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Growth by MOCVD of In(Ga)AlN alloys, and a study of gallium contamination in these layers under nitrogen and hydrogen carrier gas

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ABSTRACT

We have studied the growth of In(Ga)AlN under nitrogen and hydrogen, changing the precursor flows, temperature and growth rate to examine the effect of these parameters on the indium incorporation and layer morphology. Under hydrogen carrier gas, we successfully incorporated indium into the layers by reducing the temperature below 620 °C. We have also studied the gallium contamination in In(Ga)AlN layers, finding a linear correlation between tri-methyl indium (TMIn) flow and tri-methyl gallium (TMGa) effective flow coming from the pollution source, thought to be due to desorption from the chamber. By performing a chamber cleaning process between the GaN pseudo-substrate and the InAlN layer, we have both eliminated the gallium contamination and increased the indium content in our layers, reaching indium levels of up to 11% under hydrogen. Finally, we achieved a sheet resistance of 250 Ω/sq on wafers with a clean between the GaN and the InAlN layers, showing the potential for using this technique to produce high performance devices.

1. Introduction

As a replacement for AlGaN barrier layers in high electron mobility transistors (HEMTs), InAlN and In(Ga)AlN layers are gaining interest due to lattice matching on GaN while containing 18% indium [1] and the benefit of a stronger spontaneous polarization [2]. This results in higher sheet carrier density and therefore lower electrical resistance in the 2-dimensional electron gas (2DEG) [2]. Thus in the fabrication of InAlN ternary and In(Ga)AlN quaternary alloys by metal organic vapor phase epitaxy (MOVPE), the accurate control of the indium content in the material has a strong impact on its physical and electrical properties.

We have demonstrated in a former study [3] the benefits of hydrogen carrier gas compared to nitrogen on the surface morphology of low temperature GaN. At temperatures below 940 °C, V-defects appear where edge and mixed dislocation lines meet the surface. We showed that growing under hydrogen allows us to grow layers with the same morphology as layers under nitrogen carrier gas which have a growth temperature 100 °C higher. For instance V-defect diameters at 840 °C under nitrogen are the same as those at 740 °C under hydrogen. Moreover the hydrogen carrier gas also improved the overall topology of the surface.

These previous results motivated the comparison of InAlN layers under nitrogen carrier gas and under hydrogen carrier gas, even though hydrogen is known to make it harder to incorporate indium [4]. An additional difficulty of growth of InAlN layers is the gallium pollution of these layers, coming from the MOVPE growth chamber [5–8]. This study will investigate further this effect, how it is affected by different growth conditions, including carrier gas [4], and how the chamber cleaning by chlorine affects this pollution. As the gallium is a contaminant, the layers will be globally referred to as In(Ga)AlN.

For characterization of InAlN layers, X-ray diffraction is commonly used to determine their composition [9]. However, growth on silicon requires AlGaN buffer layers which have broad peaks in (0002) XRD profiles. These can hide other peaks which have similar peak diffraction angles such as those corresponding to In(Ga)AlN layers, which have a low intensity, especially for layers of only 10 s of nm thick. X-ray Photoelectron Spectroscopy (XPS) is a direct chemical characterization which avoids these problems, and which makes it a very good technique for measuring In(Ga)AlN layers. This direct characterization simplifies interpretation compared to X-ray diffraction (XRD) or photoluminescence in which the determination of the composition requires indirect calculations.

2. Experimental details

We have grown 22 In(Ga)AlN samples by MOVPE (metal organic vapor phase epitaxy) on 200 mm (111) silicon wafers using a fully automated AIXTRON Crius R200 with a close coupled showerhead system. The nitride structures used for this study have a simple buffer

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Table 1
Summary of In(Ga)AlN growth conditions.

Carrier gas	Temperature (°C)	TMIn/(TMIn+TMAl) gas (%)	TMIn/(TMIn+TMAl) alloy (%)	Thickness (nm)	Growth rate (nm/min)	Study
N2	730	41	9,8	200	3	TMIn flow
	730	48	13,2	200	3	
	730	53	15,8	200	3	
	730	41	10	30	3	Temperature
	770	41	9	30	3	
	810	41	4	30	3	
	730	41	10	30	3	Growth rate
	730	41	10,4	30	4,5	
	730	41	10,3	30	6	
H2	750	41	0	13	3	Growth rate
	750	41	0	13	3	
	750	41	0	13	3	
	750	41	0	13	3	
	750	41	0	30	3	TMIn + TMAl flows
	750	48	0	30	3	
	750	57	0	30	3	
	750	58	0	30	3	
	750	60	0	30	3	
	750	65	0	30	3	
	610	41	2,9	13	3	Temperature + growth rate
	610	41	5,5	13	6	
	675	41	0	13	3	

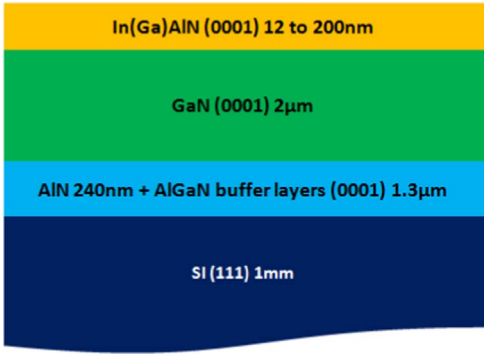

Fig. 1. Schematic representation of the growth structure.

layer using an AlN nucleation layer on nominally on-axis silicon (111) substrates, followed by AlGaN layers to control the strain in the GaN, followed by a 2 µm thick bulk layer of GaN grown at a temperature of 1035 °C, and finally the In(Ga)AlN layer. The precursors were tri-methyl indium (TMI), tri-methyl gallium (TMGa), tri-methyl aluminum (TMAl) and ammonia for indium, gallium, aluminum and nitrogen respectively. Different growth conditions were used for this final layer changing the TMIn flow, TMAl flow, the temperature, the growth rate and the thickness as summarized in Table 1. We kept a V/III ratio between 9000 and 18000 depending of the growth rate or TMIn flow and the pressure was at 100 mbar unless otherwise stated. Temperature of samples during the growth is measured using the EpiCurve TT module from Laytec. It combines a pyrometer and a reflectance measurement giving us an emissivity corrected temperature in six points at half radius of each wafer. We did the mean of these six values for each sample (Fig. 1).

To characterize these layers, we used first a Bruker Fast-scan AFM in tapping mode for to observe the topology and measure the roughness on 1×1 µm^2 fields. Secondly, in order to get the alloy composition of each sample we performed X-ray Photoelectron Spectroscopy (XPS) measurements using a PHI Versaprobe II. In this tool, the elemental transition used is the K_α line of aluminum. The spatial resolution at the surface is nearly 10 µm due to an X-ray beam focusing system. To estimate the composition of our alloy we first measured high resolution spectrum for each element (one orbital chosen depending on its theorical intensity in reference tables) of the expected alloy (cf. Fig. 2). Then we used the Multipak software to treat the signal. The software allowed us to measure the area below each peak for each element. Then we used an updated table available in the software in which all the sensitivity factors are classified by chemical elements. Each area is divided by this sensitivity factor, and finally the ratio between the different elements of the alloy was made to get the relative composition between indium, gallium and aluminum in the grown layer. The sensitivity factors permitted us to have an uncertainty of 5% on the relative composition of each element. We did not used reference samples measured by Rutherford backscattering spectrometry (RBS) to get new sensitivity factors and decrease the XPS uncertainty. 5% uncertainty was enough to get interesting trends. In addition, we benefited of an in-situ sputtering system in the XPS tool which allowed us to generate a composition profile through the thickness of the samples. The samples measured in this study had a flat profile, and so the values shown represent an average along the profile. Note that the each measurement point reveals the composition on a depth of 3 nm. That is the reason why we are seeing a composition gradient at each interface in the profile shown in Fig. 3.

3. Results and discussion

3.1. In(Ga)AlN growth under nitrogen

For the growth of In(Ga)AlN layers under nitrogen, we investigated the ratio between TMIn and TMAl precursors, the temperature, and the growth rate. For the first of these variables, we examined the effect on morphology and the indium composition in the InAlN layers while changing the TMIn flow and keeping the TMAl flow constant. Fig. 4 shows the AFM scans with the indium composition in the gas phase and in the alloy shown below.

These three layers are 200 nm thick and we see that if we increase the TMIn flow at a constant TMAl flow we increase the indium incorporated in the alloy, as expected, increasing the incorporated indium from 9.8% to 15.8%. There is also a direct effect on the morphology, with increasing indium increasing the diameter of grains in the surface structure and also increasing the roughness. High resolution transmission electron microscopy images (not shown here) proved that the grain morphology is present only at the surface, and the InAlN layer is continuous below.

In Fig. 5 we can see the effect of the temperature on the In(Ga)AlN