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A majored thermal nitridation process on GaAs (100) surfaces: optical, structural and chemical analysis

H. Vilchis, Víctor, M. Sánchez-R, and A. Escobosa
Department of Electrical Engineering,
Centro de Investigación y de Estudios Avanzados del Instituto Politécnico Nacional,
México D.F. 07360, México,
e-mail: heber_vil@hotmail.com

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Gallium nitride (GaN) thin layers were obtained by thermal nitridation of gallium arsenide (GaAs) [100] surfaces at Vapor Phase Epitaxy (VPE) system. Nitride films were a mixture of hexagonal and cubic GaN phases; this was confirmed by x-ray diffraction. The principal advantages our films are that present good smooth surface morphology with a thickness near to 5 μm. Also we showed as the thermal distribution modify the quantity of material near to substrate, which allowed to obtain rough films with band-band emission correspond to cubic phase (c-GaN) with intensity bigger than smooth films and without yellow band emission.

Keywords: GaN layers; thermal nitridation; VPE system.

Películas delgadas de nitruro de galio (GaN) fueron obtenidas mediante el proceso de nitridación térmica de superficies de arseniuro de galio en un sistema de epitaxia en fase vapor. Las películas nitridadas presentan una mezcla de las fases hexagonal y cúbica del GaN. Las principales ventajas de las películas obtenidas son: presentan morfología superficial con mínima rugosidad y tienen un espesor de 5 micras. Además se demostró que la distribución térmica modifica la cantidad de material cerca del substrato, lo cual permite obtener películas con mayor rugosidad que presentan emisión banda-banda correspondiente a la fase cúbica del GaN con una intensidad mejor que las películas de menor rugosidad y sin emisión de banda amarilla.

Descriptores: Películas de GaN; nitridación térmica; sistema VPE.

PACS: 68.37Ps; 78.55Cr; 68.37Yz; 81.05Ea; 81.15Gh.

1. Introduction

The chemical nitridation process consists in expose a metal in nitrogen atmosphere. The metals of group III are employee for obtain III-N compound semiconductors, which are of great scientific interest to the development of blue light emitting and others optoelectronic devices [1]. Nitridation process can be realized by thermal, plasma or ion assisted techniques at Molecular Beam Epitaxy (MBE) or Metal-Organic Chemical Vapor Deposition (MOCVD) systems [2,3]. This process is used for the formation of a thin nitrided film used as buffer layer in the fabrication of semiconductor devices.

In the last decade O. Brandt [4] showed that the nitridation of GaAs surface substrates is an important step to obtain high quality GaN epitaxial layers. So, scientific interest has been increasingly focused on the nitridation process of gallium surfaces, since this process is strongly correlated with the development of blue light emitting devices based on GaN material [1]. Currently the interest consists in synthesizing a GaN film with cubic structure due to is more consistent with actual semiconductors technology than hexagonal GaN. But the c-GaN films obtained present a fraction of h-GaN, so is important decrease the incorporation of hexagonal phase for improve the characteristic and properties of the c-GaN films and devices based in those.

Many works have been carried out about the nitridation of GaAs surfaces, their contribution have got obtain nitride film with good qualities [1-6]. Kikuchi *et al.* [5] demonstrated that experimental conditions as nitridation time and V/III ratio can be modify the characteristic of the nitride films obtain in a MBE system. Sun *et al.* [6] obtained nitrided films by MOCVD system and showed that the temperature and time influence in the structure of the films [6]. However, there is an inconvenient in all nitride films obtained until now; the films are ultra thin when they were obtained by MBE, the thickness varied from 20 – 50 Å [1,4]. And when the films are synthesized by MOCVD the films have thickness between 15 and 40 nm [7,8].

The motivations for this work were to obtain nitride GaN films with cubic phase predominant and achieve large thickness in the films, in order to use them as buffer layer or template in the fabrication or power and optoelectronic devices. The films were synthesize at VPE system and characterized by XRD to know the chemical structure of the films, by photoluminescence (PL) characterization was determined the emission spectrum of the films, the surfaces morphology was observed by Atomic Force Microscopy (AFM), the ellipsometry measurement allowed to determine the refractive index and the thickness of the films and Secondary Ion Mass Spectroscopy (SIMS) characterization showed the impurities in the films.

2. Experimental procedure

GaN nitride films were synthesized at atmospheric pressure by vapour phase epitaxy (VPE) or chemical vapour

deposition (CVD) system in a horizontal reactor with hydrogen (H_2) as carrier gas. We used insulated GaAs substrates which were chemically etched in a solution of H_2SO_4:H_2O_2:H_2O=5:1:1 at 60°C followed by a bath of HCl:H_2O=1:1 next to rinsed with deionised water. After that, the substrates were introduced into the reactor, purged with H_2 and after 60 minutes they were annealed in H_2 at 520°C for 30 minutes to remove the surface oxide. Afterwards the substrates were nitrided at 900°C for 90 min using a NH_3 flow of 500 standard centimeters cubic (sccm). The values of temperature, time and NH_3 flow were determined from several experiments where this conditions were varied until find these parameters that allowed to the results present here.

3. Results and discussion

3.1. X-ray diffraction

Figure 1 shows the result of the x-ray diffraction measurement for the nitride films obtained. In the XRD spectrum we can see seven peaks; two of them corresponding to the substrate employee, GaAs (002) at 32.1° and (004) at 70.05°. The other peaks correspond to GaN, showing the cubic and hexagonal phases with different orientations. This indicates that the nitride films are a mixture of both phases. For c-GaN we have detected the following two orientations, (111) at 34.05° and (200) at 39.5°. For h-GaN we can see three peaks, at 36.30° for (10-11), at 58.2° for (11-20) and 64.5° for (10-13).

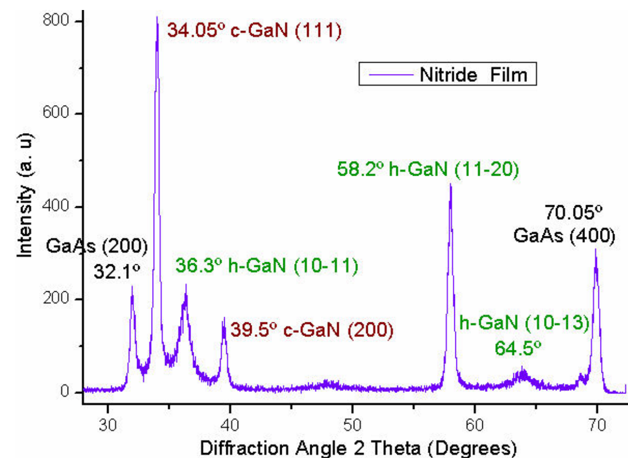

FIGURE 1. XRD spectrum for nitride films on GaAs substrates.

3.2. Atomic force microscopy

Figure 2 shows the AFM image for a nitride film, it is possible to appreciate different small islands that indicate the growth of the film is no uniform over all substrate. The root mean square (RMS) deviation is 43.9 nm, which is a low valuable of roughness considering the thickness of the films, which is almost 5μm. We think the growth was in form of island called as Stranski-Kranstanov [9]. This involves a growth of islands after the first layers, due to the decrease binding energy in the film. The islands or grains can be seen in Fig. 2.

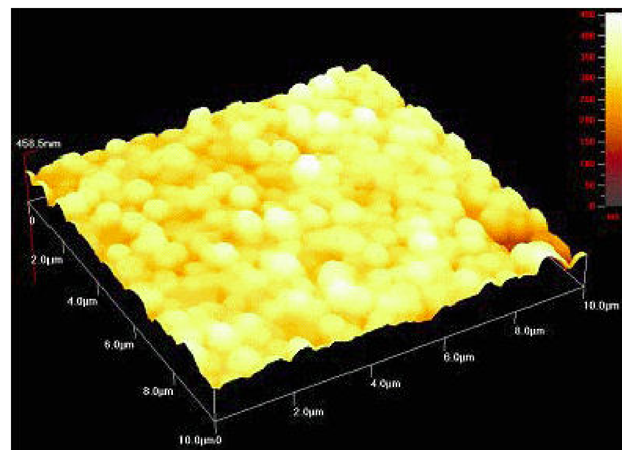

FIGURE 2. AFM surface image of nitride film.

3.3. Photoluminescence

Figure 3 shows the typical photoluminescence (PL) spectrum at room temperature for the nitride films. We can see two types of emissions. The first emission is around 388 nm or 3.195 eV, this energy indicates that the film present band-band emission corresponding to cubic phase. The wide of the peak mean there are others level with high or low energy participating in the emission. The second emission is around 524 nm or 2.362 eV, this response in GaN films is called yellow band emission and is attributed to defects in the film for the mixture of both phases.

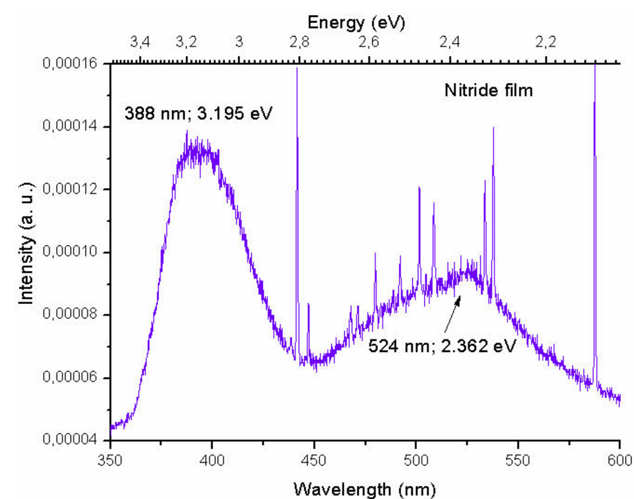

FIGURE 3. Photoluminescence spectrum of the nitride film.

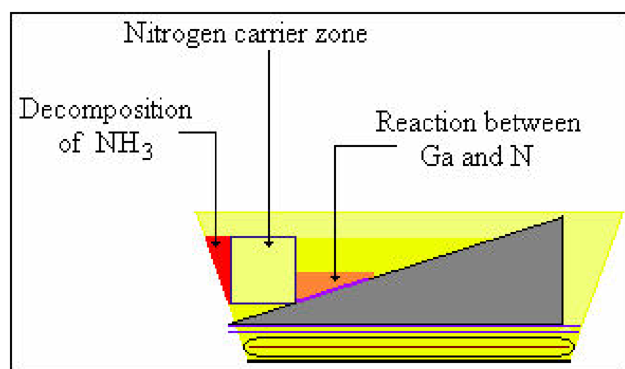

FIGURE 4. Thermal distribution near to substrate.

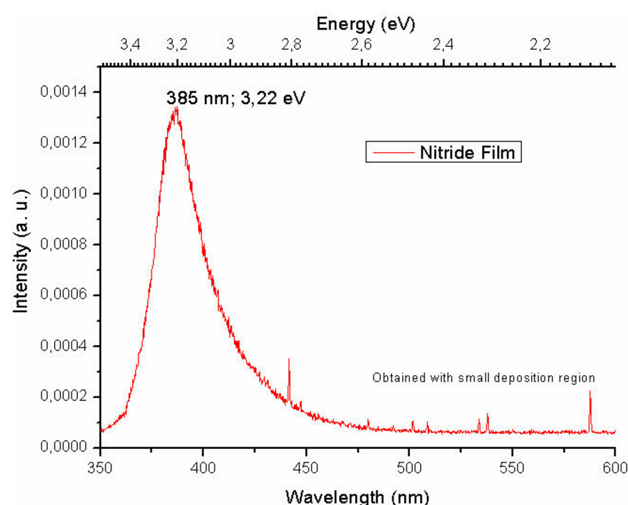

FIGURE 5. Photoluminescence spectrum of the nitride film obtained with decomposition region reduced.

3.4. Nitride films without yellow band emission.

With the same experimental conditions was possible to obtain other characteristics in nitride films when the thermal distribution near to substrate was changed. Figure 4 shows a schematic diagram about the thermal distribution. We can see two important zones, the first is the decomposition region, in this zone the ammoniac is decompose due to the energy of the lamps, so it is possible to obtain free nitrogen. The second zone is over substrates and is called deposition region because is where the gallium of surface reaction with the nitrogen free formed the GaN layers. If the decomposition region is reduced the quantity of the nitrogen free decrease and the nucleation on substrate was no homogenous in all surface, in this case the nitride films were rougher than the nitride films obtained when the decomposition region is large. The emission spectrum is modifying too, Fig. 5 showed the emission of the rough nitride film. We can see an only peak at 385 nm corresponding to band-band emission for c-GaN. The result does not indicate that the nitride film has pure cubic phase, on the other hand, the growth of the film was by islands and in the top of these the favoured structure is cubic so we cannot see yellow band emission in the photoluminescence respond.

4. Conclusions

We proposed a new experimental conditions for synthesize nitride films by thermal process at VPE system. The principal advantage is that was possible to obtain nitride films with more thickness than nitride films reported in the literature; we obtained films of 5 μm thickness. The thickness was measurement using ellipsometry characterization. Although the films are a mixture of cubic and hexagonal phase have good superficial and optical characteristics. We obtained films that present band-band emission correspond to Eg of c-GaN with good intensity and without yellow band emission at ambient temperature, Ref. 7 present GaN films with yellow band emission but is necessary low the temperature until 6 K. Also we are the first in demonstrated the influence of the thermal distribution near to substrate in the nitride films. This will be an important issue for futures experiment in MOCVD systems. When the decomposition region is reduced the films obtained were rougher but presented better emission spectrum than first films (obtained with a big decomposition zone). Chemical composition, emission intensity and surface morphology characteristic allow to determine that the nitride films are suitable for use them as substrate in the fabrication of optoelectronic devices.

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