

Lithium-diffused and annealed GaAs: An admittance spectroscopy studyH. G. Svavarsson,¹ J. T. Gudmundsson,^{1,2} and H. P. Gislason¹¹*Science Institute, University of Iceland, Dunhaga 3, IS-107 Reykjavik, Iceland*²*Department of Electrical and Computer Engineering, University of Iceland, Hjardarhaga 2-6, IS-107 Reykjavik, Iceland*

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We study lithium-diffused and annealed GaAs by admittance spectroscopy in the frequency range 10–10⁶ Hz and the temperature range 30–300 K. Li diffusion turns the GaAs semi-insulating but subsequent out-diffusion of Li increases the conductivity and makes the samples *p* type. It is demonstrated that the conduction in semi-insulating Li-diffused GaAs is due to thermally activated carriers in the valence-band percolating around insulating metallic precipitates. At high frequencies the ac conductivity is proportional to ω^x , with x being close to unity value, independent of temperature. We suggest that the percolation may be due to metallic precipitates formed during in-diffusion of lithium and following cooling. After subsequent annealing the ac conductivity becomes proportional to ω^s at high frequencies with the value of s decreasing with increasing temperature. The temperature dependency of s suggests a correlated barrier hopping mechanism in a band of defects. We attribute these defects to gallium vacancies V_{Ga} and gallium antisites Ga_{As} .

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I. INTRODUCTION

Although the effect of Li on the electrical properties of GaAs has been investigated intensively through the last four decades many questions still remain to be answered regarding the conduction mechanism.^{1–4} It is known that Li diffusion into GaAs reduces the free-carrier concentration which leads to electrical conductivity as low as 10⁻⁷ $\Omega^{-1} \text{cm}^{-1}$ in undoped, *n*-type and *p*-type starting materials.⁴ Lithium has a strong tendency to form complexes with other impurities and native defects when migrating in crystals.⁵ Annealing of Li-diffused GaAs at 400–500 °C in pure Ga or Ar atmosphere reduces the Li concentration by about two orders of magnitude.^{1,6} However, the most weakly bonded Li is removed by annealing at temperature as low as ~ 100 °C.⁷ The out-diffusion of Li atoms produces *p*-type material, a phenomenon first reported by Fuller and Wolfstirn,² but the mechanism responsible for the conductivity change has not been identified. The resulting *p*-type conductivity indicates the formation of acceptors which have earlier been assigned to gallium vacancies.^{6,8} Temperature dependence of the conductivity suggests that under certain conditions the electron transport occurs via hopping in a partially filled defect band.^{3,9} Annealing in the range 300–400 °C leads to hopping conduction which is observed up to room temperature, whereas annealing in the temperature range 400–450 °C leads to quasimetallic conduction.³ After annealing above 600 °C the conductivity is controlled by thermally activated valence-band conduction.³

Admittance spectroscopy is commonly used to probe the periodic electrical current response of devices and electron states in semiconductors when they are stimulated by a small alternating voltage. As such it is a direct way to characterize different types of conduction mechanism such as band conduction, hopping between localized centers, and percolation. The measurement is essentially that of electrical impedance and presents variations of a real and an imaginary component, each of which forms a frequency spectrum. At low frequencies the admittance consists of a purely real part

(conductance) but at high frequencies the admittance reduces to a purely imaginary part (susceptance).¹⁰ If the ac and the dc conduction is due to (completely) different processes then the measured total conductivity at particular angular frequency ω and temperature, $\sigma_{\text{tot}}(\omega)$, can be decomposed into two components,¹¹

$$\sigma_{\text{tot}}(\omega) = \sigma_{\text{dc}} + \sigma_{\text{ac}}(\omega), \quad (1)$$

where σ_{dc} and $\sigma_{\text{ac}}(\omega)$ are the dc and frequency-dependent (ac) conductivities, respectively. Band conduction (valence or conduction band) is characterized by a relaxation time τ which describes collisions with phonons and affects the band admittance σ_{b} as¹⁰

$$\sigma_{\text{b}}(\omega) = \sigma_{\text{b}}(0) \left(\frac{1 + i\omega\tau}{1 + \omega^2\tau^2} \right). \quad (2)$$

Here, $\sigma_{\text{b}}(0)$ is the admittance at zero frequency which resembles the dc conductivity. The ac conductivity due to hopping is known to have the form¹²

$$\sigma_{\text{ac}}(\omega) = A\omega^s, \quad (3)$$

where A is a constant and s is a parameter with value between 0 and 1. In the very high-frequency limit, electrons hop back and forth between pairs of sites for which the pair transition rates are approximately equal to the frequency.¹² Hence, this applies to situations similar to hopping of carriers between neutral and ionized defect centers in a band of defects. Transport of free carriers (electrons in a conduction band or holes in a valence band) limited by small insulating regions finely distributed in the crystal can also be described by the percolation theory but in a more macroscopical way. In such a case the semiconductor can be considered to be a composite of insulating regions around which the carriers can move. Metallic precipitates in a semiconductor may behave as buried Schottky barriers with overlapping spherical depletion regions.¹³ When the depletion regions overlap only partially the conductivity will be affected by percolation be-

TABLE I. Data for samples after Li in-diffusion at 800 °C and annealing.

Sample	Diffusion	Annealing	ρ (Ω cm)	E_a (meV)
1 GaAs:Si	Li 800 °C/4 h	20 °C	2.4×10^6	290
2 GaAs:Si	Li 800 °C/4 h	300 °C/10 h	2.5×10^2	48
3 GaAs:Zn	Li 800 °C/8 h	70 °C/2 h	1.0×10^6	230
4 GaAs:Zn	Li 800 °C/8 h	230 °C/10 h	1.1	14 (35)

havior and will likely lead to hoppinglike conductivity at low temperatures.¹³ Thus, admittance spectroscopy is especially suitable for materials in which the conductivity can be described by carriers percolating through conducting paths in an insulating matrix.^{10,14}

Admittance spectroscopy has, in the past, been applied to study percolation due to As precipitates in semi-insulating GaAs (Ref. 14) and in GaAs grown at low temperature by molecular-beam epitaxy (MBE).¹⁰ Furthermore, percolation, attributed to metallic precipitates, has been observed in melt-grown GaAs after Li diffusion at 800 °C.¹⁵ Here, we report on the temperature dependence of the ac conductivity in originally *n*-type and *p*-type GaAs samples which were lithium diffused at 800 °C and annealed at temperatures below 300 °C. We demonstrate that electron transport occurs either via hopping between localized centers or charges percolating around macroscopic insulating regions. We show that both systems respond to an alternating current in a similar way as will be shown in our result.

II. SAMPLE PREPARATION AND EXPERIMENT

Horizontal Bridgman grown Si-doped GaAs and Zn-doped GaAs samples were diffused with Li. The Si doped starting material had room-temperature carrier concentration $n = 2.5 \times 10^{16} \text{ cm}^{-3}$ and Hall mobility $\mu_H = 4000 \text{ cm}^2/\text{V s}$ while the corresponding values for the Zn-doped GaAs were $p = 2 \times 10^{16} \text{ cm}^{-3}$ and $\mu_H = 300 \text{ cm}^2/\text{V s}$. The Li diffusion was made at 800 °C for 4 and 8 h in open quartz ampoules under Ar flow with the samples and a piece of Li 99.9% metal immersed in a 6N Ga melt. The amount of Li was adjusted to be about 0.3 wt % of the melt. After Li diffusion the samples were cooled to room temperature in the melt. Pieces of the as-diffused samples were baked at 300 °C and 230 °C for 10 h. The samples investigated are listed in Table I. Finally, the samples were polished and etched before ohmic contacts were made. To avoid heating the whole sample, ohmic contacts were made by direct welding of Zn-coated gold wires onto the four corners of the square samples (typical size $3 \times 3 \text{ mm}^2$).

Direct current conductivity measurements were made between any two contacts on the sample by applying van der Pauw's method. Admittance spectroscopy measurements were performed using a HP4284A impedance analyzer. The frequency dependence of the ac conductivity was measured at several different temperatures in the range 30–300 K. For convenience the impedance analyzer was connected between two of the ohmic contacts in opposite corners and the absolute value of the admittance $|Y|$ was measured as a function

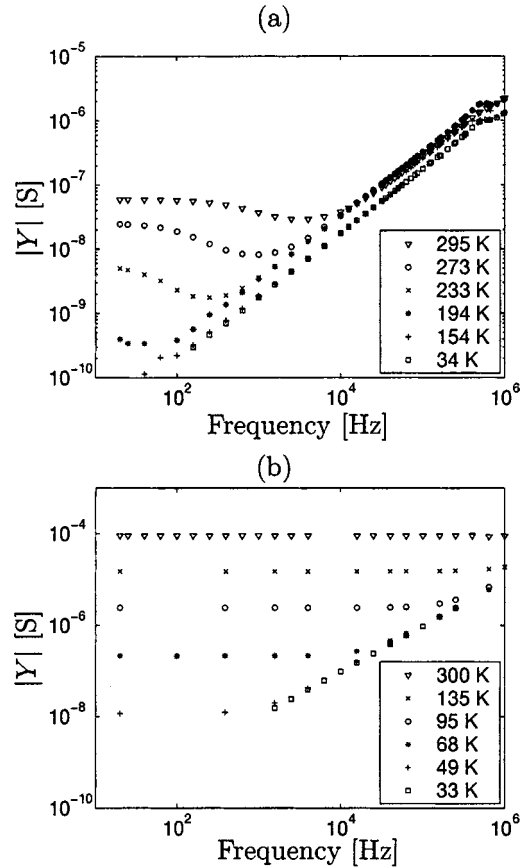


FIG. 1. The absolute value of the admittance $|Y|$ vs frequency at various temperatures for GaAs:Si; (a) Li diffused at 800 °C/4 h (sample 1) and (b) after subsequent annealing at 300 °C/10 h (sample 2).

of the frequency. Temperature-dependent Hall and conductivity measurements were made on the most highly conductive sample (sample 4). The Hall coefficient R_H was estimated from the slope of the Hall voltage versus magnetic field in the range 0–0.5 T. The apparent Hall concentrations were calculated from the Hall coefficient R_H as $p = r_H/eR_H$ assuming the Hall scattering factor to be isotropic, temperature independent, and of unity value ($r_H \equiv 1$).

III. EXPERIMENTAL RESULTS

The room-temperature transport properties of the four samples investigated are summarized in Table I. Sample 2 is a piece taken of sample 1 and annealed and sample 4 is similarly a piece taken of sample 3 and annealed. Both the *n* type and the *p* type starting materials turn semi-insulating after the Li diffusion. A subsequent annealing of the samples leaves them *p* type in both cases, as manifested by Hall measurements. The as-diffused samples have specific resistivity ρ_{dc} of the order of $10^6 \Omega \text{ cm}$ at room temperature but this value is significantly reduced by the annealing. Annealing the Zn-doped sample at 230 °C for 10 h decreases the resistivity to $1.1 \Omega \text{ cm}$ whereas annealing the Si-doped samples at 300 °C for 10 h reduces the resistivity to $250 \Omega \text{ cm}$. In Fig. 1(a) the frequency dependence of the absolute value of

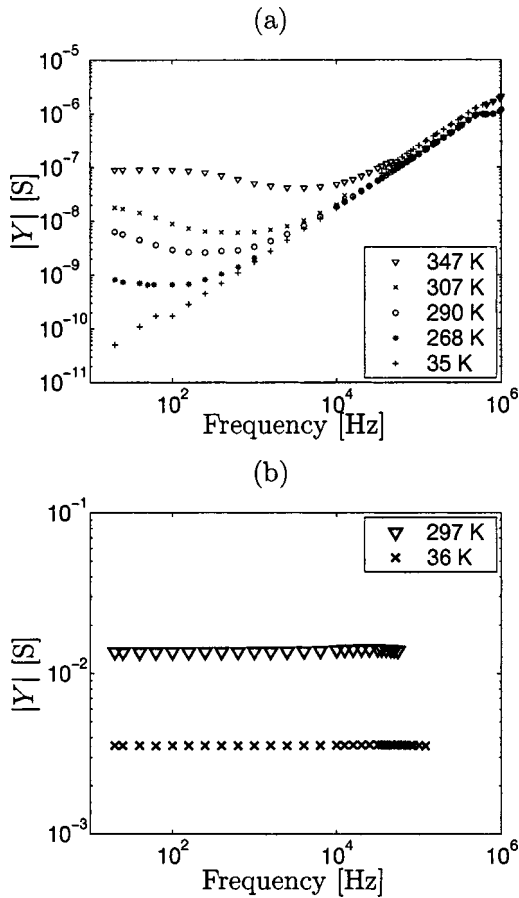


FIG. 2. The absolute value of the admittance $|Y|$ vs frequency at various temperatures for GaAs:Zn; (a) Li diffused at $800^\circ\text{C}/4\text{ h}$ (sample 3) and (b) after subsequent annealing at $300^\circ\text{C}/10\text{ h}$ (sample 4).

the admittance $|Y|$ is shown for sample 1 while similar graph for sample 2 is shown in Fig. 1(b) to illustrate the effect of annealing on the n type starting material. Both figures do in general emphasize two regimes; an essentially flat response at lower frequencies and a crossover to a nearly linear increase with increasing frequency (dispersion regime). A striking difference between the two samples is manifested in the admittance minima of the as-diffused sample 1, which disappears upon annealing. The frequency at which the minima occurs, ω_m , increases with increasing temperature. A corresponding graph for the p type starting material before and after annealing, sample 3 and sample 4, is shown in Figs. 2(a) and 2(b), respectively. There we observe features similar to those exhibited for samples 1 and 2 except that the dispersion regime of the annealed sample 4 is not reached at the highest frequency investigated.

Figure 3 shows the temperature dependence of the absolute value of the admittance for sample 2. At low temperature the conductivity is almost constant but at sufficiently high temperature the conductivity approaches the dc conductivity. It is also clearly seen that the lower the frequency the more the admittance resembles the dc conductivity. A straight-line fit to the high-temperature regime (the dc conductivity) of Arrhenius plots gives activation energies 290 and 230 meV

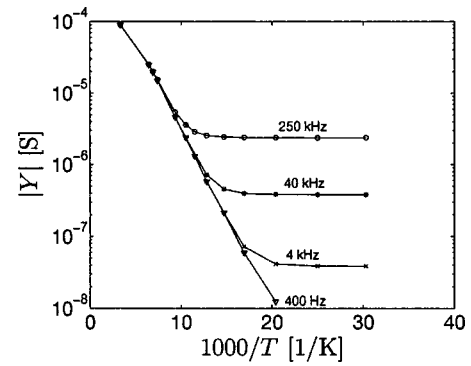


FIG. 3. Temperature dependence of the absolute value of the admittance $|Y|$ for GaAs:Si, Li in-diffused at 800°C for various frequencies and annealed at 300°C for 10 h (sample 2).

for the as-diffused samples 1 and 3 and 48 and 14 meV for the annealed samples 2 and 4, respectively. From Hall measurements the slope of $\ln(pT^{-3/2})$ vs T^{-1} gives a value of 35 meV for sample 4, which was the only sample with high enough conductivity for temperature-dependent Hall measurements.

IV. DISCUSSION

The solid solubility of Li in GaAs is roughly 10^{19} cm^{-3} at 800°C .² After Li diffusion at 800°C the total Li concentration, measured by secondary-ion mass spectroscopy (SIMS), is roughly 10^{19} cm^{-3} but decreases to $\sim 10^{16}\text{ cm}^{-3}$ after annealing at 400°C for 20 h.⁸ Since the samples investigated in this study are Li diffused at 800°C for a prolonged period of time the solubility level is expected to indicate the concentration of Li atoms or ions in the as-diffused samples. A fraction of the Li atoms is expected to compensate the original dopant of the as-grown material ($\sim 2 \times 10^{16}\text{ cm}^{-3}$) but the excess Li neutralizes native defects or autocompensates, which leaves the material semi-insulating. It has been suggested that lithium passivates shallow acceptors in GaAs but compensates shallow donors.⁶ SIMS measurements have shown that after Li in-diffusion at 800°C for 8 h and subsequent out-diffusion at 400°C for 7 h the concentrations of the $[\text{Ga}_{\text{As}}]$ and $[\text{V}_{\text{Ga}}]$ defects are $3.2 \times 10^{17}\text{ cm}^{-3}$ and $7.6 \times 10^{17}\text{ cm}^{-3}$, respectively.¹⁶ The presence of ionized interstitial Li^+ donors is believed to stabilize the gallium antisite and the gallium vacancy by forming neutral complexes such as $\text{Ga}_{\text{As}}^--\text{Li}^+$ and $\text{V}_{\text{Ga}}^--\text{Li}^+$.⁸ During the out-diffusion of Li, or annealing, these pairs break up, leaving Ga_{As} and V_{Ga} , either isolated or as complex defects.⁸

By looking at Figs. 1–3, we see that the dc conductivity is a much stronger function of temperature (exponential) than the ac conductivity (a power law) which emphasizes that a different mechanism is at work in these two regimes. It can also be seen from Figs. 1 and 2 that the dispersion sets in at frequency proportional to the dc conductivity.

In p -type material, whose conductivity is governed by conduction in a defect band, a consequence of the variation in the acceptor-acceptor separation is that some paths, for a hole traveling between two contacts, are open while others

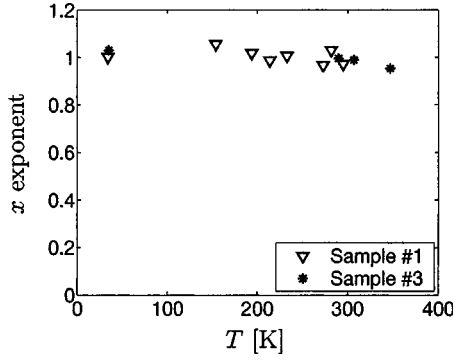


FIG. 4. Temperature dependence of the frequency exponent x for the Li in-diffused samples 1 and 3.

are closed. The general motion of a carrier in the defect band, in the presence of an electric field, is then in the nature of a percolation process. As a result of the percolation current flow, only a part of the total volume contributes to the (low-temperature) dc conduction. Therefore the contributing volume of the samples, and hence the conductivity, can be increased dramatically with alternating current.¹⁷ As the temperature increases and the defects are ionized, conventional band conduction gradually takes over and becomes the dominating conduction mechanism usually well below the room temperature. The band conduction is considered to be more or less independent of frequency, in contrast to conduction mechanism such as percolation. In a macroscopic percolation system the conductivity is only a function of the dimension of the system and thus can be expressed in terms of a scaling law $\sigma \propto \omega^x$ with a universal exponent x .¹⁰ A closer look at the data shows that the temperature dependence of the frequency exponent is different for the as-diffused samples and the annealed samples. Therefore, different exponents are defined for these two cases. In case of the as-diffused samples we refer to the exponent as x , but after subsequent annealing we refer to it as s . The values of the exponent x for the as-diffused samples 1 and 3, shown in Fig. 4, were obtained from straight-line fits in the high-frequency dispersion regime shown in Figs. 1(a) and 2(a). We found that x had a constant value close to unity throughout the whole temperature regime investigated. However the exponent s , for the annealed sample 2, shows a very strong temperature dependence, as seen in Fig. 5.

A. Percolation

The existence of a macroscopic percolation regime in the as-diffused samples 1 and 3 is explored further in Fig. 6 where a linear relationship between ω_m and σ_{dc} (the flat part of the measured admittance absolute value) is seen, which is clear indication of a conduction by carriers percolating around macroscopic insulating regions. It may therefore be concluded that metallic precipitation is present after the Li diffusion at $T=800^\circ\text{C}$ and subsequent quenching to room temperature. In an admittance spectroscopy study of low-temperature grown GaAs, Khirouni *et al.* attributed a distinct minimum in the $\sigma(\omega)$ curve to a percolation regime caused by precipitation of As in a metalliclike islands.¹⁰ A similar

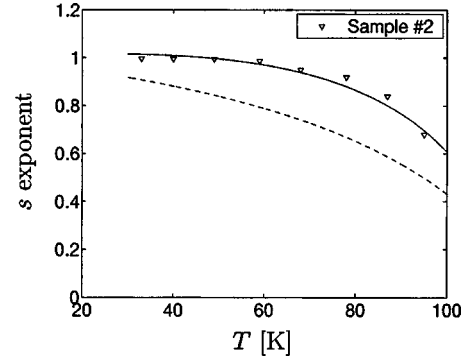


FIG. 5. Temperature dependence of the frequency exponent s for Li-diffused and annealed sample (sample 2). The dotted line is a fit plotted from Eq. (7) with $W_m=230$ meV and $\omega\tau=10^{-7}$. The solid line is a fit to the modified equation (8).

minimum is seen in our Figs 1(a) and 2(a). We suggest that the percolation may be due to GaLi precipitation during in-diffusion of Li and subsequent cooling to room temperature. Preliminary transmission electron microscope study on GaAs after Li diffusion at 800°C revealed a lattice dimension of clusters which matched that of a GaLi crystal while the existence of As and Li precipitates were ruled out.¹⁸ A precipitation of GaLi would be in the form of small clusters finely distributed in the matrix which would act as Schottky barriers and induce space-charge regions around them. Such buried Schottky barriers with overlapping spherical depletion regions have been observed in semi-insulating low-temperature MBE grown GaAs.¹³ Based on the idea of Sarychev and Brouers,¹⁹ of hopping-assisted tunneling, Khirouni *et al.*¹⁰ derived the following equation for the frequency-dependent admittance in a macroscopic percolation system;

$$\sigma(\omega) = \sigma(0) \frac{\sqrt{1 + \omega^6 \theta^6}}{1 + \omega^2 \theta^2}, \quad (4)$$

in which τ has been replaced by θ , a macroscopic relaxation time characterizing the electron scattering by insulating (depleted) regions around which the electrons percolate. When these depleted regions overlap, the transport of carriers is retarded. A plot of $\sigma(\omega)$ given by Eq. (4) exhibits a minimum for the frequency ω_m , where $\omega_m \theta = 0.856$.¹⁰ A combi-

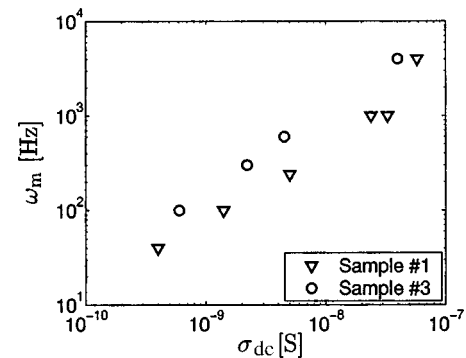


FIG. 6. Variation of the frequency at admittance minimum with dc conductivity of the as-diffused samples 1 and 3.

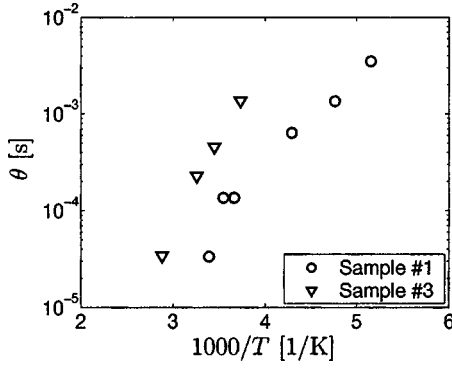


FIG. 7. Temperature dependence of the relaxation time θ for the as-diffused samples 1 and 3.

nation of the simple relation $\omega_m\theta=0.856$ and the data of Figs. 1(a) and 2(a) is used to probe the temperature dependence of θ . An Arrhenius plot of θ for samples 1 and 3, shown in Fig. 7, indicates that θ is an exponential function of the inverse temperature. A straight-line fit to the data according to

$$\theta \propto \exp(\Delta E/k_B T)$$

gives the activation energy $\Delta E \sim 340$ and 200 meV, for samples 1 and 3, respectively. These values should correspond to the thermal activation energies of 290 and 230 meV obtained from the dc conductivity^{20,21} for samples 1 and 3, respectively, and in fact the energies are in qualitative agreement with each other.

B. Hopping conductivity

The values of the exponent s for sample 2, shown in Fig. 5, were obtained from a straight-line fit in the high-frequency dispersion regime shown in Fig. 1. It was not possible to obtain values of s for the other annealed sample, 4, since the conductivity had a constant value throughout the whole frequency regime investigated, even at the lowest temperature (36 K). A strong temperature dependence of s is observed. The value decreases with increasing temperature, from being close to unity at the lowest temperature, which suggests that the ac conduction mechanism can be described by hopping of carriers between localized centers in accordance with the correlated barrier hopping (CBH) model. A polynomial fit to the values of s for sample 2 (in Fig. 5) extrapolates to $s=0$ at $T=107$ K. This indicates that hopping conduction sets in below ~ 110 K, which is in good agreement with previous experiments on Li diffused and annealed GaAs.⁹ Based on this and the observed activation energies of 14–48 meV we conclude that the defect band in the annealed samples is made up of shallow acceptors 20–50 meV above the valence band.

The physical meaning of the activation energies 290–340 and 200–230 meV, of the as-diffused samples 1 and 3, is not clear. One interpretation would be the presence of deep Li-related acceptors with a very small concentration (keeping in mind that the resistance of the as-diffused samples are four to six orders of magnitude higher than that of the annealed

samples). Hopping conduction is impossible in an empty or a full defect band. All native defects and impurities in the samples are neutralized by Li during the Li-diffusion phase, which explains why no hopping-conduction is observed prior to annealing. After annealing a portion of the Li is removed, creating a partially filled defect band, in view of the hopping conduction observed. A native single acceptor located 68 meV above the valence-band edge in p -type GaAs is known to exist in Ga-rich GaAs and is believed to be due to the $V_{\text{Ga}}\text{-Ga}_{\text{As}}$ pair.²² $\text{Ga}_{\text{As}}^{0/-}$, $\text{Ga}_{\text{As}}^{-/-}$, and $V_{\text{Ga}}^{0/-}$ have energy levels 80, 200, and 140 meV above the valence band, respectively.²³ Zn is a typical shallow acceptor in GaAs with a level ~ 35 meV above the valence band. However, a band of such defects would be closer to the valence band due to band broadening.

The CBH model describes charge carrier hops between sites over the potential barrier separating them.²⁴ For single-polaron hopping the barrier height W is related to the distance between the hopping sites R as

$$W(R) = W_m - \frac{e^2}{\pi \epsilon \epsilon_0 R}, \quad (5)$$

where W_m is the maximum barrier height (i.e., at infinite site separation) and ϵ the bulk dielectric constant. In the case of classical hopping of carriers over a potential barrier W separating two energetically favorable sites the relaxation time is

$$\tau = \tau_0 \exp(W/k_B T), \quad (6)$$

where τ_0 is a characteristic relaxation time (assumed to be an inverse optical phonon frequency $\sim 10^{-13}$ s).²⁴ Based on the CBH model, the frequency exponent s depends on the temperature as²⁴

$$s = 1 - \frac{6k_B T}{W_m + k_B T \ln(\omega \tau_0)}. \quad (7)$$

When $W_m \gg k_B T$ the frequency exponent s is practically independent of frequency and mainly dependent on temperature. When obeying this law, the hopping regime can easily be recognized by the temperature dependence of s ; the value of s should decrease with increasing temperature. In Fig. 5, Eq. (7) is fitted to our data using W_m as the fitting parameter, $\omega = 10^6$ s⁻¹ and $\tau_0 = 10^{-13}$ s (the dotted line). The closest fit was obtained with $W_m = 230$ meV but the result was at its best only in fair agreement with our data.

The CBH model assumes randomly distributed defect centers, but if one assumes paired defects instead Eq. (7) has to be modified. A correction term of the form T/T_0 , where T_0 is a constant associated with a transition to a different conduction mechanism, may be added to Eq. (7). A similar modification has been done for chalcogenic glasses where T_0 is associated with the glass-transition temperature.²⁴ The equation can then be written as

$$s = 1 - \frac{6k_B T}{W_m + k_B T \ln(\omega \tau_0)} + \frac{T}{T_0}. \quad (8)$$

By using $T_0 = 350$ K, $W_m = 230$ meV, and $\omega \tau = 10^{-7}$, we obtain a reasonable good fit to the data represented in Fig. 5

as shown by the solid line. The physical interpretation of such a correction term in our case is, however, vague and should be taken with precaution.

To summarize, deep defects located 200–340 meV above the valence band are created during diffusion of Li into GaAs. During cooling of the as-diffused samples a metallic precipitation occurs. Hence the conduction mechanism in the as-diffused samples is characterized by thermally activated holes percolating around insulating regions of the metallic precipitates. In the annealing process a portion of the Li is removed and a partially filled band of shallow defect levels, whose wave functions overlap, is invoked. This gives rise to a hopping-conduction mechanism which dominates the conductivity below roughly 100 K.

V. CONCLUSION

We report on measurements of the frequency-dependent conductivity in lithium-diffused and annealed GaAs. Li diffusion into GaAs reduces the free-carrier concentration and makes the material semi-insulating. Positron annihilation spectroscopy shows that both gallium vacancies V_{Ga} and gallium antisite defects Ga_{As} are formed during the in-diffusion of Li and increase in concentration upon out-diffusion of Li (annealing).¹⁶

We demonstrate that the dominating conduction mecha-

nism in the as-diffused samples is different from that of the annealed samples: After Li diffusion, the conduction takes place in the valence band in which free holes, provided by thermal activation of deep levels, percolate around insulating regions. The presence of percolation indicates that the material contains metallic precipitates and buried Schottky depletion regions. We suggest that the percolation may be due to GaLi, precipitating during in-diffusion of lithium and following cooling. After subsequent annealing the conduction occurs in a partially filled defect band in which electrons move by hopping between occupied and empty defects. We attribute the defect band to Ga_{As} and V_{Ga} defects, located 20–50 meV above the valence band. At high frequency the ac conductivity is proportional to ω^s . The value of s decreases with increasing temperature suggesting a CBH mechanism. A fit to our data, using the CBH model, was, however, only in a qualitative agreement with the theory and the possibility of more than one transport mechanisms operating simultaneously should be borne in mind.

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