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Adsorption of potassium and oxygen on GaAs(110): Charge transfer and enhanced oxidation

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The behavior of charge transfer and oxidation of GaAs(110) substrates, covered with potassium and/or oxygen, and after thermal treatment, is investigated by Auger and photoelectron spectroscopy, work-function measurements, and low-energy electron diffraction. The oxidation of GaAs(110) is efficiently promoted by the presence of K adatoms. The experimental results indicate that both the charge transfer from the K atoms to the substrate and the formation of several potassium oxides may be the crucial factors for the enhancement of the substrate oxidation.

I. INTRODUCTION

The interaction of alkali-metal atoms with GaAs surfaces has been studied in the past mainly with the aim of elucidating the mechanisms involved in the negative electron affinity (NEA) activation of GaAs substrates.[1–8] This was a crucial prerequisite to produce efficient photocathodes, image intensifiers, infrared-sensitive detectors, and night-vision devices.[9–11] On the other hand, the application of GaAs in present integrated-circuit technology is limited because of the lack of suitable oxidation procedures. Furthermore, the oxidation reaction of GaAs occurs at a low rate. There have been some attempts in the past years to find a catalyst able to speed the reaction[12–14] as well as reports of enhanced oxidation by illumination[15–17] or electron bombardment.[18,19]

Following early work of Goldstein[2] and Spicer *et al.*,[12] who found Cs-enhanced oxidation of the GaAs substrate while looking for an explanation of the NEA process, it has been observed that Al (Ref. 13) and Cf (Ref. 14) deposited at 300 K onto the surface enhance the oxygen adsorption kinetics by several orders of magnitude. For Cr the effect occurs only above a critical coverage (2 Å) and has been related to reactive interdiffusion of Cr at the interface.[14]

Recently we investigated the interaction between alkali metals and semiconductor surfaces from the point of view of model systems where novel ideas about charge transfer, band bending, or Schottky-barrier formation can be tested[20–22] as well as trying to understand and improve their relevant technological applications. Thus, we have shown that thick alkali-metal films can efficiently promote the massive oxidation of Si single crystals.[23–25] The identification of the mechanism responsible for this effect[26] allowed us to grow at reduced temperatures silicon dioxide films of thicknesses much larger than before.[27]

In this work we report on a systematic study of the adsorption of K adatoms on GaAs(110) surfaces prepared by cleavage or by ion bombardment and annealing as well as on the adsorption of oxygen on these K-covered substrates. Using Auger-electron spectroscopy (AES), low-energy electron diffraction (LEED), work-function measurements (WFM), and photoelectron spectroscopy (PES), the oxidation reaction as a function of the different experimental parameters (K coverage, oxygen exposure, and temperature) was investigated, which leads to a consistent picture that can also rationalize previous experimental findings. On the other hand, intriguing differences have been found between Si and GaAs.

II. EXPERIMENT

The AES, LEED, and WFM were performed in an UHV chamber equipped with a single-pass CMA, a four-grid LEED optics, and a Kelvin probe.[28] The AES data were taken by exciting Auger electrons with a 1.8-keV glancing-incidence electron gun and modulating the current collected in the CMA with a 2-V peak-to-peak signal. The PES experiments were performed in a different UHV chamber, using synchrotron radiation from the SX-700 monochromator at the "Berliner Speicherring für Synchrotronstrahlung" (BESSY), and a hemispherical electron-energy analyzer, with a total-system resolution of 0.15 eV.

The GaAs(110) surfaces were prepared *in situ* (1) by cleavage of single-crystal GaAs bars with the double-wedge, double-notch technique (PES), or (2) by subsequent ion bombardment and annealing (IBA) of a formerly cleaved surface, using Ar^+ ions with a kinetic energy of 1.5 keV and a current density of 3 $\mu A/cm^2$, and annealing temperatures of 800 K (AES, LEED, and WFM). The samples were heated by thermal radiation, controlling the substrate temperature with a thermocouple attached to the backside of the sample (accuracy ~ 50 K). Only mirrorlike cleavage surfaces were taken for the measurements. The substrates prepared by the IBA procedure showed no sign of surface contamination in the

AES data, and a low-background 1×1 pattern was detected by LEED. It should be noted here that the IBA surfaces show a slightly different stoichiometry relative to that of cleaved GaAs(110), as detected by the relative AES intensities of As and Ga (see below).

K was deposited on the surfaces from resistively heated, commercial K_2CrO_4 dispensers (SAES getters). During evaporation, the pressure rose from 1×10^{-10} to 5×10^{-10} mbar. The K coverage was determined by an AES-intensity calibration (see below). Contamination of the K-covered surfaces was carefully controlled by AES or PES, resulting in less than 1% of oxygen even for thick K coverages.

Oxygen exposures are given in langmuirs ($1\ \mathrm{L}=1\times10^{-6}$ Torr sec). Oxygen was admitted into the chamber with the hot ion-gauge filament on; thus it has to be considered that the molecular oxygen may be partially "excited" during exposure.[29] Coverages of both alkali adatoms and oxygen are given in monolayers (ML). One monolayer ($\Theta=1$) is defined as the total atomic density in the (110) plane of GaAs, i.e., $n_s=8.8\times10^{14}\ \mathrm{cm}^{-2}$.

III. RESULTS

A. Calibration of the alkali-metal coverage

K was evaporated onto the sample and the peak-to-peak AES intensities of Ga (at 48 and 52 eV), As (at 30 eV), and K (at 252 eV) were acquired simultaneously. They are shown in Fig. 1 as a function of the evaporation time for an *n*-type sample prepared by IBA. The Ga and As signals decrease as the surface is covered by K adatoms.

The linear behavior of all AES intensities with breaks taking place simultaneously for Ga, As, and K suggests a layer-by-layer growth similar to that found for K on Si(110) and Si(111),[20] Cs on GaAs(110),[4] and Cs on Si(100).[23] Actually, the solid lines are a fit to the data using a layer-growth model.[20] The fit gives values for the electron mean free path of 1.83 ML (Ga_{52}), 1.78 ML (As_{30}), and 3.5 ML (K_{252}), respectively, in excellent agreement with the literature,[30] the latter value being identical to the measured value for K/Si(100).[20] At the first break, indicated by an arrow, the K density is taken to be $4.43\times10^{14}\ \mathrm{cm}^{-2}$, i.e., $\Theta=0.5$ ML. The LEED pattern was 1×1 during adsorption, while the intensity distribution was different from that of the clean (110) surface.

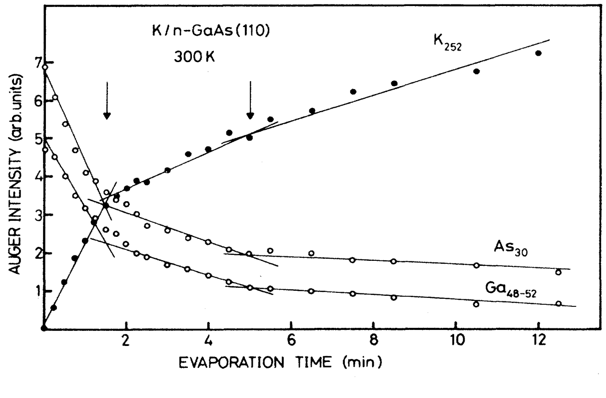

FIG. 1. Intensities of the Auger transitions of Ga, As, and K as a function of the K evaporation time on GaAs(110) and 300 K. The intensities of the Auger transition are taken to be the peak-to-peak height of the line in the $\Delta N(E)/N(E)$ spectrum. A glancing-incidence electron gun was used to maximize surface sensitivity. The arrows indicate K coverages of $\Theta=0.5$ and 1 ML, respectively.

Previous work has shown[22] that, at 300 K, Cs atoms do not react with the GaAs(110) surface while Na forms a reactive interface with GaAs(110). No intermixing or creation of interfacial defect states is detected for Na, K, Rb, and Cs at 85 K.[22] We assume that the K/GaAs(110) interface grown at 300 K is nonreactive and abrupt. There are no experimental data available concerning the atomic adsorption site for alkali metals on GaAs(110). Nevertheless, it is known that the empty electronic states of clean GaAs(110) are located on the Ga atoms,[31] and the total energy of the system should be lowered if K adsorption takes place in the vicinity of the Ga atoms,[32] possibly forming one-dimensional chains along the [110] direction. These will be the preferred adsorption sites up to $\Theta=0.5$ ML.

After completion of these sites, further K adsorption takes place until full monolayer coverage is reached, as indicated by the second arrow in Fig. 1. However, the sticking coefficient of K on the GaAs(110) surface decreases to 40% of its initial value, as suggested by the longer time needed to reach the second break in the intensity-versus-time curve. K coverages larger than 1 ML can be maintained on GaAs(110) at room temperature. However, a precise temperature control is crucial because it has been found that only 1 ML grows at 310 K, while at 290 K more than 1 ML can be stabilized on the surface. It should be noted here that the good agreement between fit and experiments in Fig. 1 is disturbed slightly at the coverage region near the first break at $\Theta=0.5$ ML. This may be interpreted in terms of a gradual decrease of the sticking coefficient near the saturation coverage of the first half-monolayer: the K atoms arriving at the K-covered parts of the surface have a lower sticking coefficient than those arriving at the bare GaAs(110) surface.

B. Work-function changes and charge transfer upon alkali-metal deposition

The work function ϕ of a semiconductor can be written as

$$\phi_p = I - e_0|V_s^p| - W_p \quad (p\ \text{type})$$

and

$$\phi_n = I - E_{\text{gap}} + e_0|V_s^h| + W_n \quad (n\ \text{type}) .$$

The ionization energy, I, contains the contribution of the surface dipoles. The band bending $e_0|V_s|=|E_v^s-E_v^b|$ is given by the energy position of the surface states and the bulk doping level. Only these contributions are surface dependent. The distances $W_n=E_c^b-E_F$ or

$W_p = E_F - E_v^b$ and the gap energy, E_{gap}, are bulk properties. Accordingly, adsorption-induced work-function changes include two independent contributions: the variation of ionization energy due to surface dipoles and the change in the band bending. Thus,

$$\Delta\phi_p(p\text{-type}) = \Delta I + e_0\Delta|V_s^p| \quad \text{(downwards band bending)} ,$$

$$\Delta\phi_n(n\text{-type}) = \Delta I - e_0\Delta|V_s^n| \quad \text{(upwards band bending)}$$

and the difference in the adsorption-induced work-function changes in p-type- and n-type-doped samples at identical coverages is

$$\Delta\phi_p - \Delta\phi_n = e_0(\Delta|V_s^p| + \Delta|V_s^n|) .$$

The work-function changes upon K deposition at 300 K onto p-type- and n-type-doped samples prepared by the IBA procedure are shown in Fig. 2. The data points coincide for both p-type- and n-type-doped samples. It is also evident that the $\Delta\phi$-versus-Θ curve displays a clearly resolved minimum followed by a rise until, at $\Delta\phi = -2.3$ eV, the absolute work function of bulk K is reached. It should be noted that such a minimum has not been observed previously for Cs/GaAs(110) at 300 K,[5,6] since relevant Cs coverages cannot be reached at this temperature, while it has been observed at lower substrate temperatures (85 K) for Na, K, Rb, and Cs.[22] The minimum in ϕ occurs at a K coverage corresponding to the first break in the AES-intensity–versus–time curve of Fig. 1, i.e., $\Theta_K = 0.5$ ML.

For Cs adsorption onto cleaved GaAs(110) surfaces, $\Delta\phi_p - \Delta\phi_n = 1.2$ eV,[33] in nice agreement with the Cs-induced band bending,[22] reflecting that in the well-cleaved sample, the bands are almost flat up to the surface, i.e., $e_0|V_s| = 0$. In samples prepared by the IBA procedure the adsorption-induced work-function changes are independent of the bulk doping, indicating that in this case the Fermi level is initially pinned at the surface (at 0.7 eV above E_v) by a high density of surface states in the gap. The same effect has also been observed for MBE-grown GaAs(100) surfaces and it has important implications for device stability.[34] It can be concluded that the data of Fig. 2 contain mainly contributions from the change in ionization energy (or electron affinity), produced by the transfer of charge and the induced dipoles between the alkali adlayer and the semiconductor surface. The data points as a function of coverage follow a Topping behavior,[35] suggesting that mutual depolarization is the major cause of the shape of the $\Delta\phi$-versus-Θ curve. The fit yields an initial dipole moment of $\mu_0 = 4.4$ D and a polarizability of $\alpha = 19.4$ Å^3, in excellent agreement with data for K/Si(100)2×1, K/Si(111)7×7,[20] Cs/Si(111)2×1,[36] and Cs/Si(100)2×1.[23] The amount of charge transferred from the alkali adlayer to the substrate can be estimated from the measured decrease in ionization energy as $\Delta Q(\Theta) = 0.063\Delta I(\Theta)/(L_{\text{eff}}\Theta)$,[23] where $\Delta Q(\Theta)$ represents the number of electrons transferred per adatom, $I(\Theta)$ is the corresponding change in ionization energy (in eV), Θ is the coverage in ML, and L_{eff} is the effective length of the dipole created at the surface in (Å). It turns out in our case that $Q(\Theta)$ decreases quickly with Θ, in such a way that at $\Theta_K = 0.5$ ML, only 0.15 electrons per K adatom are transferred to the GaAs substrate. For this estimation the effective dipole length has been taken as $L_{\text{eff}} = L_{\text{ad}}/\epsilon_{\text{ad}} + L_s/\epsilon_s$ with ϵ_s and ϵ_{ad} being, respectively, the dielectric constants of the substrate and the adsorbate complex, L_{ad} the covalent radius of K, and L_s the decay length of the metal-induced gap states (MIGS) in GaAs.[33] This type of simple approximation for the value of Q is in good agreement with elaborated theoretical calculations.[21,32]

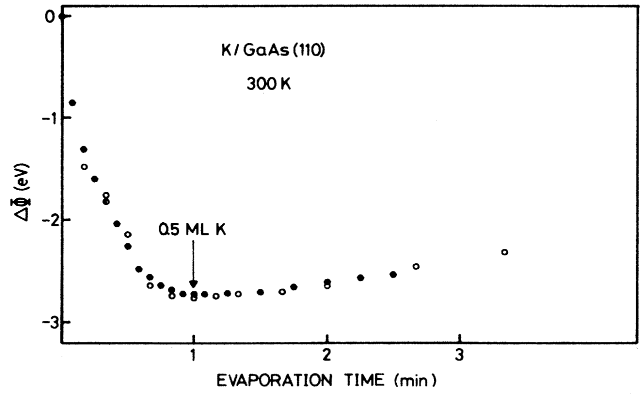

FIG. 2. Work-function changes measured with a Kelvin probe for K deposited on GaAs(110) at 300 K vs evaporation time. Solid and open circles represent data taken on n-type- and p-type-doped samples, respectively, prepared by the IBA method as described in the text.

C. Oxygen adsorption

In Fig. 3 the oxygen uptake at 300 K of a variety of GaAs(110) surfaces has been plotted as a function of the oxygen exposure. The uptake has been calibrated in monolayers following a procedure based on the ratio of the Auger peaks of O_{512} and Ga_{1070} and described in detail in Ref. 17. The solid line refers to a clean surface while the various symbols correspond to samples covered with different amounts of K. The dashed line represents the oxygen uptake under simultaneous illumination with a xenon-arc lamp (from Ref. 17). It is evident that the presence of even minute amounts of K strongly enhances the oxygen uptake kinetics. Note that the curve for $\Theta_K = 0.08$ ML essentially resembles the shape of the clean-surface curve shifted 7 orders of magnitude to lower exposures.

After 1 L exposure of O_2 at 300 K, the number of O_2 molecules that have hit the surface is 3.59×10^{14} molecules/cm^2. If during the exposure a constant sticking coefficient of 1 is assumed, the resulting coverage of oxygen would be 0.81 ML. This point is indicated in Fig. 3 and correlates nicely with the extrapolation of the data for a K coverage of 0.56 ML, suggesting that at this alkali coverage the initial sticking coefficient is probably 1.

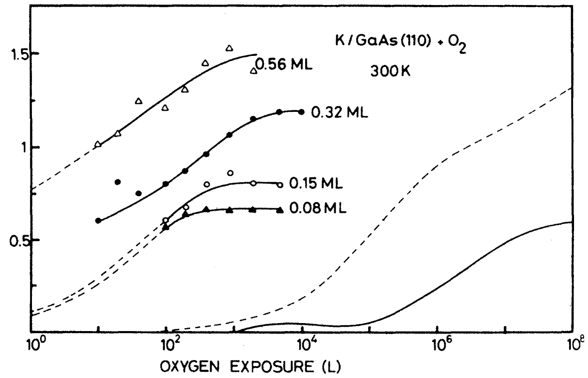

FIG. 3. Oxygen uptake in monolayers as calibrated in Ref. 17 by AES intensities as a function of the oxygen exposure at 300 K. Data points correspond to $\Theta_K = 0.08$ ML (solid triangles), 0.15 ML (open circles), 0.32 ML (solid circles), and 0.56 ML (open triangles). The solid line represents the oxygen uptake of a clean GaAs(110) surface, while the dashed line shows the uptake on a surface illuminated by a xenon-arc lamp (taken from Ref. 17).

Therefore, the alkali adlayer seems to increase substantially the initial sticking coefficient of molecular oxygen, which is known to be extremely small on a clean GaAs(110) surface.[37] It should be noted that the enhancement produced by laser irradiation is much weaker, being only a factor of 5–20 with respect to the nonilluminated sample.[16]

The conversion from chemisorbed oxygen into oxide of the substrate can be followed by changes in the line shape of the AES spectra. In clean GaAs surfaces the $M_2M_4M_4$ and $M_3M_4M_4$ Auger transitions of Ga yield two peaks at 48 and 52 eV, respectively.[38] When oxidation of the substrate sets in, this doublet is gradually replaced by a chemically shifted Ga doublet with peaks at 44 and 48 eV.[38] Thus, it is customary to control the oxidation of the sample by plotting the intensity ratio of the Ga_{52} and Ga_{48} AES peaks.[18] This ratio stays constant with increasing oxygen uptake until at oxygen coverages above 0.5 ML it begins to decrease, signaling the GaAs oxidation. The AES data are plotted in this way as a function of the oxygen exposure in Fig. 4. It can be seen in the exposure range shown that the clean sample gives a constant (no oxide formation) ratio, while samples with increasing amounts of K display a strongly enhanced oxidation kinetics.

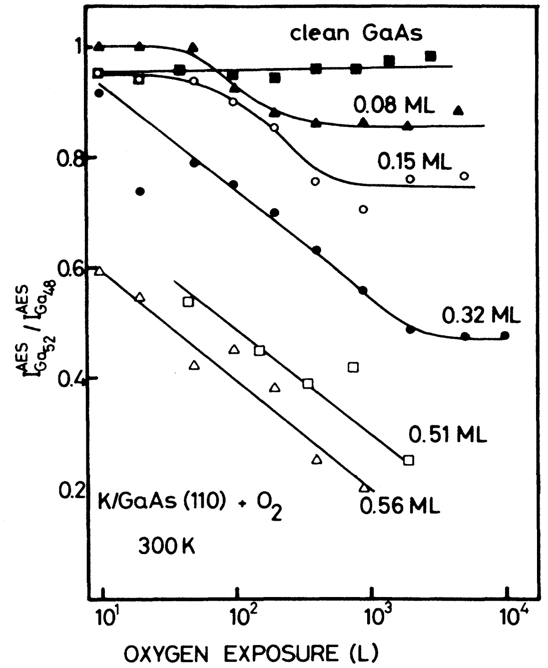

FIG. 4. Ratio of the Ga AES signals at 52 and 48 eV vs oxygen exposure at 300 K. Symbols represent the following: clean GaAs(110) surface (solid squares), $\Theta_K = 0.08$ ML (solid triangles), 0.15 ML (open dots), 0.32 ML (solid circles), 0.51 ML (open squares), and 0.56 ML (open triangles).

D. Work-function changes upon oxygen adsorption

Admission of oxygen on a K-covered GaAs(110) sample results in the work-function changes shown in Fig. 5. If oxygen is adsorbed at K coverages below the minimum in $\Delta\phi$, the average work function increases. Only if the adsorption takes place above $\Theta_K = 0.5$ ML is an additional minimum in the work function observed, which, however, only exceeds slightly the work-function minimum obtained with clean K on the GaAs(110) surface. Similar observations for K/Si(100) have been found to correlate with the formation of well-defined oxygen species on the surface as judged by low-temperature ultraviolet photoemission spectroscopy (UPS) studies:[36] The $\Delta\phi$ increase observed for $\Theta_K < 0.5$ ML reflects a preferential oxygen bonding to K which does not result in O^{2-} formation,[39,40] while the additional minimum produced during O_2 exposure at $\Theta_K > 0.5$ ML corresponds to formation of K suboxides containing O^{2-} species (i.e., K_2O).[10,36,40]

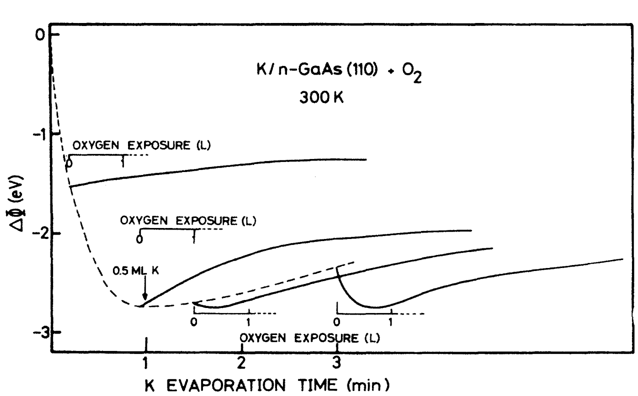

FIG. 5. Work-function changes for oxygen adsorption at 300 K on a K-covered GaAs(110) surface prepared by the IBA method. The dashed line represents the $\Delta\phi$ values for K deposition, while the solid lines represent the additional changes produced by O_2 exposure.

E. Photoemission results

In order to characterize the reaction products of the oxidation process and the mechanisms of the K-enhanced oxidation, photoemission experiments using synchrotron radiation have been carried out. Figure 6 presents photoemission spectra, taken at $h\nu=80$ eV, for cleaved GaAs(110) surfaces, covered with 1.2 ML of K and exposed to 100 L of O_2 at room temperature, and after subsequent annealing. The spectra of the clean surface show a strong emission from the As 3*d* and Ga 3*d* core-level electrons, and a weak valence-band signal. The deposition of several ML of K results in the appearance of K 3*s* and K 3*p* lines (spectrum not shown here, see Fig. 6 in Ref. 22). The band bending and changes in the electronic structure produced during deposition of K have been reported elsewhere.[22]

After an exposure of 100 L O_2 to the surface, intensive additional spectral features become visible for the As 3*d* and Ga 3*d* core levels at higher binding energies (BE's), which may be assigned to As and Ga atoms reacted with oxygen. The K 3*s* level is clearly visible, while the K 3*p* line overlaps strongly with the Ga 3*d* signal. The valence-band emission exhibits the O 2*p* multiplet structure of a nonstoichiometric K oxide, which partially overlaps with a strong O 2*p* emission with a mean BE of 5 eV, as has been observed during the oxidation of clean GaAs(110).[41] Three peaks appear at BE's of 7.3, 9.0, and 10.9 eV, corresponding to K oxides. However, a clear identification of the chemical composition of the K oxides is difficult due to the overlap with the strong peak at 5 eV. We should note that the observed BE's are very similar to those reported by Su *et al.*[39] for their so-called state 3 in the oxidation of bulk Cs.

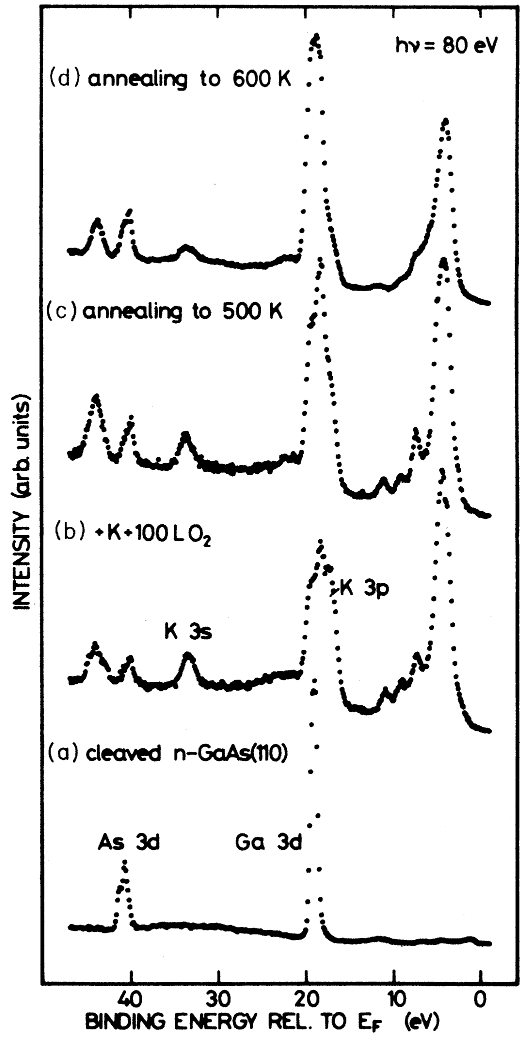

FIG. 6. Photoemission spectra taken with $h\nu=80$ eV for (a) clean *n*-type-GaAs(110) surfaces, (b) covered with 1.2 ML of K and following exposure to 100 L of O_2, (c) and (d) after subsequent annealing to 500 and 600 K, respectively.

The annealing of the interface to 500 K for 2 min leads to an increase of the relative intensities of the K 3*s* and K 3*p* signals, while the intensity of the As 3*d* and Ga 3*d* oxide components stays almost constant relative to the substrate signal. Furthermore, the valence-band signal decreases in intensity, but shows only slight structural changes. Thus it may be concluded that annealing to 500 K mostly decreases the K content in the interface. Further annealing to 600 K leads to a strong decrease in intensity of both the K core-level signals *and* the As 3*d* oxide component, and a slight increase of the relative intensity of the Ga 3*d* oxide component. Additionally, the valence-band signal reveals strong structural changes, which may be assigned to an intensity decrease of the K oxide and a growth of the signal associated to the substrate oxides. These observations indicate that K cannot be eliminated fully without changing the stoichiometry of the substrate oxides, in contrast to the case of Si.[26]

In order to illustrate the enhancement of the oxidation reaction in the presence of a K layer, and for a closer look to the As and Ga oxides produced thereby, Fig. 7

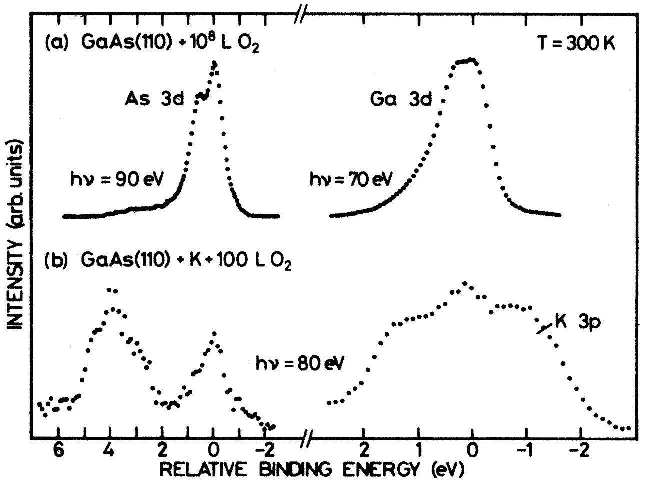

FIG. 7. As 3*d* and Ga 3*d* photoelectron spectra of GaAs(110) surfaces (a) after exposure to 10^8 L of unexcited oxygen (from Ref. 42) and (b) after deposition of 1.2 ML of K and subsequent exposure to 100 L of oxygen.

shows As 3*d* and Ga 3*d* spectra under different oxidation conditions. Figure 7(a) contains spectra from Landgren *et al.*[41] of clean GaAs(110) exposed to 10^8 L of unexcited O_2, while Fig. 7(b) shows spectra of the K-covered GaAs(110) surface after exposure to 100 L O_2. Because of similar kinetic energies of the photoelectrons, the surface sensitivities are comparable. It is clearly seen in Fig. 7 that the presence of K enhances the oxidation velocity of GaAs(110) by more than a factor of 10^6. Furthermore, similar BE shifts upon oxidation suggest that almost the same As and Ga oxides are formed. It has been shown that As atoms bonded to one, two, three, or four oxygen atoms, respectively, result in As 3*d* core-level shifts of 0.8, 2.3, 3.2, and 4.2 eV, respectively, relative to the unreacted GaAs(110) substrate lines.[41] Similarly, two oxidation states with BE shifts of 0.45 and 1.0 eV have been observed previously for the Ga 3*d* lines.[41] In the spectra of the K covered and subsequently oxidized GaAs(110) surface [Fig. 7(b)], the stronger reacted components with an average BE shift of 4 eV are dominant, suggesting that the distribution of reaction products is similar to the one obtained in a clean GaAs(110) substrate after heavy exposures (10^7 L) of excited oxygen.[13]

IV. DISCUSSION

The adsorption of K atoms onto the GaAs surface results in a charge transfer to the semiconductor substrate.[42,43] The first indication of this behavior is a strong work-function decrease of the sample. The charge transferred to the substrate seems to facilitate the capture of impinging O_2 molecules. Some evidence has been collected that the charge transfer reduces locally, i.e., in the vicinity of the K adatoms, the barrier for electron transfer from the substrate to an antibonding level of the oxygen molecule forming an O_2^- ion.[42] The O_2^- ion has a much lower dissociation energy (3.8 eV) than the neutral O_2 molecule (5.2 eV), and therefore dissociation takes place more easily on alkali-covered GaAs surfaces. The net result of these processes is an enhancement of the sticking coefficient. Oxygen atoms are thus available for quick oxidation of the substrate. The extent of the reaction is limited by the total amount of electrons available in the substrate, which in turn depends on the number of K adatoms and their degree of charge transfer. The model schematically given above will be described in detail elsewhere,[42] but it should be noted that it also provides a common basis for mechanisms already put forward to explain other oxidation processes: the thermal oxidation of silicon,[44] the K-enhanced oxidation of carbon films,[45] and the photoenhanced oxidation of GaAs.[17]

It has been proposed recently that the oxidation rate of Si is dictated by the availability of free electrons at the surface, which in the case of Si already covered by a SiO_2 layer is determined by the thermionic electron flux from the conduction band of Si into the conduction band of SiO_2.[44] This model is supported by the fact that the activation energy yielding a thermionic electron flux equal to the experimental O_2 flux is within 0.2 eV of the barrier of 3.15 eV from the bottom of the Si conduction band.[44]

On the other hand, it was shown[17] that only light with a photon energy larger than the band gap of GaAs (but smaller than that required to dissociate oxygen molecules, i.e., 5.2 eV) enhances the oxygen uptake by increasing the sticking coefficient by a factor of 100 with respect to the sample kept in the dark. These data have been replotted as a dashed line in Fig. 3. In this case the enhancement in the oxygen uptake is related to the photocreated electrons and/or holes, i.e., oxygen molecules take up an electron at the surface from the electron-hole pair created by the photons and they get broken much more easily than on a nonilluminated surface.

In the case of K atoms deposited on GaAs, the enhancement is even stronger, the initial sticking coefficient rising to unity for $\Theta_K = 0.5$ ML. However, it should be pointed out that the K atoms are actually oxidized during this process. Upon mild annealing, the K-bonded oxygen is transferred to the substrate, leading to a further oxidation of GaAs. This observation suggests that the K oxides play an important role in the enhancement of the oxidation reaction in GaAs, as was previously demonstrated for Si.[25]

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