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Wet oxidation of AlAs grown by molecular beam epitaxy

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A 1 μm thick n -type GaAs layer with Si doping density of $1 \times 10^{17}/\text{cm}^3$ and a 1000 \AA thick undoped AlAs layer was subsequently grown by molecular beam epitaxy on the n^+ GaAs substrate. To perform selective oxidation of the AlAs layer, the wafer was oxidized in N_2 bubbled H_2O vapor ambient at 370 $^\circ\text{C}$, 400 $^\circ\text{C}$, and 430 $^\circ\text{C}$, each for 3 hours. It is found that the defect-free selective oxidation of AlAs is very critically dependent on oxidation temperature. Both GaAs and AlAs layers were oxidized at the oxidation temperature of 430 $^\circ\text{C}$, and some defects were found by scanning electron microscopy (SEM) on the surface oxidized at 370 $^\circ\text{C}$. Oxidation was, however, stopped at the AlAs/GaAs interface at the oxidation temperature of 400 $^\circ\text{C}$, and no defects were found on the surface. By the slow ramp rate (5 mV/sec) high frequency (100 kHz) C - V measurement, the fixed charge density was estimated to be about $1 \times 10^{11}/\text{cm}^2$ for the metal-oxide-semiconductor (MOS) capacitor fabricated from the oxidized sample at 400 $^\circ\text{C}$. © 1994 American Institute of Physics.

There have been extensive investigations on insulator/GaAs structures by thermal oxidation, anodic oxidation, chemical vapor deposition (CVD), sputtering, etc.¹⁻⁴ However, the electric and chemical characteristics of the films and the interface were much worse than those of thermally grown SiO_2/Si structure. It is known that there would be Al clusters and grains in grown or deposited Al_2O_3 ,^{5,6} which make the film unsuitable as the gate material of GaAs metal-oxide-semiconductor field effect transistors (MOSFETs).

Since the first paper on thermal oxidation of GaAs reported,¹ there has been a number of papers on the thermal oxidation of GaAs. During thermal oxidation the reaction between GaAs and O_2 yields Ga_2O_3 and elemental As which make the interface unstable.^{7,8} The thermally oxidized film of AlGaAs also has elemental As, which affects the electric properties, in the film and at the interface.⁹

Dallesasse *et al.*¹⁰ reported that hydrolization oxidation of $\text{Al}_x\text{Ga}_{1-x}\text{As}$ -AlAs-GaAs, instead of deposited Si_3N_4 , was useful for passivation and the current blocking layer of laser diodes. In this work we further turned our attention for the oxidation to the possible MOS applications beyond the work done by them. We systematically characterized the thermal oxidation of AlAs/GaAs by scanning electron microscopy (SEM), scanning Auger microspectroscopy (SAM, Perkin-Elmer model 650), and C - V analysis. A 1 μm thick n -type GaAs layer with Si doping density of $1 \times 10^{17}/\text{cm}^3$ and a 1000 \AA thick undoped AlAs layer were grown by molecular beam epitaxy (Riber-45) on the n^+ GaAs substrate. They were then thermally oxidized in a N_2 bubbled H_2O vapor (95 $^\circ\text{C}$) ambient. The GaAs substrates used for these experiments were (100) oriented n^+ GaAs substrates with doping density of $2 \times 10^{18}/\text{cm}^3$. Oxidation was performed at temperatures of 370 $^\circ\text{C}$, 400 $^\circ\text{C}$, and 430 $^\circ\text{C}$, each for 3 hours.

In this letter, we present the temperature effects in formation of Al_2O_3 from wet oxidation of the AlAs/GaAs layer and the usability of the film as a GaAs MOS dielectric.

The SEM photographs of the oxidized AlAs surface are shown in Fig. 1. No surface defects were found for the sample oxidized at 400 $^\circ\text{C}$. But, many hemispherical type surface defects with a diameter of 0.3–0.6 μm were gener-

ated for the sample oxidized at 370 $^\circ\text{C}$ due to the possible incomplete substitution of arsenic by oxygen, which could cause high leakage current and dielectric loss. Some parts of the AlAs layer could thus contain less oxygen and form the

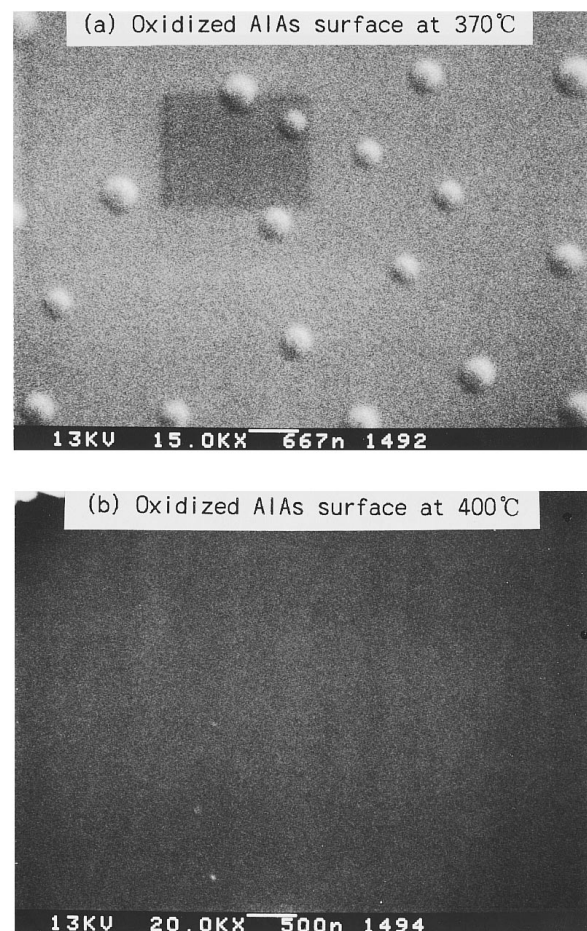


FIG. 1. SEM photographs of the thermally oxidized AlAs/GaAs surface in the N_2 bubbled H_2O vapor (95 $^\circ\text{C}$) ambient (a) at 370 $^\circ\text{C}$ and (b) at 400 $^\circ\text{C}$, each for 3 hours. The AlAs/GaAs surface oxidized at 400 $^\circ\text{C}$ is defect free, while there are some hemispherical type defects with a diameter of 0.3–0.6 μm for the sample oxidized at 370 $^\circ\text{C}$.

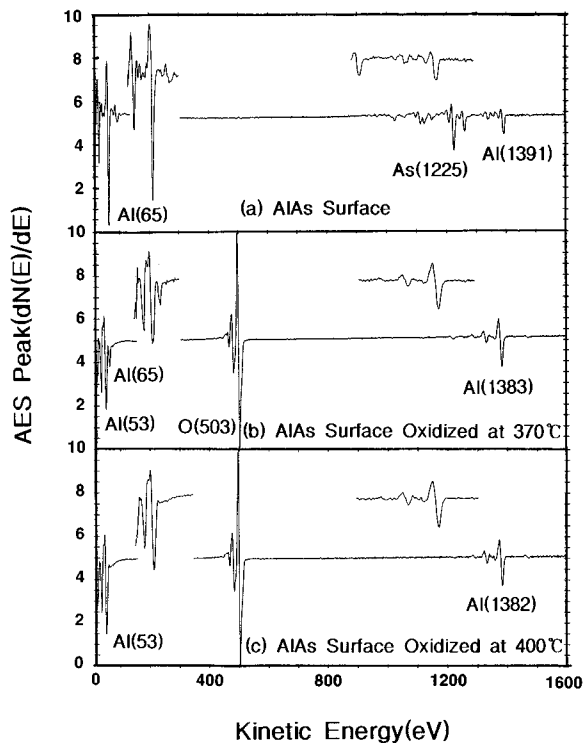


FIG. 2. AES peaks with the scanning size of $30 \mu\text{m}^2$ (a) on the as-grown AlAs/GaAs surface, (b) on the AlAs/GaAs surface oxidized at 370°C , and (c) on the surface oxidized at 400°C , each for 3 hours. The Al(*KLL*, *LMM*) peaks (a) (68 and 1384 eV) in the as-grown sample are shifted to the Al peaks (c) (35, 51, and 1392 eV) of Al_2O_3 .

hemispherical defects shown in Fig. 1(a), because the density of Al_2O_3 is higher than that of AlAs. The oxidation temperature of 370°C is, therefore, not high enough to form a clean oxidized AlAs surface.

The AES peaks after Ar ion presputter for the as-grown AlAs surface, and the AlAs surfaces oxidized at 370°C and 400°C are shown in Fig. 2. The scanning size was $30 \mu\text{m}^2$ and wide enough to cover the whole area including the defects. A small As peak is present near the kinetic energy of 1225 eV in the oxidized AlAs/GaAs [Fig. 2(b)] surface at 370°C . The As (*LMM*) peak in the as-grown surface in Fig. 1(a) has disappeared in the AlAs surface oxidized at 400°C [Fig. 2(c)], because most of the As has diffused out. It is known that Al(*KLL*, *LMM*) Auger electron kinetic energy and peak shapes vary with compounds. The Al(*LMM*) peak near 65 eV in the as-grown sample disappeared in the oxidized AlAs/GaAs surface at 400°C [Fig. 2(c)], but not in the oxidized sample at 370°C [Fig. 2(b)]. This means that the AlAs layer is not completely converted to Al_2O_3 at the oxidation temperature of 370°C . The Al(*KLL*, *LMM*) peaks near the kinetic energy of 35, 51, and 1391 eV in the oxidized AlAs/GaAs at 400°C [Fig. 2(c)] are the same as the peaks of standard Al_2O_3 .¹¹ It is, therefore, possible to conclude that the AlAs layer has been converted to the Al_2O_3 layer by the oxidation.

The AES depth profile of AlAs/GaAs layers oxidized at 430°C and 400°C is shown in Fig. 3, where the sensitivity factors of Al and O were calibrated to that of Al_2O_3 , and the

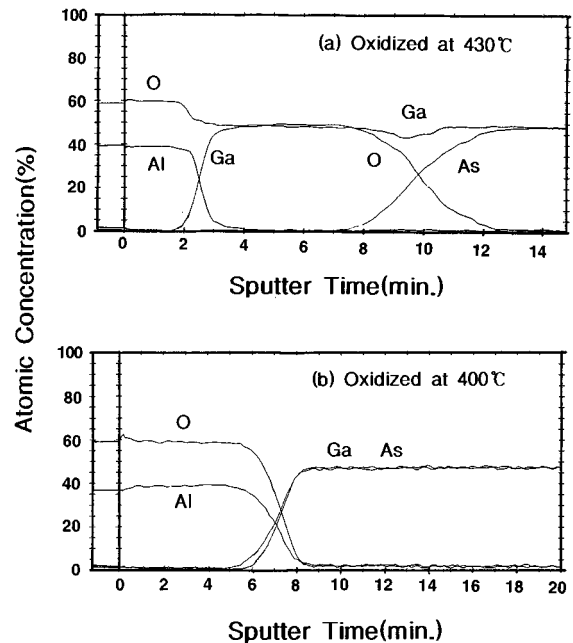


FIG. 3. AES depth profiles of AlAs/GaAs oxidized (a) at 430°C and (b) at 400°C , each for 3 hours. Both AlAs and GaAs layers were oxidized at 430°C . Oxidation was selectively stopped at AlAs/GaAs interface at the oxidation temperature of 400°C .

sensitivity factors of Ga and As were calibrated to that of GaAs. Both GaAs and AlAs were oxidized at the oxidation temperature of 430°C [Fig. 3(a)]. This indicates the oxidation does not stop at the AlAs/GaAs interface and proceeds further into the GaAs layer at the temperature of 430°C . The distributions of Al and O in the sample oxidized at 400°C [Fig. 3(b)] are almost uniform from the surface to the Al_2O_3 /GaAs interface. The oxidation does stop at the AlAs/GaAs interface, and does not proceed into the GaAs epitaxial layer. When the interface of thermally oxidized AlAs/

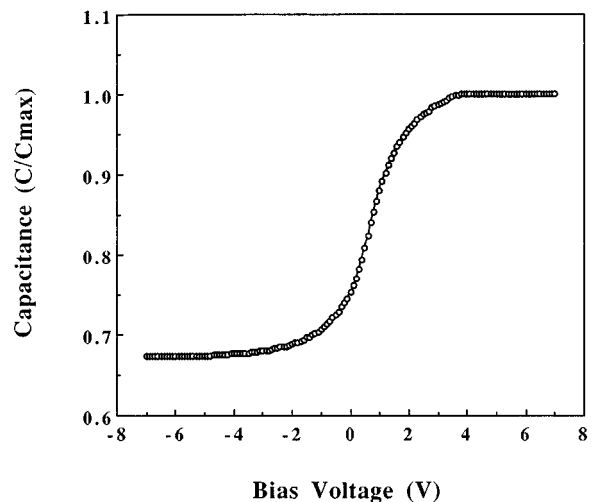


FIG. 4. The slow ramping rate (5 mV/sec) *C-V* characteristics for an MOS capacitor fabricated from the sample oxidized at 400°C for 3 hours. The curve is shifted to the right compared to the ideal *C-V* curve.

GaAs becomes sharp, this oxide would be useful to make a high quality GaAs MOS structure.

To control selective oxidation of AlAs and to form a good interface, therefore, the oxidation temperature should be adjusted to be about 400 °C. The technique employed in this experiment could be a better way of forming a good quality Al₂O₃ layer as a thin dielectric, compared to other techniques such as CVD or sputtering, which do not form high quality dielectrics.

Figure 4 shows the *C-V* characteristics of the oxidized AlAs at 400 °C. Al and In were thermally evaporated to form the gate and backside electrodes, respectively. The native oxide in the backside was etched just prior to the backside metalization. The *C-V* characteristics were measured in high frequency (100 kHz) and in negative-to-positive gate bias varying with a slow enough ramping rate (5 mV/sec), to neglect the interface trapped charge, injection, and ion-drift effects. Low negative fixed charge densities of about $1 \times 10^{11}/\text{cm}^2$ were calculated by the conventional Terman¹² method from this curve. The fixed charge density is so small that the oxidized AlAs layer converted to Al₂O₃ layer is suitable for possible GaAs MOSFETs.

In conclusion, the as-grown AlAs/GaAs layer was selectively converted to Al₂O₃/GaAs at the oxidation temperature

of 400 °C in N₂ bubbled H₂O vapor for 3 hours. The oxidation temperature is very critical to form a defect-free surface and to stop the oxidation at the AlAs/GaAs interface. The *C-V* characteristics with low interface state density show that the thermally oxidized AlAs/GaAs layer could be applicable to GaAs MOSFETs.

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