 meV above the Γ minimum, so that a regime of zero slope in the ln(n_H) vs T^{-1} curves can be observed only in samples where $E_d \gg 5$ meV, that is, the x value must be sufficiently smaller than the one for direct to indirect gap transition. In fact, a further increasing of the photoexcited electron density will make the Fermi energy rise until a significant population of the EMS(X) takes place. Ln(n_H) vs T^{-1} curves will have positive slopes in these cases and E_F can never exceed E_d. This latter situation has been described in this paper and a value $E_d(T = 0) = 40.6 \pm 0.5$ meV has been deduced through analysis of experimental C-V and Hall data in an $x = 0.35$ sample.

The capture of photoexcited electrons into bound donor states resonant with the Γ valley in samples having compositions similar to the present one has been hypothesized by other authors. After low-temperature DX center photoionization, von Bardeleben *et al.*[22,23] observed a persistent EPR spectrum, ascribed to an EMS(X) neutral

charge state in Te- as well as in Sn-doped samples. In Sn-doped samples a second neutral charge state linked to the L minima was also claimed by von Bardeleben *et al.*[11,12] to explain the observed EPR signal. Jantsch, Wilamowski, and Ostermayer[24] considered the low-temperature pinning of the Fermi energy observed under saturated PPC in an $x = 0.31$ Si-doped sample as due to an extra density of state resonant with the CB, although they did not give any analysis. Lavielle *et al.*[25] and Goutiers *et al.*[26] used variable hydrostatic pressure to measure photoexcited Hall electron densities under different x equivalent AlAs fractions in Si-doped samples. In particular, Goutiers *et al.*[26] analyzed their data using statistics for one donor center with possible Γ, L, and X-like states; however they interpreted the variations of slope in the $\ln(n_H)$ vs T^{-1} curves after different photoexcitation intensities as due to variations of energy of L- and X-like states with the effective density of donor shallow states. Moreover, since their measurements were made after illuminating at $T = 77$ K and during cooling to 4.2 K, their n_H vs T curves are probably affected by recapture of electrons into DX centers: for instance, we easily observed recapture just above 50 K in our $x = 0.35$ sample. In spite of all, they give for the X-like state an energy of 30 meV above Γ at the equivalent $x = 0.35$ AlAs fraction for a shallow state density in the 10^{18}-cm^{-3} range, which is not far from our result, especially when the critical role of the experimental uncertainty in the x AlAs molar fraction is considered.

Complications due to (i) the possible involving of shallow states of more than one type and (ii) a dependence of the energy on the effective density of shallow donor states were not considered in our analysis. They are unnecessary, at this stage, as they are not made evident from the experimental data, although a more extended analysis could be performed when similar data on samples having a slightly different x AlAs fraction are available. We have also neglected possible complications caused by the expected splitting of the EMS(X) level due to the uniaxial character of heteroepitaxial strain.[10,13]

As a further hint of criticism, we consider the possibility that the effects we observed are at least partially due to a significant population of the X conduction-band valley. Electrons in the X valley will have a considerably smaller mobility than in the Γ valley so that n_H values smaller than n_{CV} may result. If this were the case, however, a significant decrease in the μ_H Hall mobility would be observed as temperature increases under saturated PPC. On the contrary, μ_H was found practically constant with temperature, as expected for a degenerate free electron gas undergoing ionized impurity scattering within the Γ valley.[19] At low temperatures, μ_H was also found to increase monotonically as n_H increases, as expected. Details on the electron mobility data, together with the evidence that the capture of photoexcited electrons into the D^o donor state has a role in affecting spatial correlation effects amongst DX charges, are given elsewhere.[27]

III. CONCLUSIONS

The low-temperature occupation of a D^o bound donor state resonant in energy with the conduction band was demonstrated in $Al_xGa_{1-x}As$ ($x = 0.35$). The D^o state is due to isolated Si impurities in the simple substitutional configuration and has an energy of 40.6 ± 0.5 meV above the Γ minimum. A significant occupation of the D^o state can be achieved through low-temperature photoionization of DX centers if the doping level is sufficiently high ($N_d - N_a = 2.8 \times 10^{18}$ cm^{-3} in the present case). A high fraction of photoexcited electrons in the D^o level causes the following. (i) A higher value of the n_{CV} electron density (as derived by C-V measurements in Schottky barriers) in comparison with the n_H Hall density, when both measurements are taken under saturated persistent photoconductivity (SPPC). (ii) Different temperature dependences of n_{CV} and n_H in the SPPC regime. (iii) Different time dependences of n_{CV} and n_H during isothermal capture transients. During the initial stage, n_H decreases at a slower rate than n_{CV}, since electrons are continuously supplied in the Γ valley by the depopulation of the D^o level. During the final stage, when D^o is empty, n_{CV} and n_H coincide.

The possibility that the D^o state could be identified with the effective-mass state linked to the X secondary minima of the conduction band has been discussed.

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