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# Etching of GaAs(100) with Aqueous Ammonia Solution: A Synchrotron-Photoemission Spectroscopy Study

Mikhail V. Lebedev,<sup>\*,†</sup> Eric Mankel,<sup>‡</sup> Thomas Mayer,<sup>‡</sup> and Wolfram Jaegermann<sup>‡</sup>

A. F. Ioffe Physical-Technical Institute, Russian Academy of Sciences, Politeknicheskaya 26, St. Petersburg, 194021, Russia and Institute of Material Science, Darmstadt University of Technology, Petersenstrasse 32, 64287 Darmstadt, Germany

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Etching of the GaAs(100) surface with aqueous ammonia solution is studied by highly surface-sensitive synchrotron-radiation photoemission spectroscopy. It is shown that such treatment effectively removes the native oxide layer leaving the surface covered with elemental arsenic, as well as arsenic hydroxides AsOH and As(OH)<sub>3</sub>, gallium hydroxide GaOH, and gallium suboxide Ga<sub>x</sub>O. After annealing of the surface at 500 °C, the arsenic and gallium hydroxides disappear, while the excess arsenic is dimerized. The residual carbon contamination prevents disappearance of gallium hydroxide and hinders dimerization of excess arsenic on annealing. Rinsing the etched surface with 2-propanol instead of water prior to annealing results in considerable reduction of carbon contamination after annealing.

## 1. Introduction

Cleaning of semiconductor surfaces is one of the most abundant and very important processing technologies. A clean surface is absolutely essential for various epitaxial growth techniques, as well as for surface studies. Wet chemical etching procedures are widely applied in the fabrication of semiconductor devices. As opposite to dry vacuum etching, wet chemical processing is impact-free and thus does not cause surface defect formation. Moreover, wet chemical processing is the simplest and easiest to control.

GaAs(100) is one of the most intensively studied semiconductor surfaces because of its importance in electronics and optoelectronics. Wet chemical etching is widely applied in preparation and conditioning of GaAs surface. Etching with aqueous ammonia (ammonium hydroxide) solution is often used as a processing step in fabrication of GaAs-based devices.<sup>1–4</sup> This etching solution removes the native oxide layer from the GaAs(100) surface.<sup>5</sup> Moreover, treatment with aqueous ammonia solution renders the surface properties suitable for atomic layer deposition of the gate dielectric.<sup>2</sup> However, the mechanism of GaAs(100) etching with aqueous ammonia solution is not clear yet, though this mechanism is important not only for understanding the wet etching process itself, but also for gaining insight into the mechanism of surface passivation with sulfide solutions<sup>6</sup> in which the hydroxyl groups play an important role. In particular, the chemistry of interaction of hydroxide ions with the surface of GaAs and its native oxide should be revealed and methods of reducing the oxygen and carbon surface contamination<sup>5</sup> should be found. To this end a detailed investigation of the surface composition at different processing steps is necessary. Synchrotron-photoemission spectroscopy offers a highly surface sensitive analysis providing the information on chemical bonding in the topmost surface atomic layers.

In this study, different stages of the wet etching of the GaAs(100) surface with aqueous ammonia solutions are studied by means of highly surface sensitive synchrotron-radiation photoemission spectroscopy. We compare photoemission spectra taken just after etching off the native oxide layer with aqueous ammonia solution, after rinsing the etched sample with water or with 2-propanol, as well as after annealing the etched and rinsed surfaces at 500 °C in ultrahigh vacuum (UHV).

## 2. Experimental Section

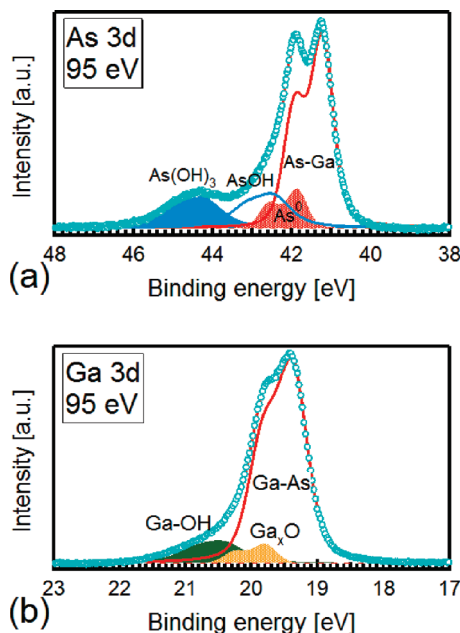
The experiments have been performed at the undulator beamline U49/2 of the BESSY II storage ring, which provides photons in the energy range between  $h\nu = 90$  and 1400 eV. Photoemission spectra were measured with the highest possible surface sensitivity. Ga 3d and As 3d core levels were measured with 95 eV excitation energy, while C 1s and O 1s core levels were measured using 350 and 650 eV excitation energy, respectively. At these energies the electron inelastic mean free path  $\lambda$  is approximately 5 Å<sup>7</sup> implying that roughly 63% of the signal stems from the first atomic layer, 23% from the second, and 9% from the third. The spectra were obtained using the Phoibos 150 (SPECS) energy analyzer of the experimental system “SoLiAS” (Solid/Liquid Analysis System)<sup>8</sup> permanently operated at BESSY. SoLiAS is equipped with an integrated electrochemistry chamber built from standard glass elements. This glass chamber is purged with inert, dry, carbon-free Ar gas and is directly attached to a special buffer chamber to allow for the transfer of the samples into UHV without contact to ambient atmosphere.

The GaAs(100) samples used in this study were cut from an n-type wafer with an epitaxially grown top layer having a carrier concentration of about  $10^{18}$  cm<sup>-3</sup>. A drop of commercial 25% aqueous ammonia solution was applied to the substrate surface for 90 s. After this treatment, the solution was blown off by an Ar jet and the sample was transferred into the analysis chamber. After photoemission analysis of the etched sample it was transferred back into the electrochemical chamber where its surface was rinsed either with water (H<sub>2</sub>O) or with 2-propanol (2-C<sub>3</sub>H<sub>7</sub>OH). All wet preparation was carried out within the Ar-

\* To whom correspondence should be addressed. E-mail: mlebedev@triath.ioffe.ru. Fax: +7(812) 297 10 17.

<sup>†</sup> A. F. Ioffe Physical-Technical Institute.

<sup>‡</sup> Darmstadt University of Technology.



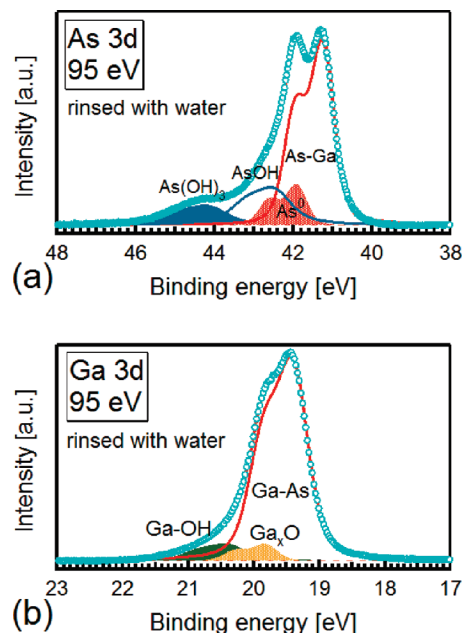
**Figure 1.** Photoemission spectra of As 3d (a) and Ga 3d (b) core levels collected from the GaAs(100) surface after etching the native-oxide layer with aqueous ammonia solution. The excitation energy was 95 eV. The dots represent the collected data after background subtraction. The spectra have been deconvoluted in the assigned components by a fitting routine.

purged glass chamber. Annealing of the etched and rinsed samples was performed in a separate UHV preparation chamber of the integrated system SoLiAS. The typical base pressure in the preparation chamber is  $5 \times 10^{-10}$  mbar, it rises in the beginning of annealing to  $1 \times 10^{-6}$  mbar, and goes back at the end of annealing to the range of  $5 \times 10^{-9} - 1 \times 10^{-8}$  mbar. The annealing temperature was controlled by a thermocouple positioned in the vicinity of the sample.

### 3. Results

Before etching both As 3d and Ga 3d core-level spectra measured with an excitation energy of 95 eV contain mainly the emission stemming from the native oxide layer, in accordance with ref 5. The As–Ga and Ga–As bulk components are hardly visible.

The As 3d and Ga 3d core-level spectra were fitted using Voigt functions with similar parameters (Gaussian width for measurement uncertainty, Lorentzian width for lifetime broadening, spin–orbit splitting, branching ratio) as used in ref 9. The As 3d spectrum of the GaAs(100) surface etched with aqueous ammonia solution can be fitted well using four different components (Figure 1a): the bulk As–Ga emission and three components shifted to higher binding energies by 0.6, 1.2, and 3.0 eV. The component with the chemical shift of +0.6 eV can be assigned to the excess elemental arsenic As<sup>0</sup>.<sup>9</sup> The energy position of the latter component is similar to that obtained for the arsenic oxide As<sub>2</sub>O<sub>3</sub> (3.2 eV).<sup>10,11</sup> However, taking into account the occurrence of a large amount of OH<sup>−</sup> ions in the solution, we tentatively assign this component to arsenic hydroxide As(OH)<sub>3</sub>. A component with exactly the same chemical shift was observed before after contact of the oxide-free GaAs(100) surface with aqueous ammonia solution and similarly assigned to As(OH)<sub>3</sub>.<sup>12</sup> With the same argument, the component with the chemical shift of 1.2 eV can be tentatively assigned to arsenic hydroxide AsOH. The Ga 3d spectrum of the GaAs(100) surface etched with aqueous ammonia solution

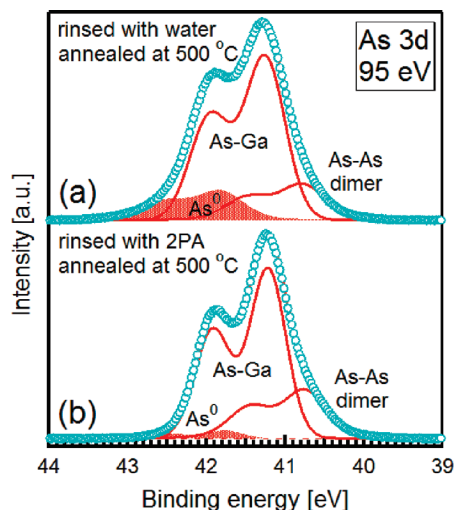


**Figure 2.** Photoemission spectra of As 3d (a) and Ga 3d (b) core levels collected from the GaAs(100) surface etched with aqueous ammonia solution and rinsed with water. The excitation energy was 95 eV. The dots represent the collected data after background subtraction. The spectra have been deconvoluted in the assigned components by a fitting routine.

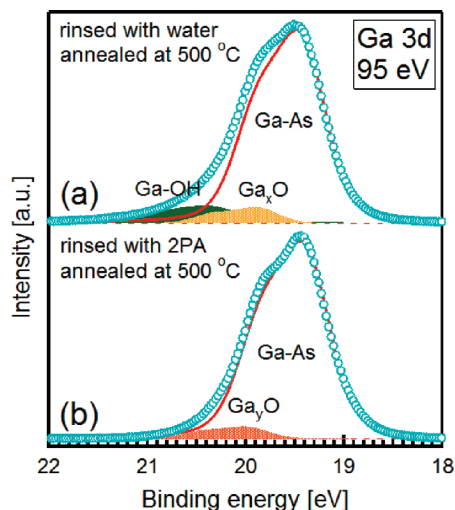
can be fitted well using three different components (Figure 1b): the bulk Ga–As emission and two components shifted to higher binding energies by 0.4 and 0.9 eV. The component with a chemical shift of about 0.9 eV was observed previously after etching the GaAs(110) surface with a Br<sub>2</sub>/H<sub>2</sub>O solution and was assigned to gallium hydroxide GaOH.<sup>13</sup> The other chemically shifted component can be assigned to gallium suboxide Ga<sub>3</sub>O. The ratio of integrated As 3d to Ga 3d emissions is about 1.5 if the cross-section correction<sup>14</sup> is taken into account. The cross-section corrected ratio of the bulk photoemission components As–Ga/Ga–As is equal to 1 as should be expected.

When the GaAs(100) surface after etching with aqueous ammonia solution is rinsed with water or with 2-propanol, the As 3d and Ga 3d spectra are changed slightly (Figure 2). In particular, the component tentatively assigned to As(OH)<sub>3</sub> and the GaOH-related component are decreased, while the elemental arsenic component is increased. As a result, the ratio of integrated As 3d to Ga 3d emissions increases by 5–7%. No essential difference between As 3d and Ga 3d core-level spectra obtained from the etched GaAs(100) surface after rinsing with water or with 2-propanol was observed.

After annealing, the As 3d and Ga 3d core-level spectra are changed considerably (Figures 3 and 4). In particular, after annealing at 500 °C, the components tentatively assigned to arsenic hydroxides disappear from the As 3d spectrum, which supports this assignment. Disappearance of these components is accompanied by the reduction of the cross-section corrected ratio of integrated As 3d to Ga 3d emissions to 0.9. Simultaneously, a new component shifted to low binding energy by 0.5 eV appears in the As 3d spectra (Figure 3), which can be assigned to As–As surface dimers.<sup>9</sup> Moreover, the relative amount of the components of the As 3d and Ga 3d spectra depends on whether the surface is rinsed with water or with 2-propanol before annealing. In particular, in the As 3d spectrum the intensity of the elemental arsenic component As<sup>0</sup> is lower and the intensity of the As–As surface dimer component is



**Figure 3.** Photoemission spectra of As 3d core level collected after annealing at 500 °C in UHV of the GaAs(100) surface etched with aqueous ammonia solution and rinsed with water (a) or 2-propanol (b). The excitation energy was 95 eV. The dots represent the collected data after background subtraction. The spectra have been deconvoluted in the assigned components by a fitting routine.



**Figure 4.** Photoemission spectra of Ga 3d core level collected after annealing at 500 °C in UHV of the GaAs(100) surface etched with aqueous ammonia solution and rinsed with water (a) or 2-propanol (b). The excitation energy was 95 eV. The dots represent the collected data after background subtraction. The spectra have been deconvoluted in the assigned components by a fitting routine.

higher when the surface was rinsed with 2-propanol prior to annealing (Figure 3). On the other hand, the Ga 3d spectrum of the surface obtained after annealing the etched GaAs(100) surface rinsed with water has not changed considerably with respect to the spectrum obtained prior to annealing, whereas in the Ga 3d spectrum obtained after annealing the etched GaAs(100) surface rinsed with 2-propanol, only a small Ga-suboxide-related component with a chemical shift of +0.6 eV<sup>5</sup> remains (Figure 4).

The relative elemental concentrations of oxygen and carbon after all stages of processing are shown in Table 1. These concentrations were determined from the ratios of the areas of corresponding integrated emissions taking into account the cross-section correction.<sup>14</sup>

The evolution of the O 1s core-level spectra after etching the GaAs(100) surface with aqueous ammonia solution, rinsing with water or 2-propanol, and subsequent annealing is shown

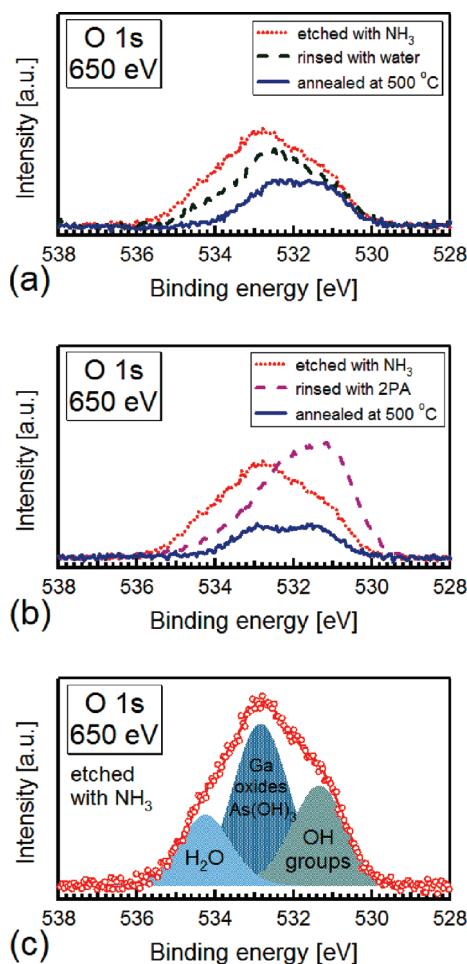
**TABLE 1: Cross-Section Corrected Relative Elemental Composition at GaAs(100) Surface after Various Treatments**

surface	O 1s (650 eV)/ Ga 3d (95 eV)	C 1s (350 eV)/ Ga 3d (95 eV)
native oxide	3.9	4.0
etched with ammonia solution	3.5	4.1

	water rinsed	2PA rinsed	water rinsed	2PA rinsed
rinsed	2.2	1.8	3.5	1.1
annealed at 500 °C	0.42	0.23	0.52	0.052

in Figure 5a,b. The spectra are normalized to the beam current of the electrons in the storage ring, that is, to the photon flux density and plotted after Shirley background subtraction.<sup>15</sup> The O 1s spectrum of the GaAs(100) surface obtained after etching off the native oxide layer with aqueous ammonia solution can be fitted with three components (Figure 5c). The relative intensities of these components are changed after rinsing the etched surface with water or with 2-propanol. After annealing the surfaces at 500 °C, the high-binding-energy component disappears and the intensities of the other two components decrease considerably. The O 1s spectra after annealing the



**Figure 5.** Core-level O 1s spectra obtained after etching the GaAs(100) surface with aqueous ammonia solution, rinsing with water (a) or 2-propanol (b) and annealing at 500 °C. (c) Deconvolution of the spectrum of O 1s core-level collected from the GaAs(100) surface after etching the native-oxide layer with aqueous ammonia solution. The excitation energy was 650 eV. The dots represent the collected data after background subtraction. The spectrum has been deconvoluted in the assigned components by a fitting routine.



etched GaAs(100) surface rinsed with water or 2-propanol have similar shape (Figure 5), but the intensity of the O 1s emission with respect to the Ga 3d emission is lower by a factor of 2 when the GaAs(100) surface was rinsed with 2-propanol prior to annealing (Table 1).

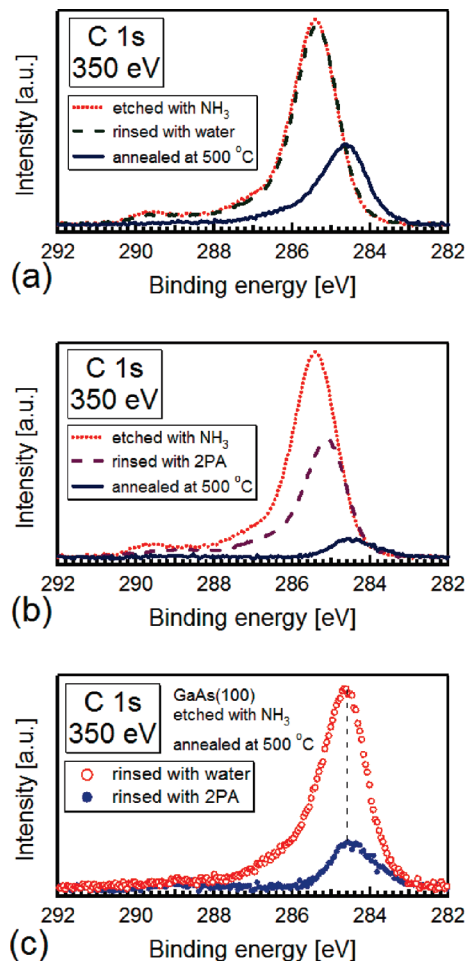
The lowest-binding-energy component positioned at about 531.3 eV can be assigned to surface hydroxyl groups (Ga–OH and As–OH species). This component increases considerably after rinsing the etched GaAs(100) surface with 2-propanol (Figure 5b) indicating 2-propanol molecules sticking to the surface.<sup>16</sup> The highest-binding-energy component that occurred at about 534.2 eV can be assigned to H<sub>2</sub>O<sup>17</sup> adsorbed from etching solution. Rinsing of the etched GaAs(100) surface with water results in some reduction in the intensity of this component (Figure 5a), while after rinsing the etched GaAs(100) surface with 2-propanol this component becomes hardly visible (Figure 5b). After annealing the surface at 500 °C, this component disappeared completely from the O 1s spectra (Figures 5a,b). A similar component appeared in the O 1s photoemission after the interaction of the oxide-free GaAs(100) surface with aqueous ammonia solution.<sup>12</sup> The middle component of the O 1s spectra can be assigned to gallium oxides and As(OH)<sub>3</sub> species.<sup>12</sup>

The evolution of the C 1s core-level spectra after etching the GaAs(100) surface with aqueous ammonia solution, rinsing with water or 2-propanol, and subsequent annealing is shown in Figure 6a,b. The spectra were normalized to the beam current of the electrons in the storage ring and plotted after Shirley background subtraction.<sup>15</sup> Rinsing with water has no obvious effect on the shape and intensity of the C 1s emission (Figure 6a). After annealing, the C 1s emission is shifted by 0.8 eV to low binding energies and its intensity is reduced by a factor of 2.5, approximately. It should be noted that the other core-level spectra (O 1s, As 3d, Ga 3d) shift very little (by 0.2 eV or less); therefore, the shift of the C 1s emission is not caused by the band bending.

On the other hand, after rinsing the etched GaAs(100) surface with 2-propanol, the intensity of the C 1s emission reduces by a factor of approximately 2 and the ratio of integrated C 1s to Ga 3d emissions by a factor of 4 (Table 1). Simultaneously, the total spectrum is shifted to lower binding energies by 0.3 eV, which was accompanied by a similar shift of the low-binding-energy component in the corresponding O 1s spectrum (Figure 5b). The position of the Ga 3d and As 3d core-level spectra remains essentially unchanged. The intensity of the C 1s spectrum is reduced by an order of magnitude after annealing the etched GaAs(100) surface rinsed with 2-propanol (Figure 6b). Moreover, the ratio of the integrated C 1s to Ga 3d emissions in this case is lower by an order of magnitude than the same ratio obtained after annealing the etched GaAs(100) surface rinsed with water (Table 1). Comparison of the C 1s emission after annealing the etched GaAs(100) surfaces rinsed with water or with 2-propanol (Figure 6c) shows that in the 2-propanol case the high-binding-energy shoulder at about 286 eV has disappeared. The main peak of the residual C 1s emission with a binding energy of 284.6 eV can be assigned to graphitic carbon/hydrocarbon species.<sup>18</sup> The shoulder at a binding energy of 286.0 eV can be associated with C–O/C–OH bonds.<sup>18</sup>

#### 4. Discussion

The native-oxide layer on the GaAs(100) surface consists of a mixture of elemental arsenic As<sup>0</sup>, different arsenic oxides, such as As<sub>2</sub>O, AsO, and As<sub>2</sub>O<sub>3</sub>, and different gallium oxides, such as Ga<sub>2</sub>O, GaO, and Ga<sub>2</sub>O<sub>3</sub>.<sup>10</sup> Aqueous ammonia solution acts as a complexing agent for oxidized Ga and As species



**Figure 6.** Core-level C 1s spectra obtained after etching the GaAs(100) surface with aqueous ammonia solution, rinsing with water (a) or 2-propanol (b) and annealing at 500 °C. (c) Comparison of the core-level C 1s spectra collected after annealing at 500 °C the GaAs(100) surface etched with aqueous ammonia solution and rinsed with water or 2-propanol. The excitation energy was 350 eV.

forming soluble compounds such as (NH<sub>4</sub>)<sub>3</sub>AsO<sub>4</sub> and NH<sub>4</sub>Ga(OH)<sub>4</sub>.<sup>19</sup> As a result, the native oxide layer is removed from the GaAs(100) surface after contact with aqueous ammonia solution (Figure 1). On the other hand, comparison of the As 3d and Ga 3d spectra shown in Figure 1 with those obtained after the contact of the oxide-free GaAs(100) surface with aqueous ammonia solution<sup>12</sup> shows astonishing similarity. In both cases, the surface is As-rich and covered mostly with elemental arsenic and As hydroxides like AsOH and As(OH)<sub>3</sub>. Therefore, we can conclude that after etching off the native oxide layer the OH<sup>−</sup> ions of the basic solution are adsorbed at the pristine GaAs(100) surface forming surface arsenic and gallium hydroxides. However, the gallium hydroxides are soluble in aqueous ammonia solution<sup>19</sup> and washed away from the surface, which results in the increase in the As-to-Ga ratio. The insoluble elemental arsenic and arsenic hydroxide surface layer passivates the surface forming an etch stop. It should be noted that As(OH)<sub>3</sub> is not observed after exposure of the wet processed sample to air,<sup>19</sup> which is avoided in our experimental procedure.

Before and after rinsing with water the O 1s core-level spectra of the etched GaAs(100) surface contain a high-binding-energy component assigned to water<sup>17</sup> (Figure 5a), which may be expected as the etched surface is covered by dipolar hydroxyl groups that should form hydrogen bonds with water molecules. The occurrence of hydroxyl groups may explain hydrophilicity

of the GaAs(100) surface etched with aqueous ammonia solution as found by contact-angle measurements.<sup>20,21</sup> The quantitative analysis of the composition of the GaAs(100) surface after the etching with aqueous ammonia solution (Figure 1) indicates that the hydroxide coverage is approximately one monolayer thick.

Rinsing the etched GaAs(100) surface with water results in some increase in the ratio of integrated As 3d to Ga 3d emissions and causes some reduction in the oxygen contamination (Figure 5a), while the carbon contamination is not varied (Figure 6a). After rinsing with water the intensities of the components assigned to As(OH)<sub>3</sub> and GaOH are reduced, while the elemental arsenic component is increased somewhat (Figure 2). The increase in the As-to-Ga ratio indicates that water rinsing washes away some weakly bound GaOH species. In addition, some of the hydroxyl groups are withdrawn from As–OH and the elemental arsenic concentration is increased. Accordingly the O 1s spectrum indicates some decrease of the oxygen contamination; particularly the water-related component is decreased due to the decreased surface concentration of hydrophilic OH-groups (Figure 5a). The fact that the shape of the C 1s spectrum after water rinsing remains unchanged indicates that water does not react with the residual carbon contamination.

Similarly, rinsing the etched GaAs(100) surface with 2-propanol results in decreasing the amount of surface As(OH)<sub>3</sub> and GaOH species, whereas the elemental arsenic component and the As-to-Ga ratio are increased. Accordingly in the O 1s spectrum the As(OH)<sub>3</sub> related component (Figure 5b) is reduced somewhat. The low binding energy component of the O 1s spectrum increases considerably, which evidence the adsorption of 2-propanol molecules. The water component in the O 1s core-level spectrum is hardly visible (Figure 5b), that is, water adsorbed after etching is probably substituted with 2-propanol molecules after rinsing. On the other hand, there is no evidence of the dissociative adsorption of 2-propanol molecules<sup>16</sup> on the GaAs(100) surface etched with aqueous ammonia solution. So, it can be concluded that all surface dangling bonds on the etched GaAs(100) surface are saturated with hydroxyl groups and 2-propanol molecules are bound to these surface hydroxyl groups. This conclusion is supported by the fact that after etching with aqueous ammonia solution the GaAs(100) surface is covered with approximately one monolayer of hydroxide.

As opposed to the case of rinsing with water, rinsing with 2-propanol reduces considerably the intensity of the C 1s emission on etched GaAs(100) (Figure 6b). Moreover, the C 1s spectrum shifts to lower binding energies to an energy position obtained after contact of clean GaAs(100) surface with 2-propanol at room temperature.<sup>16</sup> Also the shapes of the C 1s spectra coincide in these two situations. Thus, it can be concluded that 2-propanol dissolves the residual carbon contamination in a great extent and removes it from the GaAs(100) surface. As a consequence, rinsing the etched GaAs(100) surface with 2-propanol instead of water results in considerably reduced carbon contamination after annealing in UHV (Figure 6c). It should be noted that the carbon contamination hinders dimerization of the excess elemental arsenic on the surface (cf, Figure

3a,b) and disappearance of gallium hydroxide on annealing (cf. Figure 4a,b).

## 5. Conclusions

Synchrotron-radiation photoemission spectroscopy has been used to study etching of the native oxide covered GaAs(100) surface with aqueous ammonia solution and annealing at 500 °C after rinsing with 2-propanol as compared to water. It has been shown that etching effectively removes the native oxide layer leaving the surface covered with elemental arsenic, arsenic and gallium hydroxides, as well as gallium suboxide. The GaAs(100) surface obtained after etching with aqueous ammonia solution is hydrophilic due to the occurrence of surface hydroxyl groups. After annealing of the surface at 500 °C, the arsenic hydroxides disappear and excess arsenic is dimerized, while gallium hydroxides are converted to residual suboxide. The residual carbon contamination prevents the disappearance of gallium hydroxide and hinders dimerization of excess arsenic on annealing. Rinsing the etched surface with water does not change the carbon contamination before annealing. Rinsing with 2-propanol before annealing results in considerable reduction of the carbon contamination before and even more so after annealing.

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