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Microstructure and wet oxidation of low-temperature-grown amorphous (Al/Ga,As)


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Amorphous and polycrystalline compounds of (Ga,As) and (Al,As) grown at very low temperatures by molecular-beam epitaxy are characterized. The ultimate microstructure and the amount of excess arsenic incorporated in the (Ga,As) or (Al,As) layers are found to depend on the arsenic overpressure during the low-temperature growth. With lower arsenic overpressure, a polycrystalline structure prevails and less excess arsenic is observed inside the layer. In contrast, a high incorporation of excess arsenic achieved by high-arsenic overpressures leads to the formation of amorphous films. Upon wet oxidation, the lateral oxidation rate of (Al,As) is found to depend on the crystallinity of the (Al,As) layer and the amount of excess arsenic. During the same process, recrystallization proceeds in the (Ga,As) layer.

I. INTRODUCTION

Since its discovery in 1990, the robust native oxide formed by water-vapor oxidation of crystalline Al-bearing compound semiconductor has been used in many applications ranging from electronic to optical devices. The oxide of AlGaAs compounds via wet oxidation is mechanically robust and chemically stable. It has been used as the gate oxide for III–V-based field-effect transistors (FETs), although only depletion-mode devices were reported. To realize an enhancement-mode FETs requires further optimization of oxidation processing to reduce the high density of interface traps, residual As, and microcrystallites inside the oxides. Applications of oxides, to date, have predominantly focused on optical device fabrication. The nonconducting nature allows the oxide to confine electrical current, and the selective lateral oxidation with respect to Al concentration facilitates the formation of distributed Bragg reflectors (DBRs) for vertical-cavity surface-emitting lasers (VCSELs). A large reduction in the refractive index results from 3.2 to 1.6 when AlAs is oxidized. The larger difference of refractive indices between the Al oxide and GaAs achieved by laterally oxidizing the initial AlAs/GaAs pair permits the formation of effective DBR mirrors without the need for a large number of pairs. Consequently, a reduced number of DBR pairs is required, which lessens the stringent requirement in epitaxial growth control. In addition, the larger difference of refractive indices benefits from a wide bandwidth of maximum reflection which is also critical to VCSEL operation. Although oxides formed from Al-bearing compounds have shown great advantages in various applications, they are almost exclusively grown on lattice-matched systems, such as AlGaAs/GaAs, InAlAs/InP, and AlGaSb/GaSb. Little attention has been paid to wet oxidation.

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II. EXPERIMENT

Low-temperatures MBE (LT-MBE) materials have been grown at a substrate temperature around 100 °C, measured by the substrate thermocouple. For all samples used in this work, a GaAs buffer layer was first grown at 580 °C after desorption of the surface oxides was complete. The wafer was then allowed to cool to about 100 °C for the deposition of amorphous compounds. The arsenic flux is provided and controlled by a valved solid-arsenic cracker. During deposition, three As overpressures were chosen: $5 \times 10^{-7}$, $2 \times 10^{-7}$, and $8 \times 10^{-8}$ Torr. Reflection high-energy electron diffraction (RHEED) intensity oscillations were used to determine the column-III fluxes (either Ga or Al), and hence, the growth rate at normal growth temperatures. In most cases, a growth rate of 0.5 ML per second is used. The same fluxes were then maintained during the growth of low-temperature compounds. It was noted that a featureless RHEED pattern was seen, indicative of amorphous growth, during low-temperature epitaxy with As overpressures at $5 \times 10^{-7}$ or $2 \times 10^{-7}$ Torr. For the $8 \times 10^{-8}$ Torr case, however, a faint ring structure in the RHEED pattern is observed, which indicates the possible growth of polycrystalline material.

Water-vapor oxidation of the low-temperature-grown compounds was performed in a 2 in. open-tube furnace. The water vapor is carried by N$_2$ gas bubbling through water held at 85 °C at a flow rate of 100 sccm. The oxidation has been performed at temperatures ranging from 270 to 500 °C.

All samples are examined by cross-sectional transmission electron microscopy (XTEM) to determine the film thickness and growth rate with a Philips CM-12 operated at 120 kV. Compositions of the low-temperature arsenides and their depth profiles of the atomic constituents were analyzed by scanning Auger electron spectroscopy (AES) with a cylindrical mirror analysis detector.

III. RESULTS AND DISCUSSION

A. Low-temperature growth of (Ga,As) and (Al,As) compounds

Two series of (Ga,As)/(Al,As) stacks have been used to study the growth rate of the low-temperature materials. The stacks of (Ga,As) and (Al,As) layers are grown on GaAs substrates with different As overpressures. In the first series, the (Ga,As) layers are grown with different As overpressures of $5 \times 10^{-7}$, $2 \times 10^{-7}$, and $8 \times 10^{-8}$ Torr, which are separated by three (Al,As) spacer layers all grown with the same As overpressure of $5 \times 10^{-7}$ Torr. Each layer [either (Ga,As) or (Al,As)] is designed to be 500 Å thick, according to the RHEED intensity oscillations, and was grown at a rate of 0.5 ML per second. It is known that the excess As is incorporated in the form of antisite defects in crystalline GaAs deposited at low temperatures of around 200 °C with approximately 1% excess As in the film. Since the growth temperature in this work is even lower, it is expected that more than 1% excess As will be incorporated. As a reference, the thermal equilibrium vapor pressure of As$_3$ reduces dramatically from $10^{-5}$ Torr at 200 °C to $10^{-10}$ Torr at 100 °C. Thus, the sticking coefficient of As during growth at 100 °C is expected to increase significantly, but by how much is not certain.

The actual thickness of each layer is measured by XTEM analysis and the growth rate is calculated. The growth rate increases from 83 to 107 Å/min as the pressure increases from $8 \times 10^{-8}$ to $5 \times 10^{-7}$ Torr. (b) shows a cross-sectional TEM micrograph of (Ga,As)/(Al,As) heterostructures. Each (Al,As) layer is grown with a different As overpressures from top to bottom: $8 \times 10^{-8}$, $2 \times 10^{-7}$, and $5 \times 10^{-7}$ Torr, respectively. All (Ga,As) layers are grown at 5 Torr As overpressure. The (Al,As) layer at $8 \times 10^{-8}$ Torr shows the polycrystalline structure and other layers are amorphous in nature.
The 30% increase in the growth rate of (Ga,As) as As overpressure increases from $8 \times 10^{-8}$ to $5 \times 10^{-7}$ Torr indicates that a considerable amount of excess As is incorporated during growth at 100 °C. Moreover, RHEED and XTEM both indicate that (Ga,As) grown with As overpressures at $2 \times 10^{-7}$ and $5 \times 10^{-7}$ Torr are actually amorphous. Apparently, the flux of As vapor is so high when it impinges on the sample that it changes the long-range crystalline GaAs structure into mostly short-range ordering. To evaluate the amount of excess As in the different layers of the (Ga,As)/(Al,As) heterostructures, we have employed AES. By comparing the AES spectra of Ga peaks obtained from the low-temperature-grown (Ga,As) and the crystalline GaAs substrate, we have noticed that both peak shape and position are very close, suggesting a similar surrounding environment for Ga atoms in these two compounds. This is expected because As is the excess constituent in the LT (Ga,As). On the other hand, the arsenic AES spectrum varies, indicating that As atoms experience different neighboring atomic arrangements in LT (Ga,As) and zinc-blende GaAs. Consequently, the sensitivity factors for As Auger electrons differ. Quantitative assessment of As concentration in the LT (Ga,As) as a function of As overpressure during growth would be incorrect if the sensitivity factor for As obtained from crystalline GaAs is used. Instead, we have used the sensitivity factor for As obtained from (Ga,As) grown at As overpressure of $8 \times 10^{-8}$ Torr to determine the excess As in the (Ga,As) layers deposited with As overpressures of $2 \times 10^{-7}$ and $5 \times 10^{-7}$ Torr. We found the excess As to be 39% and 66%, respectively, for layers grown at $2 \times 10^{-7}$ and $5 \times 10^{-7}$ Torr. Assuming the As/Ga ratio in the LT (Ga,As) grown at $8 \times 10^{-8}$ Torr is close to 1, supported by the stoichiometric polycrystalline zinc-blende structure, we have obtained, concentration-wise, (Ga,As$_{1.39}$) and (Ga,As$_{1.60}$) for the LT amorphous (Ga,As) compounds grown at $2 \times 10^{-7}$ and $5 \times 10^{-7}$ Torr. To further justify the quantitative assessment of excess As, we need to compare the growth rate for LT (Ga,As). The molar volumes for crystalline As and Ga are 13.1 and 11.8 cm$^3$. For zinc-blende GaAs it is 27.2 cm$^3$, which contains 2 mol of atoms, one for Ga and the other As.$^{20}$ From the molar volume point of view, 2 mol of As atoms will make up just about the same volume as 1 mol of GaAs. Therefore, incorporation of excess As during deposition of GaAs should not drastically change the molar volume, and hence, the growth rate. To an accuracy within 10%, amorphous (Ga,As), whose composition is Ga$_{1+x}$As$_{1+x}$, should occupy a volume similar to that of $(1+x/2)$ times zinc-blende GaAs, i.e., increased growth rates. Consequently, we can deduce the excess As from the growth rates in Fig. 1(a). They are (Ga$_{1.39}$As$_{1.39}$) and (Ga$_{1.60}$As$_{1.60}$) for LT arsenides deposited with As overpressures at $2 \times 10^{-7}$ and $5 \times 10^{-7}$ Torr, respectively. The obtained excess As values are reasonably consistent with those obtained from AES spectroscopy, given that the As sensitivity factor may vary as the excess As increases with high-As overpressure.

In the second series of (Ga,As)/(Al,As) stacks, the growth conditions have been switched. Amorphous (Ga,As) layers are used as spacer layers and have been grown with a fixed As overpressure at $5 \times 10^{-7}$ Torr. The three (Al,As) layers are grown with As overpressures at $5 \times 10^{-7}$, $2 \times 10^{-7}$ and $8 \times 10^{-8}$ Torr. Figure 1(b) shows the microstructure of each layer. The top (Al,As) layer is grown at As overpressure $8 \times 10^{-8}$ Torr. The TEM analysis shows it has a polycrystalline microstructure. The bottom (Al,As) ($5 \times 10^{-7}$ Torr) and middle (Al,As) ($2 \times 10^{-7}$ Torr) layers are amorphous. The crystallinity change in (Al,As) with the As overpressure is similar to that in (Ga,As).

The same method was used to find the growth rate and excess As in (Al,As) of the second series of (Ga,As)/(Al,As) heterostructures. The growth rate of the (Al,As) layers determined by TEM increases from 85 to 134 A/min with increasing the As overpressure, as shown in Fig. 1(a). The growth rate of amorphous (Al,As) was found to be higher than that of (Ga,As) in the first stack by about 20%. It is believed that the discrepancy is a result of variation in the measured substrate temperature. A small difference in deposition temperature between the two samples can, in fact, induce a significant difference in excess As incorporation. However, the general trend in both materials is that the growth rate increases with the As overpressure. Consequently, for the (Al,As) layers, the AES indicated that 18%, 40%, and 92% excess As are incorporated in the film for As overpressures of $8 \times 10^{-8}$, $2 \times 10^{-7}$, and $5 \times 10^{-7}$ Torr, respectively. The volume change obtained from the layer thickness indicated that 38% and 87% excess As are incorporated in the (Al,As) layers grown at overpressures of $2 \times 10^{-7}$ and $5 \times 10^{-7}$ Torr, respectively. The numbers are consistent with those determined from the AES analyses. Our experimental results obtained from both test samples suggest that excess As incorporated in (Ga/Al)As layers grown at low temperatures increases with the As overpressure once it exceeds the critical pressure ($8 \times 10^{-8}$ Torr, in our experiments).

**B. Wet oxidation and recrystallization of (Ga,As) and (Al,As)**

Since oxidation involves higher temperatures than those used for film deposition, recrystallization of as-grown films due to thermal annealing can take place. The postoxidation morphology is actually a combined effect of both oxidation and annealing. In this work, the same samples were used for studying the recrystallization and oxidation of amorphous (Ga,As) and (Al,As) grown with different As overpressures. Wet oxidation is conducted laterally. The samples were patterned with 20-μm-wide regions separated by 5 μm trenches using standard photolithography and reactive ion etching (RIE) with SiCl$_4$. The sample was then placed in the wet-oxidation furnace to be oxidized at 300 °C for 40 and 120 min. To study the rate of oxidation and recrystallization of LT (Ga,As), experiments were performed on the first sample with (Ga,As) layers grown at different arsenic overpressures, as shown in Fig. 2(a). TEM analysis shows amorphous multilayers except for the (Ga,As) layer grown at...
8 × 10⁻⁸ Torr, which appears to be composed of polycrystalline zinc-blende GaAs. For 40 min at 300 °C, neither the (Al,As) nor the (Ga,As) layers oxidize completely laterally through the 20 μm distance except the outermost (Al,As) cap layer which has been exposed to water directly and oxidized from the top completely. Practically speaking, layers in the middle of the regions which have not been oxidized laterally have essentially just been thermally annealed. Any morphological change is a result of recrystallization, as shown in Fig. 2(b). Polycrystalline zinc-blende GaAs structures appear in both (Ga,As) layers grown at 8 × 10⁻⁸ and 2 × 10⁻⁷ Torr, respectively. It is obvious that recrystallization of the original amorphous (Ga,As) layer grown at 2 × 10⁻⁷ Torr has taken place, but there is little volume shrinkage indicative of As loss. In the amorphous 1-(Ga,As) layer grown with the highest As overpressure, however, excess As has at least slowed down the recrystallization process, if not completely prevented it.

In the longer oxidation for 120 min, complete lateral oxidation of the 20 μm region of all (Al,As) layers deposited at 5 × 10⁻⁷ Torr is seen, as shown in Fig. 2(c), resulting in an amorphous or very-fine-grained structure. In addition, The two top (Ga,As) layers remain polycrystalline. Surrounded by oxidized (Al,As) layers, all three (Ga,As) layers are exposed to water vapor and vertical oxidation can take place. The TEM microstructures indicated that the top polycrystalline (Ga,As) layer deposited with the least As overpressure remains polycrystalline and intact without any apparent structural deterioration when oxidized for 120 min, at 300 °C. The middle (Ga,As) layer recrystallizes from amorphous to polycrystalline and sustains a minor structural deterioration near both interfaces during complete lateral oxidation of the neighboring (Al,As) layers. The bottom (Ga,As) layer, however, has completely disintegrated and oxidized with water vapor provided from the oxidized (Al,As) layers without the benefit of first recrystallizing itself into a zinc-blende GaAs polycrystalline structure. Apparently, crystalline zinc-blende GaAs is rather inert to water oxidation at 300 °C. The rate of oxidation is too low to obtain directly, and extrapolation from existing data is needed.

In a separate experiment, we have conducted wet oxidation of crystalline GaAs wafers from the surface at temperatures higher than 300 °C for different times to determine the oxidation rate. Combining the results and those from Oh et al.,²¹ we have determined the activation energy of wet-oxidizing crystalline GaAs to be 1.4 eV in the temperature range from 400 to 520 °C, as shown in Fig. 3. By extrapolation, an oxidation rate of 7 Å/h at 300 °C for crystalline GaAs is obtained. The estimated low oxidation rate is consistent with the observed negligible structural deterioration during the oxidation of the polycrystalline (Ga,As) layer. The low oxidation rate also suggests that any crystalline GaAs grains in the amorphous (Ga,As) layer grown at 5 × 10⁻⁷ Torr, if ever induced by heat treatment during lateral oxidation, are very small (in the order of 7 Å) so that they have been easily oxidized, leaving little traces for recognition.

FIG. 2. Cross-sectional TEM micrograph of a second set of (Ga,As)/(Al,As) heterostructures. (a) is the microstructure of the as-grown sample. LT-Ga,As layers are grown at different arsenic overpressures: from top to bottom 8 × 10⁻⁸, 2 × 10⁻⁷, and 5 × 10⁻⁷ Torr surrounded by LT-Al,As layers all grown with the same arsenic overpressure of 5 × 10⁻⁷ Torr. (b) shows the microstructure of the middle part of a 20 μm region of the sample oxidized at 300 °C for 40 min. The top (Al,As) layer is already oxidized and the other (Al,As) layers are not oxidized because the oxidation is vertically blocked by the top (Ga,As) polycrystalline layer. Also, 40 min is too short for a complete lateral oxidation. The middle (Ga,As) layer starts to recrystallize during the oxidation/annealing process. (c) shows the structure of the sample which has been oxidized for 120 min. All (Al,As) layers are completely oxidized laterally and the bottom (Ga,As) layer is also oxidized by the water-vapor transported through the surrounding (Al,O) layers.
C. Wet oxidation of (Al,As)

It is generally known that single-crystal AlAs easily hydrolyzes once it is exposed in the air due to its high chemical reactivity with moisture. One immediately recognizes the color change in an unprotected epitaxial AlAs film when it is removed from the growth chamber. Common practice in designing Al$_{1-x}$Ga$_x$As/GaAs heterostructures requires that an inert GaAs cap layer be deposited on the Al$_{1-x}$Ga$_x$As film, especially when the Al content is high. For this reason, we planned not to conduct wet oxidation of the arsenic-rich amorphous (Al,As) compound directly from the surface. Instead, lateral oxidation is performed. To determine the lateral oxidation rate and to study its relationship with As overpressure during growth, and hence, the amount of excess As in the (Al,As) film, we have used three separate samples with the same thickness of the (Al,As) layer. The samples were grown by LT-MBE with different As overpressures of 8 × 10$^{-8}$, 2 × 10$^{-7}$ and 5 × 10$^{-7}$ Torr, respectively. Each of the (Al,As) films was capped with a 1000 Å polycrystalline (Ga,As) passivation layer, which was grown at 8 × 10$^{-5}$ Torr.

Each sample was delineated into stripe patterns with standard lithography and reactive ion etching. They were then oxidized at 300, 350, and 400 °C for different lengths of time with 85 °C water vapor and 100 sccm N$_2$ as the carrier gas. The oxidation front, and hence, the oxidized distance from the edges of the stripes, can be examined with an optical microscope. The results of lateral oxidation at 300 °C are shown in Fig. 4. To gain some insight of the chemical dependence on the oxidation rate, we also calculated and plotted the oxidation rate of the epilayer AlAs layer in Fig. 4 as a reference. Since 1990, all work on wet oxidation of the AlGaAs compound have been conducted at temperatures higher than 400 °C, below which negligible reactions occur. There is no report on the oxidation of single-crystal AlAs at 300 °C. The rate of oxidation at 300 °C can only be extrapolated from the data of wet oxidation performed at higher temperatures.23 Furthermore, use of the extrapolated data has to be exercised with great care. In reality, the rate might be much smaller because a phase change of Al is seen around 400 °C. Nevertheless, in comparison with the extrapolated rate for single-crystal AlAs, oxidation of all (Al,As) compounds in this work proceeds several times faster. The data indicate that the sample grown with As overpressure of 2 × 10$^{-7}$ Torr exhibits the fastest oxidation rate, while the one with 5 × 10$^{-7}$ Torr oxidizes the least.

Incorporation of excess As and the multigrained polycrystalline structure of (Al,As) deposited with As overpressure at 8 × 10$^{-8}$ Torr result in a rather porous structure compared to the single-crystal AlAs layer. The porous structure enables water vapor to permeate much faster laterally, and oxidation of many grains with a grain size on the order of 1000 Å each can take place simultaneously. As a result, depending on the extent of lateral water permeation, the oxidation rate increases due to the increased surface contact between water and AlAs grains. In contrast, the compact structure of single-crystal AlAs limits the reaction to taking place only on the confined-side surface. It is understandable that lateral oxidation of a multigrained (Al,As) layer grown at 8 × 10$^{-8}$ Torr is faster than single-crystal AlAs. With increased As overpressure at 2 × 10$^{-7}$ Torr, the (Al,As) layer is amorphous or multigrained but with extremely fine grain sizes. Since grains with reduced size will take even less time to oxidize, the slightly faster rate of oxidation in 2 × 10$^{-7}$ Torr as compared to that in 8 × 10$^{-8}$ Torr is also expected, as shown in Fig. 4.

On the other hand, as the excess As incorporation increases with the As overpressure from 8 × 10$^{-8}$ to 2 × 10$^{-7}$ and 5 × 10$^{-7}$ Torr, a different oxidation mechanism needs to be taken into account. Ashby and co-workers have estimated the thermodynamic free-energy change for oxidation of AlAs at 400 °C.23,24 A modified form for (Al,As) with excess As is shown in Eq. (1). With larger excess As, thermodynamically it is less likely for (Al,As) to oxidize because oxidation of metallic arsenic with water is not a favorable chemical reaction, as shown in Eq. (2):
FIG. 5. AES profiling of an (AlAs) layer exposed to the atmosphere for 10 days. It shows that only the top 300 Å is oxidized, indicating an improved stability against moisture due to the incorporation of excess As.

\[
2(Al,As_n) + 3(1 + n)H_2O \rightarrow Al_2O_3 + nAs_2O_3 + 3(1 + n)H_2, \\
\Delta G^{(608)} = (-473 + \Delta n \times 131) \text{ kJ/mol} \quad n = 1 + \Delta n, \\
As_2O_3(\ell) + 3H_2 \rightarrow 2As + 3H_2O(\ell) \Delta G^{(608)} = -131 \text{ kJ/mol},
\]

Removal of metallic As, which has a smaller partial pressure as compared to As_2O_3, becomes critical to the wet oxidation rate is much reduced in the presence of excess As. As a result, the lateral oxidation of (Al,As) grown with As overpressure at 5 \times 10^{-7} \text{Torr}. Enhanced stability against water vapor due to excess As is also noticed in Fig. 2(a), in which the outermost layer of (Al,As) of the as-grown stack shows little sign of structural deterioration when exposed in air. To study its stability, we have grown a 3000 Å amorphous (Al,As) layer (5 \times 10^{-7} \text{Torr}) on a GaAs substrate and exposed it in atmosphere for 10 days. The AES spectrum shown in Fig. 5 indicates that only the top 300 Å contains a detectable amount of oxygen. A bare single-crystal AlAs counterpart would not survive as long.

IV. CONCLUSION

In summary, as much as 90% excess As can be incorporated in the (Ga,As) and (Al,As) compounds grown at low temperatures of about 100 °C. The excess As incorporation correlates with the growth rates, which increase with the As overpressure. In addition, large excess As content during growth may change the microstructure from polycrystalline to amorphous as well as impede the recrystallization process. Upon wet oxidation at temperatures as low as 300 °C, fine-grained or slightly amorphous (Al,As) oxidizes much faster than single-crystalline AlAs. Too much excess As, on the other hand, slows down the oxidation due to the thermodynamically unfavorable reaction between metallic As and water vapor.

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