

# The effects of temperature and oxygen concentration on the photoluminescence of epitaxial metalorganic vapor-phase epitaxy GaAs:O

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Semi-insulating epitaxial GaAs:O prepared in a metalorganic vapor-phase epitaxy growth process using DEALO  $[(C_2H_5)_2AlOC_2H_5]$  as the oxygen source has been characterized by temperature-dependent (12–300 K) photoluminescence. Oxygen-related deep level photoluminescence bands were detected at  $\sim 0.8$  and  $\sim 1.1$  eV. The relative intensities of the two bands were sensitive to both oxygen concentration and temperature. At a given temperature, an increase in oxygen concentration led to an increase in the intensity of the lower energy band relative to the higher energy band. A similar effect occurred at a given oxygen concentration as the temperature was raised. Band edge luminescence was also measured and was observed to quench when the oxygen concentration exceeded  $\sim 10^{18}$  cm<sup>-3</sup>. The results indicate that oxygen is incorporated differently in epitaxial GaAs than in bulk GaAs. We propose that the difference is due to the incorporation of Al when DEALO is used in the growth of epitaxial GaAs:O. We suggest equally plausible microscopic models, based on the number of nearest-neighbor Al associated with O and multiple charge states, to explain the properties of the oxygen-related photoluminescence.

## I. INTRODUCTION

Semi-insulating semiconducting materials are of fundamental interest as well as being important in the fabrication of several device structures. These materials can assist in current confinement in the case of buried heterostructure lasers and improve deleterious backgating and sidegating of electronic device structures. Such semi-insulating materials are generally prepared by introducing deep levels into semiconductors. Such deep levels can compensate carriers derived from shallow impurities and can result in the subsequent formation of very high-resistivity materials. Both bulk and epitaxial semi-insulating GaAs have been prepared and used in these device applications. Conventional routes to the formation of bulk semi-insulating GaAs include transition-metal doping (most commonly Cr)<sup>1,2</sup> and EL2 incorporation.<sup>3-5</sup> Bulk semi-insulating GaAs prepared from either method suffers property limitations that have restricted its application in many device applications. Transition-metal-doped GaAs is problematic because of the high dopant diffusion coefficient, low dopant solubility, and thermal instabilities.<sup>6,7</sup> In the case of EL2-containing GaAs, the maximum achievable EL2 concentration is not sufficient for many applications.

Epitaxial semi-insulating GaAs can also be prepared by transition-metal doping,<sup>8,9</sup> but it possesses the same practical disadvantage as bulk transition-metal-doped GaAs. Epitaxial semi-insulating GaAs can be formed in the molecular-beam epitaxy (MBE) based growth. The use of very low MBE growth temperatures results in the formation of nonstoichiometric GaAs.<sup>10</sup> In this growth process, excess As is incorporated, which results in the formation of As precipitates in the

epitaxial GaAs layer after annealing. These As precipitates are thought to lead to the semi-insulating behavior.<sup>11,12</sup> This material has been successfully used to isolate GaAs-based transistor structures,<sup>10</sup> but it experiences thermally stimulated out diffusion of the excess As unless appropriate buffer layers, serving as diffusion barriers, are employed.

The growth of low-temperature nonstoichiometric GaAs by metal organic vapor phase epitaxy (MOVPE) is extremely difficult due to the high pyrolysis temperatures associated with the conventional MOVPE As sources. Recently, attention has turned toward the use of epitaxial oxygen-doped GaAs for semi-insulating applications. The existence of oxygen as a defect in bulk GaAs has been known for some time.<sup>13,14</sup> The low solubility ( $\sim 10^{15}$  cm<sup>-3</sup>) of oxygen in bulk GaAs has made it difficult to unambiguously ascertain the effects of oxygen on the properties of GaAs. Only recently has the relationship of oxygen to the semi-insulating properties of bulk GaAs been elucidated. Localized vibrational mode studies<sup>15-18</sup> revealed the presence of two oxygen-related defects in GaAs, the interstitial and off-center substitutional defects. Interstitial oxygen is electrically inactive. In the off-center substitutional defect, O- $V_{As}$ , oxygen moves from the central position of a tetrahedral As site toward two neighboring Ga atoms and molecular Ga—O—Ga and Ga—Ga bonds are formed. The O- $V_{As}$  defect has negative- $U$  behavior and possesses three charge states which introduce band-gap states located 0.15 and 0.58 eV below the conduction-band edge.

Our present work focuses on the characterization of MOVPE-grown epitaxial oxygen-doped GaAs for semi-insulating applications. Recent studies have indicated that oxygen can be reproducibly incorporated into epitaxial MOVPE GaAs at concentrations far in excess of those found in bulk GaAs.<sup>19-22</sup> The oxygen precursors DMALO

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TABLE I. The concentrations of oxygen and aluminum determined from SIMS analysis of the samples discussed in this study and the V/III ratio used in the growth of each sample.

Sample	[O](cm <sup>-3</sup> )	[Al](cm <sup>-3</sup> )	(V/III)ratio
1	2×10 <sup>16</sup>	7×10 <sup>17</sup>	40
2	3×10 <sup>17</sup>	1×10 <sup>18</sup>	40
3	3×10 <sup>18</sup>	3×10 <sup>18</sup>	40
4	3×10 <sup>19</sup>	5×10 <sup>18</sup>	40
5	1×10 <sup>20</sup>	2×10 <sup>19</sup>	100

[(CH<sub>3</sub>)<sub>2</sub>AlOCH<sub>3</sub>]<sup>19-21</sup> and DEALO [(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>AlOC<sub>2</sub>H<sub>5</sub>]<sup>22</sup> have been demonstrated to controllably introduce oxygen concentrations ranging from about 10<sup>15</sup> to 10<sup>20</sup> cm<sup>-3</sup> into GaAs. Deep level transient spectroscopy (DLTS) measurements<sup>20-22</sup> have indicated that deep levels accompany the incorporation of oxygen. Correspondingly high resistivities (>10<sup>9</sup> Ω cm) have been achieved.<sup>23</sup>

Little is known about the microscopic nature and energy-level structure of oxygen-related defects in DMALO- or DEALO-based MOVPE GaAs or about the relationship between oxygen-related defects in MOVPE and bulk GaAs. Previous studies have demonstrated that both Al and O are incorporated into epitaxial GaAs when DMALO or DEALO is used as the source of oxygen.<sup>19-22</sup> The atomic arrangement of Al and O, forming the defect itself, remains an open question. Since Al is present in DMALO- or DEALO-based epitaxial GaAs:O, we expect a different defect structure than that reported for bulk GaAs:O.<sup>16-18</sup> We are pursuing the nature of oxygen incorporation in epitaxial GaAs:O in ongoing characterization studies. In this paper, we report the results of temperature-dependent photoluminescence studies of epitaxial GaAs:O. Both near-infrared (near-IR) and band-edge photoluminescence (PL) spectra have been measured.

## II. EXPERIMENT

Samples were grown in a conventional horizontal low-pressure (78 Torr) reactor using (CH<sub>3</sub>)<sub>3</sub>Ga, AsH<sub>3</sub>, and DEALO with H<sub>2</sub> as the carrier gas. All samples were grown at 600 °C and consisted of a 5 μm GaAs:O layer atop a 0.1 μm layer of a nominally undoped GaAs buffer layer on a SI GaAs substrate.<sup>22</sup> The concentrations of oxygen and aluminum in each sample were determined by secondary-ion-mass spectroscopy (SIMS) analysis and are summarized in Table I along with the AsH<sub>3</sub> to (CH<sub>3</sub>)<sub>3</sub>Ga ratio used in the growth of each sample.

PL was excited with an argon-ion laser and detected with a 1 m monochromator equipped with either a Ge or PbS detector. PL spectra were collected using standard lock-in amplifier techniques. All spectra were corrected for detector and system response. A photoquenching of the oxygen-related near-IR PL was observed when excitation intensities above ~15 W/cm<sup>2</sup> were used. As a consequence, the PL spectra reported in this paper were obtained using an excitation intensity of 2 W/cm<sup>2</sup>. The samples were mounted in a variable-temperature cryostat and measurements were obtained over the range 12–300 K.

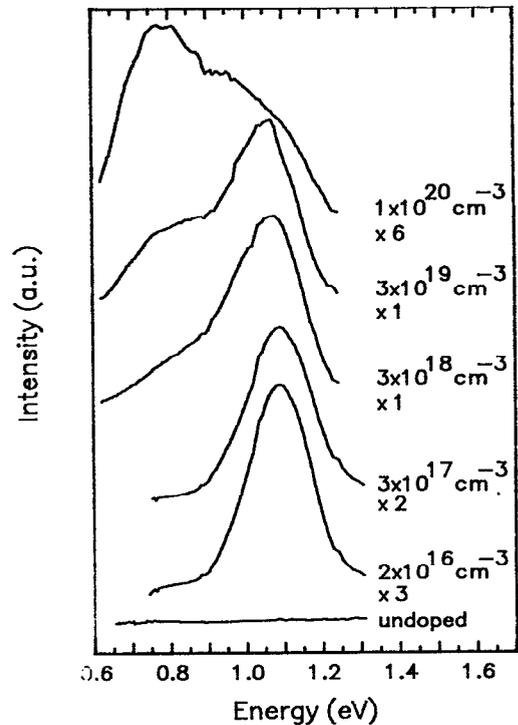


FIG. 1. The effect of oxygen concentration on the deep level photoluminescence of epitaxial MOVPE GaAs:O at 12 K. At low concentrations the spectrum is dominated by a peak at ~1.1 eV, which shifts to slightly lower energy as the oxygen concentration is increased. At higher oxygen concentrations the spectrum is dominated by a ~0.8 eV peak, which does not shift as the oxygen concentration is varied.

## III. RESULTS

The near-IR PL spectra at 12 K for the series of five epitaxial GaAs:O samples are shown in Fig. 1. The spectrum of a nominally undoped epitaxial sample of GaAs is also shown. Broad discernible PL bands located at approximately 0.8 and 1.1 eV were observed in the near-IR spectra obtained from the oxygen-doped samples. Pronounced intensity effects were observed as the oxygen concentration was varied. At low oxygen concentrations, the higher energy PL band dominated the spectrum. As the oxygen concentration was increased, the lower energy PL band gained intensity until, in the highest concentration sample, the lower energy PL band dominated the spectrum.

Figure 1 indicates that small changes in PL peak position of the lower energy band accompanied changes in oxygen concentration. The lower energy PL band first appeared as a resolvable shoulder at ~0.8 eV when the oxygen concentration was ~10<sup>18</sup> cm<sup>-3</sup> and became more pronounced at higher oxygen concentrations. The overlap of the higher energy PL band with the lower energy PL band prevented us from accurately quantifying the PL peak position of the lower energy PL band. No shift of the lower energy PL band with increasing oxygen concentration was apparent. Since the higher energy PL band was resolved over a wider range of oxygen concentrations we were better able to quantify its shift with oxygen concentration. The maximum of the higher energy PL band was located at ~1.09 eV when the oxygen concen-

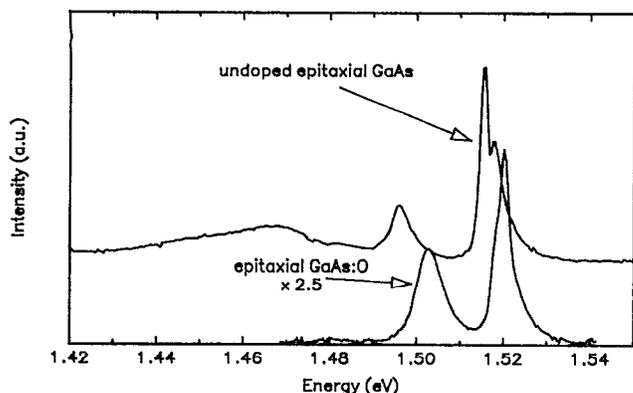


FIG. 2. Band-edge photoluminescence spectra of nominally undoped and oxygen-containing ( $2 \times 10^{16} \text{ cm}^{-3}$ ) epitaxial MOVPE GaAs samples. The spectrum of the undoped sample consists of  $(e,h)$ ,  $(D,X)$ , and  $(e,C)$  transitions. The spectrum of the oxygen-doped sample consists of two peaks.

tration was  $\leq 10^{16} \text{ cm}^{-3}$ . A continuous shift of the higher energy PL band from  $\sim 1.09$  to  $\sim 1.06$  eV occurred as the oxygen concentration was raised from  $\sim 10^{16}$  to  $\sim 10^{19} \text{ cm}^{-3}$ . The significant overlap of the two PL bands in the  $\sim 10^{20} \text{ cm}^{-3}$  sample makes it difficult to unambiguously determine the position of the higher energy PL band. A Gaussian deconvolution of the spectrum of the  $\sim 10^{20} \text{ cm}^{-3}$  sample provides an estimated position of 1.04 eV for the higher energy PL band.

We have also measured band-edge PL in oxygen-doped and nominally undoped epitaxial GaAs. Near-edge features at 1.518, 1.515, and 1.496 eV were observed in the nominally undoped sample. These peaks are illustrated at 12 K in Fig. 2 and are due to band-to-band,  $(D,X)$ , and  $(e,C)$  transitions, respectively.<sup>24</sup> Two near-edge features were observed in the samples with oxygen concentrations of  $2 \times 10^{16}$  and  $3 \times 10^{17} \text{ cm}^{-3}$ . At 12 K, peaks at 1.520 and 1.503 eV were observed in both samples. By analogy to the nominally undoped sample, these peaks are most likely due to excitonic and band-to-carbon transitions. Figure 2 illustrates these features at 12 K. A slight shift in the energies of these peaks relative to the nominally undoped sample was observed. The shift was too large to be accounted for by the Al concentration obtained from our SIMS results. Double crystal x-ray diffraction measurements indicated that our samples are strain-free, so the shift is not due to strain. At the present time, we are uncertain of the origin of the shift and, hence, the peak assignments described above should be viewed as tentative. In both oxygen-doped samples, an increase in temperature resulted in a quenching of the lower energy carbon peak relative to the higher energy excitonic peak. The band-edge PL in both samples was quenched by  $\sim 50$  K. No band-edge PL was observed in samples with an oxygen concentration above  $\sim 10^{18} \text{ cm}^{-3}$  under our experimental conditions.

Significant intensity effects were observed in the samples as the temperature was varied. Regardless of the oxygen concentration, a strong quenching of the higher energy PL band was observed as the temperature was raised. Weaker quenching of the lower energy PL band was observed. Figure 3 illustrates the effect of temperature on the near-IR PL of epitaxial GaAs containing  $3 \times 10^{18} \text{ cm}^{-3}$  oxy-

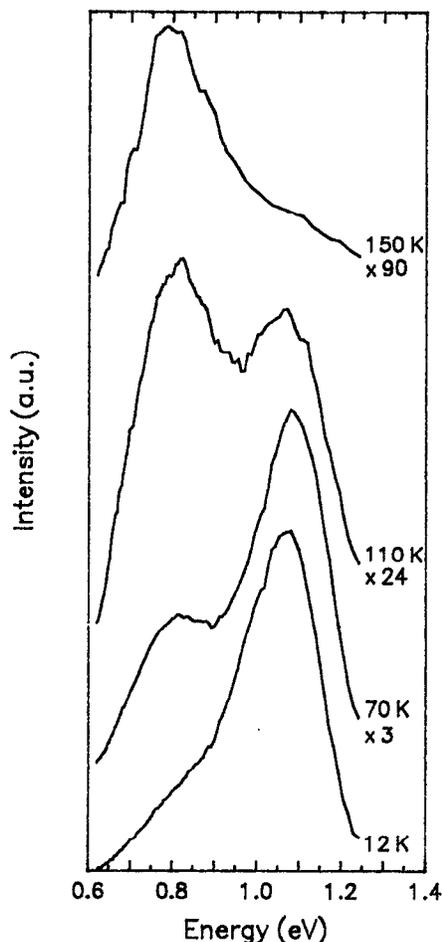


FIG. 3. The effect of temperature on the deep level photoluminescence of epitaxial MOVPE GaAs:O containing  $3 \times 10^{18} \text{ cm}^{-3}$  oxygen. The spectrum is dominated by the  $\sim 1.1$  eV peak at low temperature. The  $\sim 0.8$  eV peak increases in relative intensity and eventually dominates the spectrum as the temperature is increased.

gen. At 12 K, the higher energy band dominated the spectrum and the lower energy band appeared as a shoulder. As the temperature was raised, a quenching of the higher energy band relative to the lower energy band occurred until, at temperatures above  $\sim 100$  K, the lower energy band was the most intense band in the spectrum. A similar intensity reversal was observed above  $\sim 85$  K in the sample with an oxygen concentration of  $2 \times 10^{19} \text{ cm}^{-3}$ . The lower intensity reversal temperature for the  $2 \times 10^{19} \text{ cm}^{-3}$  sample is consistent with the higher intensity of the lower energy PL band relative to the  $3 \times 10^{18} \text{ cm}^{-3}$  sample at 12 K. In the  $1 \times 10^{20} \text{ cm}^{-3}$  sample, the lower energy PL band was already more intense at 12 K and became increasingly more pronounced as the temperature was increased.

No evidence of the lower energy PL band was seen in the  $3 \times 10^{17} \text{ cm}^{-3}$  sample, at 12 K. We were able to observe the lower energy band above  $\sim 80$  K, due to the quenching of the higher energy band, as illustrated in Fig. 4. The weak signal strength required the use of the more sensitive Ge detector for the measurement of the lower energy band. As a consequence, we were unable to measure the complete band, since the lower energy portion of the band is outside of the

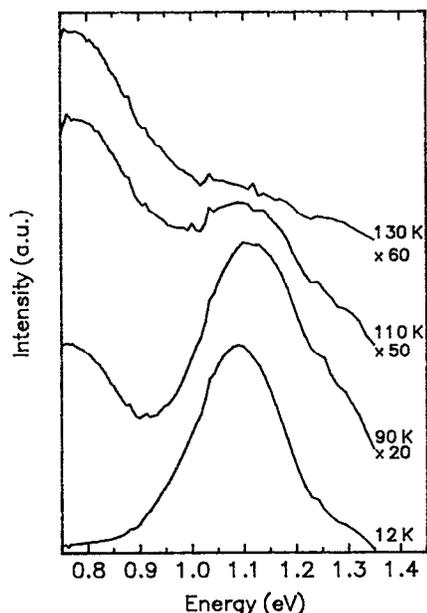


FIG. 4. The effect of temperature on the deep level photoluminescence of epitaxial MOVPE GaAs:O containing  $3 \times 10^{17} \text{ cm}^{-3}$  oxygen. At 12 K, the  $\sim 0.8 \text{ eV}$  peak is not present. The development and growth of the  $\sim 0.8 \text{ eV}$  peak with increasing temperature is evident.

range of the Ge detector. The relative intensity of the lower energy band increased with increasing temperature above 80 K as the higher energy band continued to quench. A similar effect was observed in the  $2 \times 10^{16} \text{ cm}^{-3}$  sample.

In all samples, complete thermal quenching occurred above  $\sim 100 \text{ K}$  for the higher energy PL band and above  $\sim 150 \text{ K}$  for the lower energy PL band. No shift of either near-IR PL band was observed with temperature in any of the measured samples, within our ability to identify peak maxima.

We also observed a photoquenching of the near-IR oxygen-related PL, as noted above. The photoquenching was induced by exposing the samples to high intensity argon laser or focused white light excitation. The quenching led to a reduction in PL intensity which varied with time until stabilization was achieved. Under an incident flux of  $50 \text{ W/cm}^2$ , stabilization was observed after an exposure time of 15 min. The magnitude of the quenching varied with the excitation intensity. Additionally, greater quenching was observed for the higher energy PL band than for the lower energy PL band. The lower energy PL band became increasingly prominent as the quenching proceeded.

#### IV. DISCUSSION

Our experimental results can be viewed in terms of two possible microscopic models of the GaAs:O defect. Within the first proposed model, the presence of two discernible PL bands in the near-IR may suggest that two structurally different electrically active oxygen-related defects are present in epitaxial GaAs:O. This would contrast the situation in bulk GaAs:O where only one electrically active oxygen-related defect has been reported.<sup>16–18</sup> The near-IR PL spectrum of bulk GaAs:O contains only one feature (a broadband

centered at  $0.63 \text{ eV}$ ) attributable to oxygen.<sup>14,25</sup> We do not observe such a PL peak at  $0.63 \text{ eV}$  in DEALO-based epitaxial GaAs. Differences in the DLTS spectra of bulk (LEC)<sup>26</sup> and DEALO-based epitaxial<sup>22,23</sup> GaAs:O have also been reported. These observations lead us to conclude that oxygen is incorporated differently in bulk and DEALO-based epitaxial GaAs.

The most likely source of structural variability in epitaxial GaAs:O results from the co-incorporation of Al with the O in our growth technique. The strong Al—O bond should result in the direct bonding of at least one Al for each oxygen incorporated into the layer. In other words, DEALO introduces oxygen in the form of an Al—O moiety in which it is reasonable to expect that O is nominally associated with an As site and Al occupies a Ga site. Supporting evidence for the association of O and Al includes the absence of a localized vibrational mode due to free-substitutional Al in DMALO-grown GaAs:O.<sup>27</sup> At low oxygen concentrations, we expect the individual Al—O moieties to be isolated from each other and the principle oxygen-related defect to consist of O bonded to three Ga and one Al. In the context of such a structural variability model, the low concentration PL data indicate that the higher energy near-IR PL band is associated with this proposed configuration of oxygen.

We expect that, as the oxygen concentration is increased, some of the Al—O moieties are no longer isolated and that a new oxygen-related defect could develop in which the O atom is locally associated with two Ga and two Al nearest neighbors. In this model, the lower energy near-IR PL band is attributed to this new defect. The continuous intensity increase observed for the lower energy near-IR PL band with increasing oxygen concentration would reflect the increased occurrence of this new defect configuration. The PL spectra of Fig. 1 indicated that oxygen concentrations on the order of  $\sim 10^{19} \text{ cm}^{-3}$  are required to observe an appreciable concentration of the new defect.

The suggestion that two (or more) structurally distinct Al—O defects are present in our samples is strongly supported by data reported for the DEALO growth process used to prepare our samples.<sup>22</sup> These data reveal a nonlinear dependence of the concentration of O on the concentration of Al. The concentration of Al was reported to vary linearly with the mole fraction of DEALO used in the growth while the concentration of O exhibited a nonlinear power-law dependence with an order of about four. These observations imply that multiple oxygen-bearing species are involved in the incorporation process.

Additional supporting evidence for the model is found in the study by Tsai *et al.*<sup>28</sup> on the effect of oxygen on the PL spectrum of epitaxial  $\text{Al}_{0.06}\text{Ga}_{0.94}\text{As}$ . Tsai and co-workers showed that a PL band located at  $0.8 \text{ eV}$  was due to oxygen and proposed that an Al—O complex was responsible for the luminescence. No PL band near  $1.1 \text{ eV}$  was reported. The presence of an oxygen-related PL band at  $0.8 \text{ eV}$  in the low Al alloy composition samples of Tsai *et al.* is consistent with our observations that the  $0.8 \text{ eV}$  band observed in DEALO-based epitaxial GaAs is due to an oxygen-related defect in which oxygen interacts with more than one Al. The absence of the  $1.1 \text{ eV}$  PL band is also consistent with this model

because the probability of observing oxygen associated with only one Al is much lower in  $\text{Al}_{0.06}\text{Ga}_{0.94}\text{As}$  than in the lower Al concentration epitaxial systems considered by us. The results of Tsai *et al.* are supportive of the trends observed by us. Finally, we note that an oxygen-related defect model based on differences in the number of nearest-neighbor Al has also been suggested to explain the multiple DLTS peaks observed in DEALO-based epitaxial GaAs:O.<sup>23</sup>

A second possible explanation of our results is to assign the two near-IR PL bands to two different charge states of a single structural oxygen-related defect. A charge state model is suggested by the presence of multiple charge states for the electrically active oxygen-related defect in bulk GaAs. In a charge state model, the variations in the intensities of the two near-IR PL bands with oxygen concentration are attributed to a change in the relative population of the two charge states.

At this point, we have no direct knowledge of the nature of absolute charge states for the oxygen-related defect. It is useful, however, to discuss our results in the context of the charge state model described for bulk GaAs:O.<sup>17,29</sup> The electrically active (off-center substitutional) oxygen-related defect in bulk GaAs is known to be a negative- $U$  center with three charge states (zero, one, and two electron states) and two levels in the band gap (1/0 at  $E_c - 0.14$  eV and 2/1 at  $E_c - 0.58$  eV).<sup>17</sup> Since DLTS studies have indicated that the oxygen-related states in DEALO-based epitaxial GaAs:O are in the upper half of the band gap,<sup>22</sup> the near-IR emission observed must be, under the assumption of the bulk charge state model,<sup>17,29</sup> from the defect charge state levels 1/0 and 2/1 to the valence band. In order to explain our PL results, therefore, it is necessary that the zero and one electron states be present simultaneously in DEALO-based epitaxial GaAs:O. In the context of the bulk GaAs:O charge state model, photopumping the material with above gap light converts the zero and one electron states into the one and two electron states, respectively, and the two near-IR PL bands are due to radiative recombination to the valence band to restore the original charge states: 2/1 ( $\sim 0.8$  eV) and 1/0 ( $\sim 1.1$  eV). This model is plausible, but has the difficulty that the one electron state, known to be metastable in bulk GaAs:O, plays a prominent role in the luminescence. A similar alternative explanation of our PL results would be a three charge state model in which positive- $U$  ordering is present.

## V. SUMMARY

In summary, we have demonstrated the presence of two oxygen-related PL bands, at  $\sim 0.8$  and  $\sim 1.1$  eV, in epitaxial GaAs:O containing oxygen concentrations ranging from about  $10^{16}$  to  $10^{20}$   $\text{cm}^{-3}$  prepared in a MOVPE process using DEALO as the oxygen source. We have shown that an increase in either the oxygen concentration or the temperature leads to a quenching of the higher energy band relative to the lower energy band. We have proposed two possible explanations of the luminescence. The first model attributes the two PL bands to two chemically different oxygen-related defects. In this model, considering the strength of the Al—O bond, the 1.1 eV PL band is proposed to be due to substitutional oxygen associated with three Ga and one Al atoms and the 0.8 eV PL band to substitutional oxygen associated with two

Ga and two Al atoms. In a second model, the two PL bands are assigned to two different charge states of a single oxygen-related defect. In this model, changes in the relative intensities of the two PL bands with increasing oxygen concentration are attributed to changes in the concentrations of the two charge states. Future work will attempt to clarify our understanding and will focus on absorption, excitation, electrical, and high-pressure measurements designed to better elucidate the structural nature of the oxygen-related defects, the locations of the oxygen-related states in the band gap, the assignments of the near-IR photoluminescence bands, and the effect of these states on the electrical and optical properties of epitaxial GaAs.

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