DYNAMICS OF WET OXIDATION OF HIGH-AL-CONTENT III-V MATERIALS

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ABSTRACT

Oxidation of layers of high-Al-content III-V materials by water vapor has become the enabling process for high-efficiency vertical cavity surface emitting lasers (VCSELs) and has potential applications for reducing substrate current leakage in GaAs-on-insulator (GOI) MESFETs. Because of the established importance of wet oxidation in optoelectronic devices and its potential applications in electronic devices, it has become increasingly important to understand the mechanism of wet oxidation and how it might be expected to affect both the fabrication and subsequent operation of devices that have been made using this technique. The mechanism of wet oxidation and the consequence of this mechanism for heterostructure design and ultimate device operation are discussed here.

INTRODUCTION

The rapid oxidation of AlGaAs with high aluminum mole fraction ($>85\%$ Al) using flowing $\text{H}_2\text{O}/\text{N}_2$ gas mixtures [1] is an enabling technology for high-efficiency vertical cavity surface emitting lasers (VCSELs). Threshold currents below $10\ \mu\text{A}$ and threshold voltages only $50\ \text{meV}$ above the emitted photon energy have been obtained [2]. Wet-oxide-based VCSELs have even demonstrated greater than 50% wall-plug efficiency [3]. The wet oxidation process is also being studied for electronic applications such as gallium-arsenide-on-insulator (GOI) metal semiconductor field effect transistors (MESFETs) [4] and metal-insulator-semiconductor field effect transistors (MISFETs) [5]. If such devices are to go beyond laboratory demonstration to widespread commercial availability, it is imperative to understand the many factors that influence the extent of oxidation in a particular device and the influence of wet oxidation on relevant device properties. To first order, oxidation rates are determined by Al mole fraction. However, additional effects related to layer thickness and the nature of the adjacent layers in a device can dominate over mole-fraction effects. Alteration of adjacent unoxidized layers due to the generation of elemental As during the oxidation process can change important optical and electronic properties of a device. A mechanism for wet oxidation and the consequence of this mechanism for heterostructure design and ultimate device operation are discussed here.

EXPERIMENT

Prior to lateral wet oxidation of Al-III-V heterostructures, the Al-containing layer is exposed by etching a mesa structure to expose the layer to be oxidized. Wet oxidation is usually performed between 375 and $500\ ^\circ\text{C}$ in a tube furnace with a water-saturated nitrogen stream passing over the sample to be oxidized [6]. Oxidation begins at the exposed edges and proceeds inward. Because of the strong temperature dependence of the oxidation, a three-zone furnace is especially desirable for well-controlled oxidation profiles. Water temperature and gas flow can be varied to control the supply of H_2O reactant to the exposed layer edges. Typically, water temperatures between 75 and $95\ ^\circ\text{C}$ are employed. The oxidized length can be determined either optically (for sufficiently deep oxidations) or using scanning electron microscopy (SEM). Lateral

oxidation results presented here were obtained with 80 ± 0.5 °C water and a flow rate of 3.0 slm through a 4"-diam. 3-zone tube furnace.

The Raman spectra presented here employed planar 2- μm -thick $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$ layers on GaAs that were oxidized from the surface down rather than laterally from an exposed edge [7]. Prior to wet oxidation, a 300-Å GaAs cap was selectively removed using a citric acid/peroxide mix [5:1 of (1g citric monohydrate/1g H_2O):30% H_2O_2]. Samples were heated to the reaction temperature ($400 < T < 455$ °C) in dry nitrogen. The nitrogen flow (0.4 slm, 2-in. diam. tube) was then switched to bubble through 80 ± 1 °C water. The reaction was terminated by switching to a dry nitrogen flow. Raman spectra were measured in the $x(y',y'+z')\bar{x}$ backscattering configuration (y' and z' parallel to (110) planes) using 514.5-nm light at $< 85 \text{ W/cm}^2$.

RESULTS AND DISCUSSION

To first order, oxidation rates are determined by the Al mole fraction in the oxidizing layer. (Fig. 1). However, the time dependence of the reaction rate for samples with identical Al mole fraction has been observed to vary depending on individual processing conditions. Many workers operate in a reaction-rate-limited regime (linear time dependence) to facilitate more precise control of oxidized depth in device structures and others report diffusion-limited behavior (parabolic time dependence). Extrapolations of oxidized depth vs. time curves often fail to pass through the origin, with deviations in both directions having been reported. This complicates mechanistic interpretations. However, sufficient information is available about the temperature and composition dependence of wet oxidation rates to support the following understanding of the important dynamic characteristics that determine the time dependence.

Fig 1. Composition dependence of wet oxidation

The Deal and Grove model for oxidation [8] describes the temporal dependence of an oxidation process as the sum of a linear and a parabolic term,

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$$\frac{d^2}{k_{\text{diff}}} + \frac{d}{k_{\text{rxn}}} = t \quad (4),$$

where the linear term dominates when the oxidation rate is reaction-rate limited and the parabolic term dominates when the rate is diffusion limited. A range of dependences on time have been reported for wet oxidation of AlGaAs. The oxidation of the most preferred device composition of $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$ is generally reported to be linear from 380 to 440 °C [6], while the oxidation of AlAs has been reported to have a parabolic dependence from 370 to 450 °C [9]. Another study of AlAs has reported a linear time dependence at 356 °C, a parabolic dependence at 516 °C, and a mixed linear/parabolic dependence at intermediate temperatures [10]. Yet another study of AlAs has shown linear dependence at $T \leq 350$ °C and parabolic behavior at $T \geq 375$ °C [11].

To explain why a shift occurs between reaction-rate-limited and diffusion-limited regimes, it is necessary to have an understanding of the chemical nature of the reaction. Raman studies of partially oxidized planar AlGaAs structures always show the presence of a significant amount of elemental As that has been liberated during the oxidation process. Raman spectra of partially oxidized films are dominated by crystalline elemental As peaks at 198 and 257 cm^{-1} and a broad feature between 200 and 250 cm^{-1} peaking near 227 cm^{-1} due to amorphous As [12]. At higher reaction temperatures, significant amounts of As_2O_3 are also detected. The broad feature centered at 475 cm^{-1} is due to amorphous As_2O_3 [13]. This species, if present, is below the Raman detection level for oxidations performed at 400 and 425 °C, but it is observed at relatively constant levels throughout the oxidation at 450 °C (Fig 2). As the oxidation front advances into AlAs or AlGaAs films, one observes a relatively constant Raman signal from As and a- As_2O_3 while the intensity of the AlAs-like phonon steadily decreases as the AlGaAs is converted to oxide [14].

Fig. 2. Time evolution of Raman spectra of 2 μm layer of $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$ oxidizing at 450 °C.

Due to the strong ability of low-oxidation state Al to donate electrons to other atoms, H^+ from water can serve as the oxidizing agent for the reaction. In the process, H^+ becomes zero-valent H^0 and can serve as a reducing agent for another species. Both As_2O_3 and As are present as intermediates in the wet oxidation process. Their presence can be explained as follows.

Water has been shown to adsorb dissociatively on AlAs at 100 K under UHV conditions to produce Al-O, Al-OH, and As-H type species on the AlAs surface [15]. Since literature values for the thermodynamic quantities of these surface species are unavailable, we have chosen to employ the molecular analogs [16], leading to Eqns. 1 and 2 at a reaction temperature of 425 °C.

Under these typical wet oxidation conditions, one would expect both reactions to proceed readily. At 425°C, further reaction of As-H can decompose to give As and H_2 , as suggested by the Raman spectra (Eqn 3). It can also react with water to produce As_2O_3 , but the free energy change is quite small (Eqn. 4). Given the low-temperature UHV results, it is likely that the As_2O_3 observed results from the combination of reactions like Eqn 2 and 4. Both AsH_3 and As_2O_3 can be readily converted to elemental As (Eqns. 3 and 5).

The transition from reaction-limited (linear) to diffusion-limited (parabolic) behavior for a particular AlGaAs composition will depend on the relative temperature dependences of Eqn. 1-5 as Ga is substituted for Al. Experimental results to date suggest that Eqn. 1 becomes increasingly important for higher Al contents and higher reaction temperatures.

Porous $\gamma\text{-Al}_2\text{O}_3$	Al_2O_3 As_2O_3	AlGaAs	As_2O_3 formation and conversion to As balanced.
Porous $\gamma\text{-Al}_2\text{O}_3$	Al_2O_3 As_2O_3	AlGaAs	As_2O_3 formation faster than conversion to As.

Fig. 3. Schematic of effect of increase in rate of As_2O_3 formation relative to its reduction to As for removal to leave behind a porous As_2O_3 film.

The relationship between the rate of formation and conversion of As_2O_3 to As (Eqn. 1, 4, and 5) and the rate of loss of As from the oxidized layer to leave behind a relatively As-free, porous AlO_x matrix will determine the time dependence of the oxidation rate (Fig. 3). The existence of a thin, dense, amorphous region of a few nanometers thickness has been observed at the oxidation front by transmission electron microscopy (TEM) [17]. Behind this dense region is a less dense region of amorphous $(\text{Al,Ga})_2\text{O}_3$ that extends back to the exposed mesa edge. The time-evolution of the thickness of the dense region will determine whether linear or parabolic behavior dominates. When the removal of As is sufficiently fast to balance the rate of formation of As_2O_3 , a relatively constant thickness of dense oxide consisting of Al_2O_3 and As_2O_3 will be found near the oxidation fronts it moves deeper into the layer; this will produce the relatively constant As_2O_3 Raman intensity observed in films that were partially oxidized at 450°C . Under these conditions, a linear time dependence will appear because the diffusional contribution to the reaction rate does not increase significantly as the front moves deeper into the layer for typical reaction times. For long reaction times, the deviation from linearity will become increasingly apparent. If reaction conditions are changed to preferentially increase the formation of As_2O_3 relative to As loss, a steadily increasing thickness of the dense, As_2O_3 -containing layer will form and the diffusion-limited parabolic time dependence will become dominant.

The transition from linear to parabolic behavior should be favored by conditions that increase the rate of Eqn. 1 relative to that of Eqn. 5. The greater amounts of As_2O_3 seen in the Raman spectra for oxidation at 450°C vs. 425°C and the relatively constant As signal under the two conditions (Fig. 4) suggest that higher temperatures preferentially enhance Eqn. 1 vs. Eqn. 5 for $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$. The transition from predominantly linear to predominantly parabolic with increasing temperature has been previously been reported for AlAs [9-11].

Figure 4. Temperature dependence of Raman spectra a $2\ \mu\text{m}$ layer of $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$.

Replacing Al with Ga in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ will affect the favorability of Eqn. 1 and 2, since ΔG^{698} for Eqns. 1 and 2 for GaAs are respectively +10 kJ/mol and +32 kJ/mol, and may thereby retard wet oxidation as Ga content increases. In contrast, Eqns. 3-5 do not explicitly include either Al or Ga. Consequently, the reduction of As_2O_3 to As should be relatively independent of the Al/Ga ratio. One would, therefore, expect that there might be a change in the time dependence of the total wet oxidation reaction when the rates for Eqn. 1-2 are increased or decreased faster than the rates for Eqn. 3-5. For example, the slower rate of Eqn. 1 for higher Ga-content AlGaAs could produce linear behavior under some conditions that produce parabolic behavior for higher Al-content material. This has, in fact, been observed during oxidation of a structure containing 45-nm layers of both 98% and 94% AlGaAs oxidized at 440 °C (Fig. 5). In contrast, the slower rates of formation of As_2O_3 from both compositions at 400 °C permits the reduction and loss of As to keep pace with the oxidation process, and linear behavior is observed for both compositions. The transition from reaction-limited to diffusion limited for these samples lies between 0.2 and 1.3 $\mu\text{m}/\text{min}$ oxidation rates for the H_2O temperature and flow rate employed (3.0 slm in a 4"-diam. furnace, 80 ± 0.5 °C H_2O).

Fig. 5. Temperature dependence of the oxidation of 450-Å layers of $\text{Al}_{0.09}\text{Ga}_{0.02}\text{As}$ and $\text{Al}_{0.94}\text{Ga}_{0.06}\text{As}$ within the same sample.

Two additional device design characteristics can have a profound effect on the oxidation rate of a specific layer of AlGaAs as its Al mole fraction. These are the thickness of the layer to be oxidized and the close proximity of a more rapidly oxidizing layer. Under some conditions, both can prove more important than Al mole fraction in determining the relative oxidation rates of different layers in the same structure.

The dependence of oxidation rate on layer thickness is illustrated in Fig. 6 for $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$ and $\text{Al}_{0.94}\text{Ga}_{0.06}\text{As}$ oxidized at 425 °C under conditions that yield a linear time dependence for thick layers. For layers thicker than 75 nm, there is very little dependence on layer thickness. In contrast, there is a rapid drop-off of the oxidation rate as the layer thickness decreases below 75 nm. The decrease in oxidation rate as the layers become thinner is sufficient to invert the relative oxidation rates for higher and lower Al-mole-fraction materials when a relatively thick layer of lower Al content is compared to a thin layer of higher Al content. The

mechanism of this decrease in rate has not been definitely identified. It has been suggested that the decrease in very narrow channels is related to the vapor transport coefficient at the gas-oxide interface [18]. An alternative model has been proposed based on the surface energy of the curvature observed at the oxide tip [19]. The latter model assumes identical contact angles at the top and bottom interfaces; however, a pronounced asymmetry is generally observed, with the contact angle generally being greater at the top interface [17]. In addition, the activation energies so derived are unusually large compared to other literature values. Hence, the gas transport model, though not quantitatively evaluated, appears preferable at this time.

Fig. 6. Thickness dependence of the wet oxidation rate of AlGaAs.

Fig 7. Cross sectional SEM of a partially oxidized (400 °C, 90 min) sample of alternating 450 Å layers of $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$ and $\text{Al}_{0.94}\text{Ga}_{0.06}\text{As}$ separated by GaAs barriers with thicknesses

varying from 120 Å near the substrate to 7 Å near the surface. Dark regions are oxide, light regions are unoxidized.

The oxidation rate of a lower-Al-content layer can be appreciably increased when it is in close proximity to a faster-oxidizing layer. The effect of such close-proximity faster-oxidizing layers [14] is shown in Fig. 7 for 45-nm layers of undoped $\text{Al}_{0.94}\text{Ga}_{0.06}\text{As}$ separated from 45-nm layers of undoped $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$ by thin layers of undoped GaAs (0.7, 1.5, 3, 6, 9, and 12 nm) oxidized at 400 °C for 90 min. Although no enhancement of the reaction of the $\text{Al}_{0.94}\text{Ga}_{0.06}\text{As}$ layer is observed after this reaction time with 9 and 12 nm barriers, there are progressively greater enhancements of the oxidation of the $\text{Al}_{0.94}\text{Ga}_{0.06}\text{As}$ layers that are separated for the already oxidized $\text{Al}_{0.98}\text{Ga}_{0.02}\text{As}$ by 6, 3, 1.5, and 0.7 nm GaAs layers. All $\text{Al}_{0.94}\text{Ga}_{0.06}\text{As}$ layers exhibit rate enhancements at longer reaction times. The oxidation of the $\text{Al}_{0.94}\text{Ga}_{0.06}\text{As}$ layers can be modeled as the sum of the unenhanced rate plus a contribution derived from the diffusion of something into the layer from the already oxidized adjacent layers [20]. The close-proximity enhancement is not significant only in the presence of barriers. Structures with graded compositions result in profiles that show appreciably deeper oxidation than expected from compositional effects alone [21]. This proximity enhancement is especially important since compositional grading is used to fabricate tapered oxide current apertures for optimum VCSEL performance [6,22] and to minimize stresses in the oxidized devices.

Because elemental As is a major product in wet oxidation, it is necessary to consider its influence on important device properties. The presence of large amounts of elemental As at the oxide/semiconductor interface will result in the injection of As interstitials, As_i , into the adjacent unoxidized semiconductor. It will also lead to the generation of additional types of point defects [23]:

Reaction of As with the surface GaAs can lead to the generation of Ga vacancies, V_{Ga} . Interstitial As can diffuse into the material and generate Ga interstitials by displacement to form As antisite defects, As_{Ga} . Antisite As_{Ga} can also form by reaction with Ga vacancies. The increased concentrations of these point defects can be quite deleterious for devices made using wet oxidation.

The most gross evidence for the large quantities of point defects that are produced during wet oxidation is the appearance of As precipitates quite remote from the original oxidation. Such As precipitates have been observed widely dispersed through the adjoining layers and even segregated at the GaAs surface of a wet-oxidized sample [24]. Precipitates have also been reported in the InGaAs channel of a GOI MESFET that had a 10 nm $\text{Al}_{0.25}\text{Ga}_{0.75}\text{As}$ layer interposed between the channel and the oxidized layer [25].

The photoluminescence from quantum wells that are near wet-oxidized regions exhibits changes that are typically seen in the presence of excessive numbers of point defects. The photoluminescence is decreased for quantum wells (QWs) situated 20 and 40 nm from oxidized

AlAs [26]. The time and temperature dependence of this degradation is consistent with the presence of oxidation-generated point defects that have diffusivity comparable to As in GaAs. Post-oxidation hydrogen treatment partially restores the photoluminescence intensity from GaAs quantum wells separated from the oxidized layer by 25-nm AlGaAs spacers. [27] While oxidation reduces the PL intensity to 6% of the original intensity, post-oxidation H-annealing restores the intensity to 67% of its original level. Such PL restoration is to be expected from hydrogen passivation of As-associated deep levels, e.g., As_{Ga}, that were formed during oxidation by indiffusion of excess As.

Intermixing at quantum well interfaces by heating is seen to be greater near oxidized AlGaAs regions than near unoxidized regions of the same device. However, even the QW emission from the regions that are not immediately above or below oxidized material exhibit a blue-shift. Such behavior is expected if there is increased mobility of column III atoms in the presence of excess As [28]

In addition to the observation of As precipitates in the InGaAs channel of a GOI MESFET, a deterioration of both the mobility and the free carrier concentration is obtained following oxidation. This deterioration was minimized by decreasing oxidation temperature and/or oxidation time [25]. Both process alterations would reduce the diffusion of As-derived defects into the device, thereby minimizing device degradation.

SUMMARY

Wet oxidation of high-Al-content AlGaAs produces both elemental As and As₂O₃ as important products that can remain near the reaction front. The thickness of the As₂O₃-containing region at the oxide/semiconductor interface will determine the time-dependence of the process. A shift from reaction-rate-limited (linear) to diffusion-limited (parabolic) time dependence is favored by increasing temperature or increasing Al mole fraction. The elemental As that is produced in the reaction acts as a source of defects that can affect important chemical and electrical properties of devices. Free As is an inherent part of the wet oxidation process that cannot be totally avoided so applications of these oxides must either not depend strongly on concentrations of point defects in the material or attempts must be made to design around the endemic As problem.

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