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Thermal oxidation of InAlP

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Producing insulating layers on III–V semiconductors is crucial for a number of important device applications. Al-containing thermal oxides on AlGaAs and InAlAs have been found to possess good insulating characteristics and oxides on InAlP have recently been shown to be even more promising. This paper presents data on the thermal oxidation at 500°C in moist nitrogen (95°C) of MBE-grown InAlP layers ($In_{0.525}Al_{0.475}P$ and $In_{0.494}Al_{0.506}P$) lattice matched to GaAs. The oxides (20–300 nm thick) have been characterized by Auger electron spectroscopy, X-ray photoelectron spectroscopy, Rutherford backscattering spectroscopy, transmission and scanning electron microscopy. Oxides are amorphous and appear to be a mixture of indium phosphates and aluminum oxide. The oxidation kinetics are parabolic, and the InAlP layer with the higher Al content oxidizes slightly faster. Electrical measurements performed on metal-insulator-semiconductor (MIS) structures indicate that the oxide has good electrical properties, exhibiting low current densities (up to 14 V), making the oxide films potentially useful for some device applications.

Keywords: III–V semiconductors, InAlP, thermal oxidation, surface-analytical techniques

1. INTRODUCTION

Producing chemically and electrically stable surfaces on III–V semiconductors is crucial for a number of important device applications. Passivation layers can be produced by deposition of nitride or oxide or created by a variety of oxidation processes including thermal oxidation. Thermal oxidation data for AlGaAs and InAlAs in GaAs- and InP-based heterostructure devices have been reported [1–7], and the Al-containing oxides have often been found to possess good insulating characteristics. Recently, Al-containing thermal oxides on InAlP have been shown to be even more promising [8–10].

This paper presents data on the thermal oxidation of InAlP at 500°C in moist nitrogen (95°C). The composition and nature of the oxide have been determined by Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), Rutherford backscattering spectroscopy, transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The oxides are found to exhibit good electrical properties.

2. EXPERIMENTAL

Two ~ 1µm thick InAlP layers ($In_{0.525}Al_{0.475}P$ and $In_{0.494}Al_{0.506}P$), doped with 1×10^{18} cm^{-3} Si, grown by molecular beam epitaxy (MBE) were lattice matched to a 50 nm-thick *n*-GaAs buffer layer (doped with 1×10^{18} cm^{-3} Si) on a Si-doped GaAs substrate. Oxidations were performed in a Lindberg/Blue furnace at 500°C in moist nitrogen (N_2 bubbled through H_2O at 95°C with gas transfer through heated tubes to the oxidation furnace).

After oxidation samples were analyzed by AES (PHI 650 system); XPS (PHI 5500 with a monochromated AlK$_\alpha$ source; TEM (Philips EM 430T) operating at 250 keV; SEM (Hitachi S-4700 FESEM). Electrical measurements were performed on metal–insulator–semiconductor (MIS) structures.

3. RESULTS AND DISCUSSION

3.1 Oxide growth and oxide composition

Oxidations were performed at 500°C in moist nitrogen for periods of time ranging from 6 minutes to 4 hours. As seen in Figure 1, the oxidation kinetics are parabolic (after a brief incubation period), and the InAlP layer with the higher Al content oxidizes slightly faster. Oxides ranging in thickness from ~ 20 nm to ~ 300 nm have been characterized by Auger, XPS, and TEM.

TEM micrographs of the oxide formed after 1 hour on $In_{0.494}Al_{0.506}P$ are shown in Figure 2. The lower magnification image [Figure 2(a)] illustrates a uniformly-thick oxide on InAlP on GaAs. The higher magnification image

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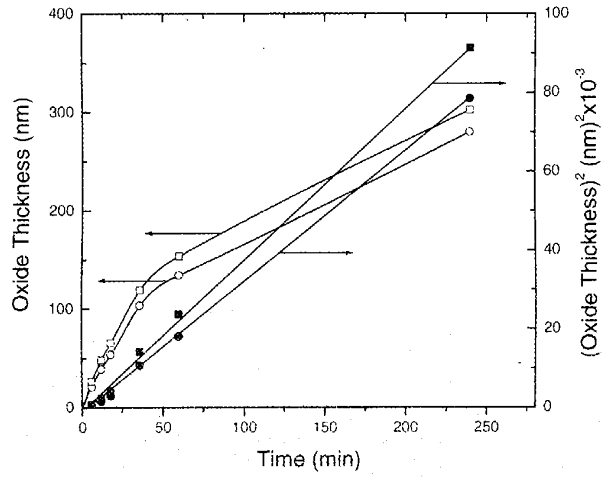

Figure 1 Oxide thickness and (oxide thickess)^2 vs. time, for $In_{0.525}Al_{0.475}P$ (○,●) and $In_{0.494}Al_{0.506}P$ (□,■) oxidized at 500°C in moist nitrogen. Oxide thickness determined by TEM or SEM measurements of cross-sections. Parabolic kinetics are observed after a brief incubation period.

[Figure 2(b)] shows the uniform oxide and the presence of particles near the oxide/InAlP interface which have been attributed to unoxidized indium [9,10]. The bulk of the oxide is amorphous as deduced for both electron diffraction and X-ray diffraction measurements.

An Auger profile of the oxide of Figure 2 is shown in Figure 3. The Auger sensitivity factors for P, Al and In in the oxide are found to be quite different from those in the substrate. Therefore, the sensitivity factors in the oxide have been based on the oxide composition as determined by RBS. RBS analysis of oxides formed after 6, 18 and 36 minutes (whose Auger profiles have similar characteristics to those in Figure 3), gives an In:P:Al:O ratio of 0.08:0.17:0.08:0.67. The In, P and Al ratios in the oxide are the same as in the substrate and the oxygen is ~ 67%. Therefore, as seen in Figure 3, P is the major component in the oxide, the Al level is fairly constant and In appears to be depleted in the outer part of the oxide and increases at the interface.

An indication of the chemical composition of the oxide can be obtained from XPS measurements of the oxide formed after 6 minutes of oxidation. This oxide, as seen in the TEM micrograph of Figure 4, is quite uniform in thickness (~ 26 nm), and the darker regions (< 10 nm diameter) which appear to be crystalline are likely, as noted above, to be small particles of unoxidized indium. O 1*s*, P 2*p* and In 3*d* XPS data are shown in Figure 5. Curve fitting was carried out using the binding energies of Hollinger *et al.* [11,12], Faur *et al.* [13], and an In_2O_3 standard for the various relevant species. Curve-fitting of the O 1*s* signal [Figure 5 (a)] suggests the possible presence of several species. These include small components of the oxide species In_2O_3 and P_2O_5 and the phosphate and polyphosphate species $InPO_4$, $In(PO_3)_3$ and $In(PO_y)_x$ [14]. The peak positions for P_2O_5 and $In(PO_y)_x$ are practically coincidental and thus it is questionable whether both species are present in the layer. Similarly, the peaks for $InPO_4$ and $In(PO_3)_3$ coincide, and thus only one of these species may be present in the layer. The yield corresponding to the latter two species is significantly greater than the yields from In_2O_3, P_2O_5 and $In(PO_y)_x$ indicating that one, or both of these species dominate.

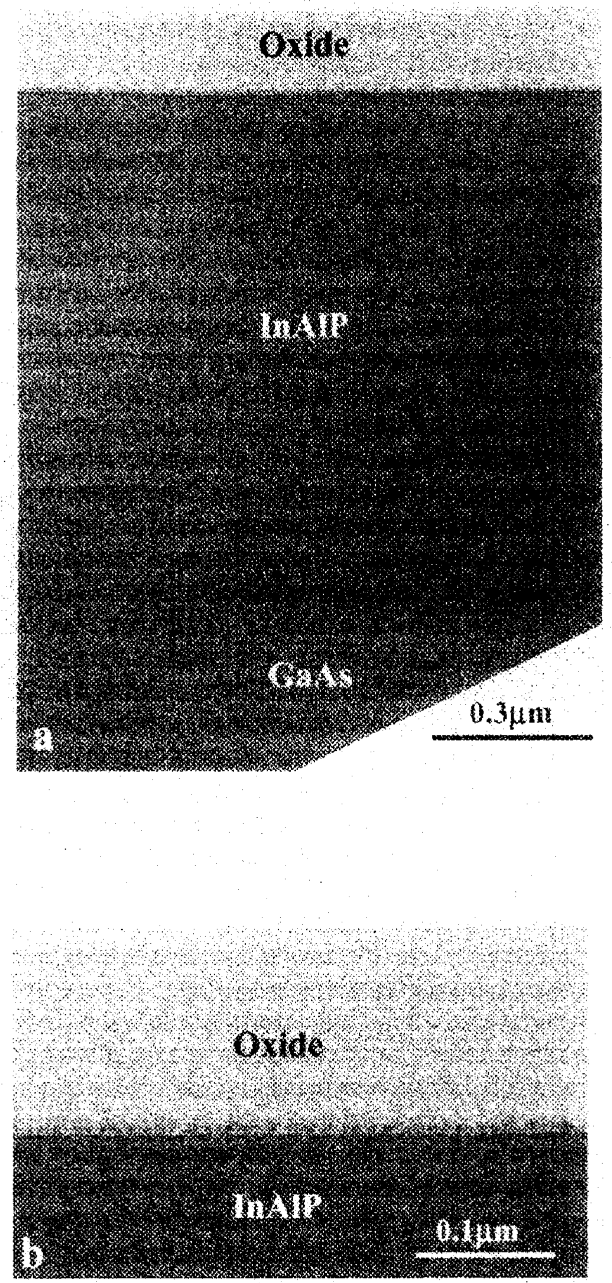

Figure 2 TEM micrographs of ~ 154 nm-thick oxide formed on $In_{0.494}Al_{0.506}P$ after 1h at 500°C in moist nitrogen. Cross-section prepared by ion milling.

Curve-fitting for the P 2*p* signal [Figure 5(b)] is consistent with the data for the O 1*s* peak. Thus, peaks are included for both $InPO_4$ and $In(PO_3)_3$, and one of, or both P_2O_5 and $In(PO_y)_x$, since the individual peaks for the two species occur at similar energies. The relative yields again indicate the dominance of $In(PO_3)_3$ and $InPO_4$ over other phosphorus-containing species. The curve-fitting of the In 3*d* peaks [Figure 5(c)] supports the presence of one of, or both, $InPO_4$ and $In(PO_3)_3$, species, which cannot be separated. These species are the main indium-containing constituents of the layer. The contribution from In_2O_3 and

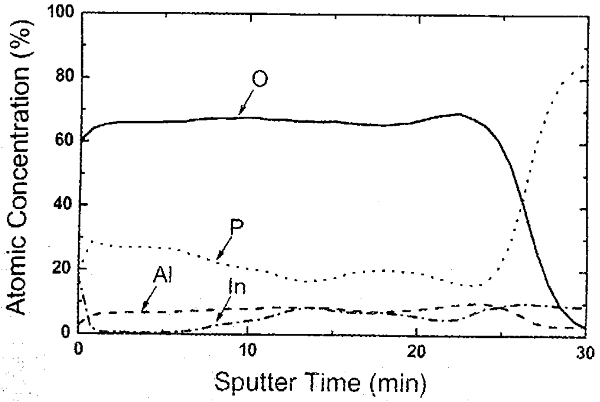

Figure 3 Auger electron spectroscopy (AES) profile of ~ 154 nm-thick oxide formed on $In_{0.494}Al_{0.506}P$ after 1h at 500°C in moist nitrogen (TEM micrographs of the oxide shown in Figure 2). Auger sensitivity factors in the oxide are based on RBS analysis of the oxide composition; the profiles have been truncated just after the oxide/InAlP interface. Sputtering was by 1 keV argon ions.

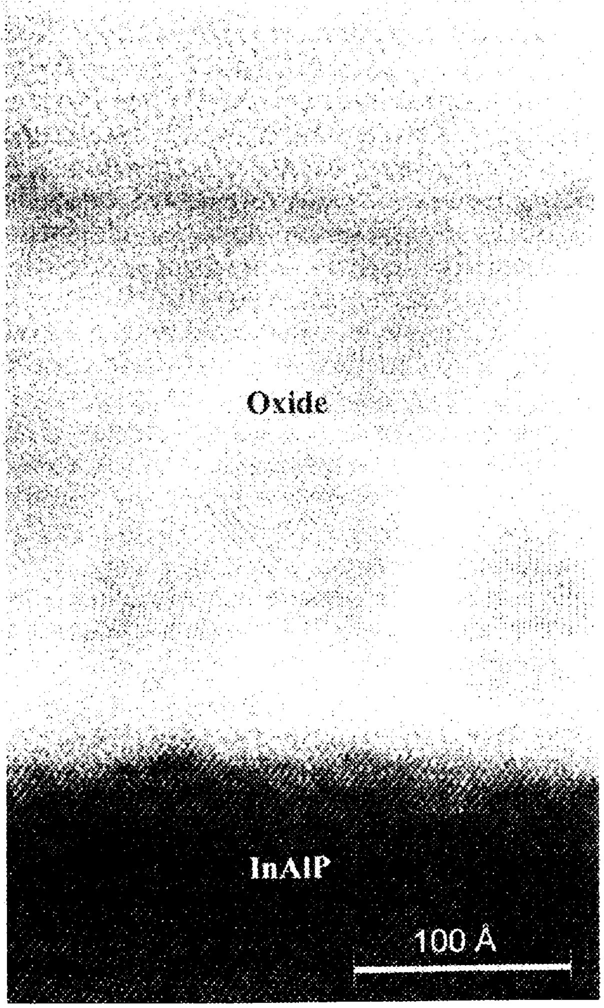

Figure 4 TEM micrograph of ~26 nm-thick oxide formed on $In_{0.494}Al_{0.506}P$ after 6 min at 500°C in moist nitrogen. Cross-section prepared by ion milling.

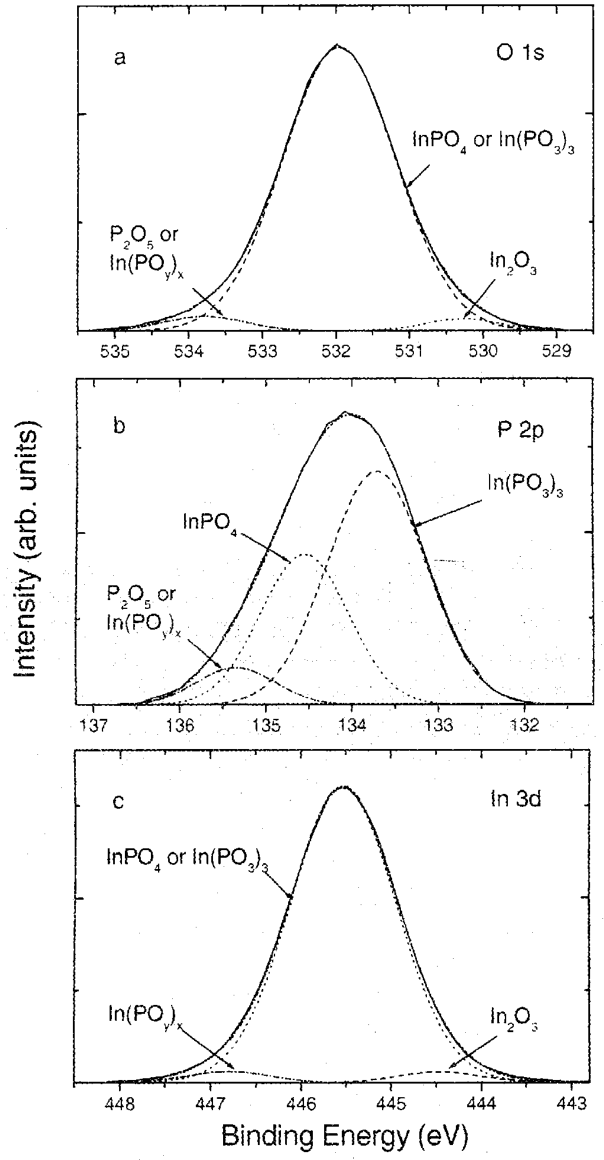

Figure 5 Curve-fitted X-ray photoelectron spectra (XPS) of ~ 26 nm-thick oxide formed on $In_{0.494}Al_{0.506}P$ after 6 min at 500°C in moist nitrogen. (a) O 1*s*; (b) P 2*p*; (c) In 3*d*. (TEM micrograph of the oxide shown in Figure 4).

$In(PO_y)_x$ is small. Examination of the Al 2*p* XPS peak (not shown) confirms the presence of Al_2O_3 (and not $AlPO_4$) in the oxide. The main components of the oxide formed on InAlP from XPS and Auger data are therefore $In(PO_3)_3$, $InPO_4$ and Al_2O_3. Only small amounts of In_2O_3 and P_2O_5 are present.

3.2 Electrical measurements

Electrical measurements were performed on metal-insulator-semiconductor (MIS) structures, with aluminum gates evaporated through a shadow mask and annealed at 450°C for 5 minutes in forming gas. Figure 6 shows the variation of current density as a function of gate bias for a ~ 48 nm -thick, oxide film formed on $In_{0.494}Al_{0.506}P$ after 12 minutes of oxidation at 500°C in moist nitrogen. Linear characteristics could be indicative of ohmic conduction, either electronic or ionic [15]. The breakdown voltage for

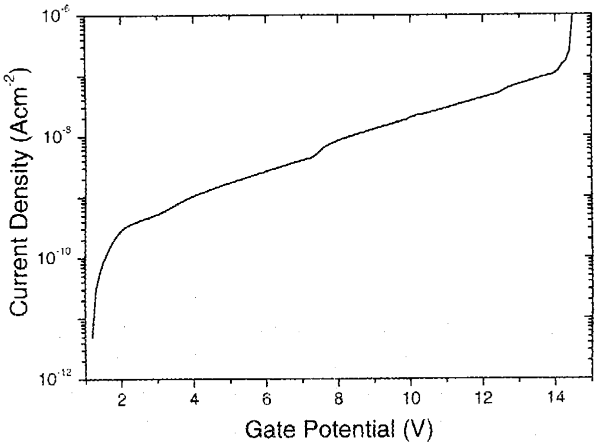

Figure 6 Variation of current density with gate bias voltage for a ~ 48 nm-thick oxide film formed on $In_{0.494}Al_{0.506}P$ after 12 min at 500°C in moist nitrogen.

this film was 14.2 V, corresponding to a breakdown field of 3.0 MVcm^{-1}. Thus the films should be useful as insulators in some device applications. Since the leakage current was small, the quasi-static capacitance could be obtained. Using a ramp voltage of 0.1 Vs^{-1} scanned from –ve to +ve potentials, the accumulation capacitance was measured and this resulted in a dielectric constant of 8.1 ± 0.2.

4. CONCLUDING REMARKS

Thermal oxidation of InAlP layers ($In_{0.525}Al_{0.475}P$ and $In_{0.494}Al_{0.506}P$) at 500°C in moist nitrogen produces amorphous, insulating oxide which is a mixture of indium phosphates and aluminum oxide. The oxidation kinetics are parabolic, and the InAlP layer with the higher Al content oxidizes slightly faster. Electrical measurements indicate that the oxide has good electrical properties, making the oxide films potentially useful for some device applications.

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