

**Photoemission study of GaAs(100) grown at low temperature**

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GaAs(100) layers grown by low-temperature (LT) molecular beam epitaxy were studied by means of valence-band and core-level photoelectron spectroscopy. Small differences were found between valence-band spectra from the LT layers and corresponding layers grown at high temperature. In the Ga 3*d* spectra a new component was found in the LT-GaAs. The relative intensity of this component was found to be practically constant with varying probing depth. It is proposed that this component represents sites coordinated to the five-atom As clusters formed at As<sub>Ga</sub> antisites. This interpretation implies a higher density of antisite defects in the near-surface region than typically found in the bulk.

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**I. INTRODUCTION**

GaAs layers grown by low-temperature molecular beam epitaxy (LT-MBE), at substrate temperatures below 300 °C, exhibit properties which deviate strongly from those of layers obtained under “normal” conditions (i.e., at around 600 °C). Earlier reports include observations of extremely high electrical resistivity,<sup>1</sup> very short carrier lifetimes,<sup>2</sup> and even increased resistance against oxidation.<sup>3</sup> All these features are believed to be caused by point defects, which are generated during the LT growth at densities exceeding by orders of magnitude those prevailing at thermal equilibrium. This has led to a number of theoretical<sup>4,5</sup> as well as experimental<sup>6,7</sup> studies aiming at identification of the various possible point defects, and mechanisms of their incorporation in the lattice.<sup>8</sup> It is established that As<sub>Ga</sub> antisites are the predominant defects in LT-GaAs grown under As-rich conditions.<sup>9–11</sup>

While the properties of LT-GaAs are interesting enough to motivate extensive studies, there is an additional aspect that has boosted the interest in LT-MBE materials recently, namely, the possibility to include high concentrations of magnetic impurities in the GaAs lattice, and the discovery of carrier induced ferromagnetism in these systems.<sup>12,13</sup> This has opened prospects of combining magnetism with the well established GaAs-based technology. In this context the accommodation of As<sub>Ga</sub> antisites is extremely important, as these defects act as donors. At high concentrations these antisites are suspected to counteract the ferromagnetic ordering in two ways. First of all, their donor character directly compensates the Mn induced holes, which are believed to be responsible for the ferromagnetic coupling of the Mn acceptor ions.<sup>14–16</sup> Secondly, it has been found that the location of the antisites relative to the Mn ions can be decisive for the magnetic ordering.<sup>16</sup>

In the present study we have investigated the electronic structure of LT-MBE GaAs by means of valence-band and core-level photoelectron spectroscopy. Although this method is not useful (in its present state) for identification of defects in semiconductors, we find that the spectra from LT-MBE GaAs deviate in several respects from those obtained from GaAs grown at high temperature. Some of the features are interpreted as a direct effect of point defects. Thus, given the identity of these defects, their density can be estimated from the photoelectron spectra.

**II. EXPERIMENT**

The experiments were performed at the toroidal grating monochromator beamline (BL 41) at the MAX I storage ring of the Swedish National Synchrotron Radiation Center MAX-lab. At this beamline an MBE growth system is directly connected to the photoelectron spectrometer, allowing sample transfer under ultrahigh vacuum conditions. The MBE system is equipped with five Knudsen-type effusion cells and an As<sub>2</sub> valved cracker source. The growth is monitored by means of reflection high-energy electron diffraction (RHEED) at 10 keV, and whenever possible, growth rates are calibrated by means of RHEED oscillations. Substrate temperatures down to 400 °C were measured by an infrared pyrometer. Lower temperatures were estimated using a W-Rh thermocouple in contact with the backside of the sample holder and via the power supplied to the sample heater, both calibrated versus the pyrometer at temperatures above 400 °C. Also, the As condensation temperature (160 °C) was used as a reference. The uncertainty in the temperature determinations in the LT region is estimated to be about ±15 °C.

The samples were approximately 10×10 mm<sup>2</sup> pieces of epi-ready *n*-type GaAs(100) wafers, which were In glued on

transferable Mo holders. The LT-GaAs growth was preceded by deposition of a few hundred Å GaAs at 590 °C. The  $\text{As}_2/\text{Ga}$  flux ratio was maintained at values around 10, and the growth rates were typically 0.2 ML/s, calibrated by RHEED oscillations. During the LT growth very small attenuation of the oscillation amplitude was observed—typically a few hundred oscillations could be recorded.<sup>17</sup>

In order to avoid unintentional excessive As adsorption after growth, and to achieve similar As concentrations on surfaces prepared at different temperatures, all samples were kept for 20–30 min in the MBE chamber at a temperature of around 200 °C. In the transfer system the vacuum was in the low  $10^{-9}$  torr range, and in the electron spectrometer in the low  $10^{-10}$  range.

Immediately after transfer the surfaces were checked with low energy electron diffraction (LEED), and were rotated to keep the [110] azimuth in the plane of light incidence in the following photoemission measurements. The photoemission spectra were excited with mainly *p*-polarized light incident at 45° relative to the surface normal, and the electron energy distribution curves were obtained with a hemispherical angle-resolving electron analyzer, movable around a vertical and a horizontal axis. The angular and energy resolutions were  $\sim 2^\circ$  and 0.15–0.25 eV, respectively. Only normal emission spectra will be discussed in this paper. After the experiments the lattice constant along the growth direction in the fully strained overlayers was determined by symmetric 004 Bragg diffraction observed using a high-resolution x-ray diffractometer (Philips).

### III. RESULTS AND DISCUSSION

The atomic structure of GaAs(100) surfaces has been a subject of extensive studies in the past, and the sequence from the Ga rich  $c(8\times 2)$  to the ordered As-adlayer terminated  $c(4\times 4)$  phases, occurring with increasing surface As content, is well established.<sup>18</sup> At surface temperatures below 300 °C a  $(1\times 1)$  structure is found in electron diffraction, presumably due to adsorption of excess As in a disordered layer.<sup>19</sup> In the present studies the  $(1\times 1)$  surface geometry was seen in RHEED on samples grown below 220 °C. Various observations indicate that also in this case the  $(1\times 1)$  diffraction pattern is a result of excess-As induced surface disorder rather than a sign of a true ordered  $(1\times 1)$  surface geometry. In photoemission we find similar surface-state structures for the  $c(4\times 4)$  and  $(1\times 1)$  surfaces. In RHEED as well as LEED we see that the transition between the  $c(4\times 4)$  and  $(1\times 1)$  patterns occurs via gradual weakening and broadening of the fractional order spots, and an accompanying increase in the background intensity. This background remains relatively high after complete disappearance of the  $c(4\times 4)$  reconstruction pattern, even though the integer order spots are well defined. As mentioned above, all samples were kept after growth at a temperature of approximately 200 °C in order to acquire similar surface concentrations of post-growth adsorbed As. Immediately after this annealing the  $c(4\times 4)$  pattern could be seen only on samples prepared above 300 °C. Thus, for the sample grown at 250 °C discussed below, the surface reconstruction was removed by the

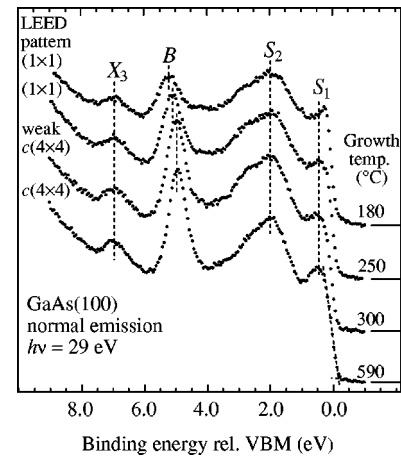


FIG. 1. Valence-band photoemission spectra from GaAs(100) grown at different substrate temperatures.

annealing. We ascribe this change to out-diffusion of As, which is incorporated in the crystal during the LT-MBE process. Such out-diffusion was recently proposed to explain changes in magnetic properties of GaMnAs subject to annealing<sup>20</sup> in the temperature range 220–260 °C. As will be shown below, the additional content of As on surfaces prepared at low temperatures is in the submonolayer range. This is sufficient to reduce the long-range order enough to remove the reconstruction pattern in electron diffraction.

Valence-band photoelectron spectra from LT-GaAs are generally quite similar to those obtained from layers grown at high temperature. Figure 1 shows normal emission spectra excited with 29-eV photons, from samples prepared at four different substrate temperatures. The spectra have been aligned at the valence band maxima (VBM), and we see that this alignment results in coincidence of the  $X_3$  density-of-states (DOS) peak around 7 eV. The general similarity is evident, and we note in particular the two structures  $S_1$  and  $S_2$ , which reflect emission from two surface states characteristic of the  $c(4\times 4)$  reconstruction.<sup>21</sup> The fact that these structures are present in all spectra indicates that the underlying surfaces are basically the same. This supports the interpretation of the  $(1\times 1)$  diffraction pattern as a result of randomly distributed As atoms (or dimers) in excess of those defining the  $c(4\times 4)$  geometry. The apparent shift of the  $S_1$  surface state peak is ascribed to modifications of the surface dimers, as discussed in the section on core level spectra (see below). Before focusing on the differences between the spectra, we should stress that the photon energy as well as the photoelectron emission angles were carefully checked to ensure that the data represent excitations in the same phase-space region. The most obvious difference is the varying intensity of peak *B*, which reflects a direct interband excitation from the  $p_z$  band. For samples grown below 300 °C the interband excitation becomes clearly reduced (the intensity ratio between the 590 and 180 °C spectra is around 2.8), and the width of this peak is slightly increased (FWHM=0.65 and 0.81 eV for the 590 and 180 °C spectra, respectively). At the same time we find that the  $X_3$  DOS peak is also reduced, in fact its intensity is proportional to that of peak *B* within

the accuracy of our experiment. Attenuation of direct interband-transition peaks is generally observed in angle-resolved photoelectron spectra as a result of surface disorder, in which case the photoelectron beams are dispersed over a wide range of emission angles. Provided the spectra do not probe directly the critical points in the Brillouin zone, however, the intensity of the DOS peaks is then relatively enhanced for the same reason.<sup>22</sup> The reduced intensity of interband excitation features observed here (direct as well as indirect ones), must therefore have another explanation. We can also rule out the influence of the surface reconstruction in this context, as a large reduction is seen between the 250 and 180 °C samples, both exhibiting (1×1) diffraction patterns. Somewhat similar intensity variations can be seen between spectra from GaAs layers grown by MBE with As<sub>4</sub> and As<sub>2</sub> sources.<sup>23</sup> Although in this case the authors emphasized the preparation dependence of surface-state structures, one can see that also the intensities of the bulk-state features depend on the preparation method. In the case of LT-MBE growth it is known that very high densities of As antisite defects (in the range of 10<sup>20</sup>/cm<sup>3</sup>) are introduced in the GaAs layers.<sup>9–11</sup> It is possible that similar defects are more common in the layers prepared with As<sub>4</sub> than with As<sub>2</sub>. Another case that should be mentioned in this context is the almost total attenuation of bulk-state emission from the corresponding band in InAs, after adsorption of small amounts (0.05 monolayer) Cs.<sup>24</sup> The modification in the last case consists in supply of electrons from the Cs adatoms. In the case of LT-GaAs the As antisite defects (up to ~10<sup>20</sup>/cm<sup>3</sup>) act as donors, so there is a supply of free electrons in this case as well. We propose speculatively that these modifications cause a reduction of the amplitude of *p<sub>z</sub>*-derived band states in the near-surface region. A definite explanation obviously requires further detailed investigations.

A second difference also concerns peak *B*, namely, its energy position. We see that for the samples grown at low temperatures this peak shifts somewhat towards higher binding energy. A very similar observation in Ga<sub>1-x</sub>Mn<sub>x</sub>As has recently been reported by Okabayashi *et al.*, who, however, ascribe the shift to the presence of Mn in the LT-GaAs host matrix.<sup>25</sup> In view of the present results that interpretation appears questionable. It is well known that the lattice constant of LT-GaAs is increased relative to that of HT-GaAs due to the high density of As<sub>Ga</sub> antisites.<sup>9–11</sup> Assuming that the slight lattice expansion has negligible influence on the band structure, the photoelectron spectra may still be modified via shifts of the final free-electron-like states. Qualitatively it is easy to see that a reduced size of the Brillouin zone (corresponding to an increased lattice constant) should shift the final-state bands towards lower energy. In a photoemission experiment at fixed photon energy, this implies that the initial state is also shifted in the same manner. Under these assumptions, and using a local dispersion of around -8 eV/Å<sup>-1</sup> (according to photoemission experiments),<sup>19</sup> and a lattice expansion  $\Delta a_z/a_0 = 7.5 \times 10^{-4}$  (determined for our 180 °C sample), we find that the energy shift due to lattice expansion alone should be around 10 meV in this case. As this is more than an order of magnitude smaller than the observed shift, we must conclude that the experimentally

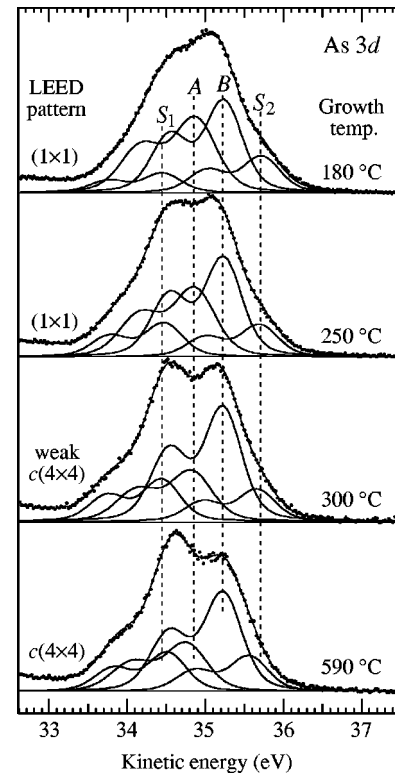


FIG. 2. As 3*d* core-level photoemission spectra, excited with 81 eV photon energy, from GaAs(100) grown at different substrate temperatures.

observed shifts are due to another effect. A possible explanation is a changed inner potential, which is an effective parameter that could well be sensitive to the modified dielectric properties of LT-GaAs.

Core-level spectroscopy has been applied very successfully in the past for characterization of semiconductor surfaces. In particular the As 3*d* spectrum from GaAs(100) surfaces has been subject to systematic investigations aiming at identifying the surface components and relating them to the nature of the different surface reconstructions.<sup>21,26,27</sup> Focusing on the most As-rich surfaces, which are of interest in the present study, the *c*(4×4) surface was found to occur for a range of surface As contents, but its detailed geometry and even its composition is not as well established as that of the less As-rich *c*(2×8) surface.<sup>28</sup>

The As 3*d* core-level spectra obtained from the four samples discussed above are displayed in Fig. 2. The energy scale in this figure refers to that of the 590 °C spectrum. The other spectra have been shifted (to compensate for slightly different surface Fermi-level pinnings). It is clear that the spectrum of the *c*(4×4) surface is rather complex, and although there is fair agreement in literature on its general interpretation, differences are found in the detailed analysis. To obtain consistent fits of different spectra from this surface, using a least-square fitting procedure, we found that four components must be considered, as in Ref. 27. Due to the extensive overlap between these components, it is clear that the fitting is nevertheless not unique—slight changes in, for example, broadening parameters may influence strongly

TABLE I. Fitting parameters and peak separations for the core-level spectra in Figs. 2–4. HT refers to the sample grown at 590 °C and LT to the 180, 230, and 250 °C samples (the 300 °C-sample displays intermediate component separations). All energies are given in eV. The peak separations for different As 3*d* fits vary within ~20 meV.

	As 3 <i>d</i>		Ga 3 <i>d</i>	
	HT	LT	HT	LT
Spin-orbit splitting	0.69		0.44	
Branching ratio	1.69		1.62	
Lorentzian width	0.170		0.155	
Gaussian width	0.42 <sup>a</sup>	0.46 <sup>a</sup>	0.42	0.46
$\Delta E(B-S_1)$	0.77	0.77		
$\Delta E(B-S_2)$	-0.33	-0.47		
$\Delta E(B-A)$	0.48	0.36		
$\Delta E(B-B')$				0.41

<sup>a</sup>All except component A, in which case the Gaussian width was 0.53 eV.

the relative intensities as well as the precise peak positions. Since the Ga 3*d* spectrum is considerably less complex (see below), the degree of arbitrariness could be reduced by imposing a fixed energy separation of 21.85 eV between the As and Ga 3*d*<sub>5/2</sub> bulk components.<sup>29</sup> As another aid in the fitting process we generated difference spectra (not shown) to determine the nature of the changes between the different spectra. The energy location of the component reflecting the excess surface atoms could be determined from differences between the 180 and 590 °C spectra. The fitting parameters and the relative energy positions of the different components are listed in Table I. Referring to previous studies, the components are ascribed to As atoms in the dimers defining the *c*(4×4) surface unit cell (*S*<sub>1</sub>), As atoms in the last full As layer of the substrate (*S*<sub>2</sub>), atoms in the bulk (*B*), and atoms (or molecules) in the adsorbed amorphous overlayer (*A*). This assignment justifies the somewhat larger broadening applied in the fitting of the last component (see Table I). The energies of the *S*<sub>1</sub> and *A* doublets are in fairly good agreement with those found in Ref. 26, but the position of *S*<sub>2</sub> is about 0.16 eV higher in the present fit. On this detail our fit is in good agreement with Ref. 23.

With lowering growth temperatures we see that the overall spectral shape is gradually changed. The sequence actually resembles that found in Ref. 26, in which case the spectra were recorded after sequential annealings. However, in the present case the spectral changes are not due to loss of surface As—on the contrary, we find that the relative As intensity is increasing somewhat with decreasing growth temperature. The fits show that the changes occurring for the samples grown below 300 °C can be mainly ascribed to increasing amplitude of component A. The accompanying reduction of the *S*<sub>1</sub> surface component could then be explained as an effect of gradual incorporation of the *c*(4×4) As dimers into the disordered As overlayer, in the sense that the top-layer As atoms get adsorbed As molecules as nearest neighbors. The combined relative intensity of *S*<sub>1</sub> and *A* is about 10% larger for the 180 and 250 °C samples. As already

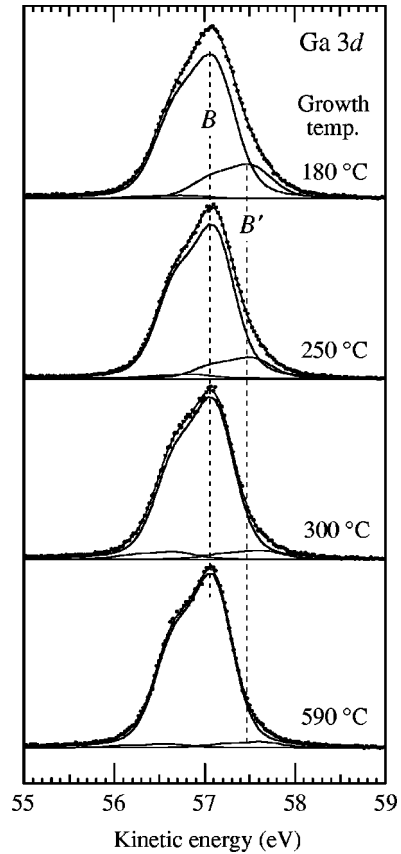


FIG. 3. Ga 3*d* core-level photoemission spectra, excited with 81 eV photon energy, from GaAs(100) grown at different substrate temperatures.

mentioned, we ascribe this increased surface content of As to out diffusion of atoms incorporated during the LT growth. The fact that the *S*<sub>2</sub> component, representing As atoms in the last full As layer of the substrate, remains relatively unaffected indicates that only small amounts of As are involved in the removal of the *c*(4×4) surface reconstruction. It is also noted that the relative positions of the four components are somewhat different in the spectra from the LT samples. The shifts associated with component A are interpreted as an indication that this component represents a set of peaks of similar nature, not individually resolved in the present analysis but taken into account by the use of a larger Gaussian width for A. We conclude that apart from a small increase in the surface As content, the spectra from LT-GaAs do not differ in any qualitative way from those of surfaces grown at high temperature. Considering the complexity of the As 3*d* spectra, we are not able to extract any further details that may be hidden in the mixture of the four components. In particular, any search for a component that might arise due to population of As<sub>Ga</sub> antisites in the bulk at a concentration level of 1% is of course futile. However, as will be shown next, indirect evidence of such antisite defects are found in the Ga 3*d* spectrum.

In Fig. 3 we show a set of Ga 3*d* spectra, recorded parallel with the As spectra discussed above. Since previous studies of GaAs have been mostly concerned with the As-rich surfaces, the Ga spectra have attracted significantly less

attention. Starting with the spectrum from the high-temperature GaAs we see that it is significantly “cleaner” than the corresponding As spectrum. This is not so surprising, as the surface is fully As covered, and thus practically all Ga atoms are fourfold coordinated to As. Using the same Lorentzian broadening as adopted in previous studies,<sup>27</sup> we have to include three components for the best fit, but by a slightly increased broadening the two additional components can be eliminated entirely. The bulk component is thus completely dominating, and there is no indication of any significant structure that could be ascribed to Ga in surface sites as inferred from ion scattering experiments.<sup>28</sup> The spectrum from the 300 °C sample is very similar to the high temperature spectrum. Turning to samples grown at still lower temperatures, a gradual development of a tail towards high kinetic energy is easily seen. The least-squares fitting analysis reveals that the LT-GaAs spectra are fitted with two components. The appearance of a new component in the LT-GaAs spectra is not expected, since the surface As content is not reduced to change the coordination of the Ga atoms in the outermost surface layers. The intensity of the new component is quite high—in the spectra from the 250 °C sample it is around 12%, and for the 180 °C sample it is around 18% of the total Ga intensity. Suspecting that the new component is not surface related (as a consequence of the high As content at the surface), we must consider the possibility that it is bulk derived. As already pointed out, the LT-GaAs is quite rich in point defects, dominated by neutral As antisites. For growth temperatures around 200 °C the defect densities are very high, in the range of  $1 \times 10^{20}/\text{cm}^3$ , i.e., 0.5–1% of the As atoms in the bulk are in such defect sites. Considering each antisite as a center of an  $\text{As}_5$  cluster, it is clear that the 12 Ga atoms coordinated to this cluster can be expected to deviate from those in “normal” bulk sites. Thus, provided that the combined effect of modified charge transfer, Madelung potential, and final-state screening at these defect-coordinated sites is sufficiently large, it becomes likely that a new component in the Ga core-level spectrum should be observable, with a relative intensity in the range of 10% of the total intensity. Qualitatively it is reasonable to expect a shift towards larger kinetic energy because of the donor character of the antisite defects.

To test this hypothesis further and examine whether the additional structure is surface or bulk derived, we have exploited the tunability of probing depth with varying kinetic energy of the photoelectrons. The sample examined in this case was one grown at 230 °C substrate temperature, so the intensity of the additional component is expected to be similar to that in the 250 °C spectrum in Fig. 3. In Fig. 4 we show spectra obtained in normal emission, using three different photon energies. With 81-eV photons the kinetic energy of the photoelectrons is around 57 eV, which is in the range of the minimum mean free path in GaAs,<sup>30</sup> slightly less than 4.0 Å. With reducing kinetic energies the mean free path is increasing, and at 17 eV kinetic energy, obtained with 41-eV photons, the mean free path is around 6.2 Å. Since the growth was observed to proceed layer-by-layer (very nice RHEED oscillations were recorded during the whole growth process), the sample is known to consist of a regularly alter-

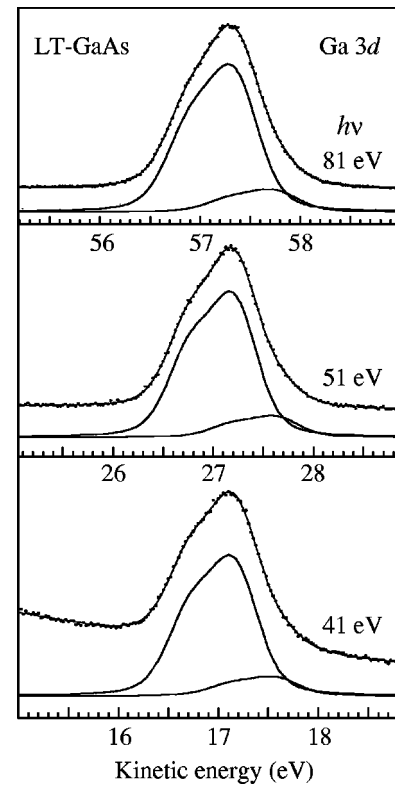


FIG. 4. Ga 3d core-level photoemission spectra from LT-GaAs (grown at 230 °C substrate temperature), excited with 41, 51, and 81 eV photon energies.

nating sequence of atomic Ga and As layers along the growth direction. Using the above mean free paths, it is then straightforward to calculate the fraction of the spectral intensity contributed by the first Ga layer. For the shortest mean free path we thus obtain a fraction of 51%, and for the longest mean free path this fraction is reduced to 36%. These numbers are to be compared with  $13 \pm 1\%$  observed in the present data for the relative intensity of the high-energy Ga component. The practically constant relative contribution of the additional component confirms its bulk nature.

In consequence of this reasoning we find that the appearance of the new Ga component in spectra from samples grown below 250 °C, and the increasing magnitude of this component with lowering growth temperature, clearly suggest that its origin is related to the As antisites. From the relative intensity of the new component we can make an independent estimation of the abundance of these defects. Assuming the perfect coordination conditions mentioned above, we find that the density of As antisites is around  $3.3 \times 10^{20}/\text{cm}^3$  and  $2.4 \times 10^{20}/\text{cm}^3$  for the 180 and 250 °C samples, respectively. These numbers are a factor of 2–3 higher than those derived from studies of thick LT-GaAs layers.<sup>9</sup> A possible reason for this deviation is that the density of defects is higher in the first few atomic layers, since the surface is very As rich (though the possibility that the new component originates from only the first atomic layer is excluded). To our knowledge the layer thickness dependence has not been studied systematically, but it is worth noting that scanning tunneling microscopy studies of 20 nm thick

layers<sup>11</sup> clearly reveal higher densities of antisite defects than reported for the thick layers.<sup>9</sup> To the extent that this enhanced concentration of defects is surface related it is of course not significant for the magnetic properties in the bulk of GaMnAs (grown at similar LT conditions).

#### IV. SUMMARY

By comparative studies of photoemission spectra from GaAs(100) layers grown at high and low substrate temperatures we have found that the bulk energy bands are somewhat modified in the near-surface region in LT-GaAs. The As 3*d* core-level spectra are also modified, but the spectral deconvolutions indicate that essentially the same components are present in LT- and HT-GaAs, and that the modifications are less dramatic than suggested by the overall appearance of the spectra. On the other hand, in the Ga 3*d* spectra a new component is clearly appearing for LT-GaAs. By examining the probing-depth dependence of this component, we find

that it is bulk derived. It is proposed that the new component in the Ga 3*d* spectrum represents atoms in second-nearest-neighbor sites relative to the As antisite point defects, which are known to occur at very high densities in LT-GaAs. The clearly higher concentrations of antisite defects deduced from the photoemission data than typically observed by bulk probes such as x-ray diffraction could be due to a higher density of defects in the near-surface region.

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