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Understanding the influence of physical properties on the mechanical characteristics of Mg-doped GaN thin films

Z. Benzarti ^{a,b,c,*}, A. Khalfallah ^{d,e,f}, Z. Bougrioua ^g, M. Evaristo ^d, A. Cavaleiro ^d

^a Laboratory of Multifunctional Materials and Applications (LaMMA), Department of Physics, Faculty of Sciences of Sfax, University of Sfax, Soukra Road km 3.5, B.P.1171, 3000, Sfax, Tunisia

^b Instituto de Telecomunicações and Department of Electronics, Telecommunications and Informatics, University of Aveiro, Campus Universitário de Santiago, 3810-193, Aveiro, Portugal

^c Department of Materials and Ceramic Engineering, CICECO–Aveiro Institute of Materials, University of Aveiro, 3810-193, Aveiro, Portugal

^d CEMMPRE, Department of Mechanical Engineering, University of Coimbra, Rua Luís Reis Santos, 3030-788, Coimbra, Portugal

^e Laboratoire de Genie Mécanique, Ecole Nationale d'Ingénieurs de Monastir, Université de Monastir, Av. Ibn El-Jazzar, 5019, Monastir, Tunisia

^f DGM, Institut Supérieur des Sciences Appliquées et de Technologie de Sousse, Université de Sousse, Cité Ibn Khaldoun, 4003, Sousse, Tunisia

^g IEMN, Institute for Electronics, Microelectronics and Nanotechnology, CNRS and Lille University, Avenue Poincaré, 59652, Villeneuve d'Ascq, France

* Corresponding author. Laboratory of Multifunctional Materials and Applications (LaMMA), Department of Physics, Faculty of Sciences of Sfax, University of Sfax, Soukra Road km 3.5, B.P. 1171, 3000, Sfax, Tunisia.

E-mail addresses; zohra.benzarti@fss.usf.tn (Z. Benzarti)

Abstract

We This study investigates the impact of Mg doping concentration in GaN thin films on their physical and mechanical properties, specifically on their conductivity transition, luminescence, hardness, Young's modulus, and creep behavior. Mg-doped GaN layers were grown on sapphire substrates using the MOCVD technique, with trimethylgallium (TMG) and bis(cyclopentadienyl) magnesium (Cp₂Mg) as precursors for Ga and Mg, respectively. The TMG flow rate was set at 20 μmol/min, while the Mg flow rate was adjusted by tuning the temperature of the thermostatic bath of Cp₂Mg. Three different Cp₂Mg flow rates: 2, 7 and 9 μmol/min were utilized to explore their effects on the physical and mechanical properties of the grown epilayers. The results demonstrate that an increase in the Mg concentration leads to a conductivity transition from n-type to p-type, with the highest hole concentration $(4.5 \pm 0.2) \times 10^{17} \text{ cm}^{-3}$ and blue luminescence attained with Mg concentration equal to $2.1 \times 10^{19} \text{ atoms/cm}^3$. Moreover, the hardness, Young's modulus and the intensity of compressive stress increase with the Cp₂Mg flow rate due to the evolution of pre-existing dislocation density and point defects incorporation. Additionally, this study evaluates the creep behavior of the Mg-doped GaN thin films. It shows that both the creep stress exponent and the maximal creep depth decrease with the increase of the Cp₂Mg flow rate. This is attributed to dislocation glides and climbs governing the creep mechanism. Accordingly, this investigation highlights the importance of understanding the relationship between physical properties and mechanical characteristics of Mg-doped GaN thin films, which have potential implications for various technological applications.

Keywords: Mg doped GaN thin films; MOCVD; point defects; Nanoindentation; Creep

1. Introduction

Metalorganic chemical vapor deposition (MOCVD) is widely accepted as the most commonly used technique for the growth of GaN thin films, as it provides the highest throughput for optoelectronic devices. Basically, diodes consist of a carrier injection between n-type and p-type layers. The most critical problems encountered in nitride layer production is the achievement of p-type doping, because GaN layers have n-type doping residuals. Several dopant elements such as Zn, Be, C, Ca, and Mg have been tested. However, the dopant layers obtained compensated with high resistivity. Today, it is commonly assumed that Mg element is the only p-type dopant used to produce GaN layers of p-type, although the atomic concentration of Mg is low, about 1%~2% in GaN and it can act as acceptors [1]. Despite the low ionization rate of Mg acceptors (ranging from 1% to 5% at 300 K) [2], Nakamura et al. [3] have developed p-type doping process using Mg-doped GaN layers annealed under N₂ carrier gas in the MOCVD process. The anneal process is necessary for the electrical activation of the Mg by the removal of the Mg–H complex.

Svenska et al. [4] reported that reducing the amount of hydrogen in the growth process would be an effective way to enhance the electrical properties of the p-type GaN layer. Although efforts have been made to enhance the electrical properties of p-type GaN layers, hole mobility in Mg-doped GaN layers is still low because of the high ionization energy of the Mg atoms in GaN (around 200 MeV) [5]. Definitely, it is essential to use a high Mg concentration to obtain effective p-type GaN layers. However, the formation of point defects and/or defect

complexes is the drawbacks of using high Mg atoms incorporation in the GaN layer. In addition the self-compensation [6] is the source of the low hole mobility which produces high resistivity in the best *p*-type GaN layers [7]. Romano et al. [8] reported that the low hole density is accredited to the incorporation of Mg in electrically inactive form and/or the formation of compensating Mg-induced defects at high Mg concentrations. The researchers stated that the high Mg concentration in Mg-doped GaN layers causes the development of surface roughness for films grown using MOCVD. Vennéguès et al. [9] observed Mg precipitates or pyramidal defects in MOCVD Mg-doped GaN layers with Mg concentration higher than $2 \times 10^{19} \text{ cm}^{-3}$. The concentration of pyramidal defects was revealed to be large enough to play a remarkable role in the self-compensation of Mg-doped GaN layers. High Mg concentration links to both the saturation of free hole density and the appearance of the blue photoluminescence transition at 2.70–2.95 eV [10]. Whereas, low Mg concentration depicts luminescence peaks at 3.270 eV and 3.466 eV corresponding to donor acceptor pairs band ($D^{\circ}A^{\circ}$) and near band edge emission (NBE), respectively [11].

In our previous work, we found that high Mg incorporation was accomplished when the flow rate of TMG was reduced [12]. Indeed, we used a constant Mg flow rate of 5 $\mu\text{mol}/\text{min}$ and a TMG flow rate decreasing from 40 $\mu\text{mol}/\text{min}$ to 16 $\mu\text{mol}/\text{min}$ during the growth process of Mg doped GaN structure. We demonstrated using spectroscopy of secondary ion mass (SIMS) analysis that the incorporation of Mg atoms increased as long as the layer depth raised (i.e., decrease of TMG flow rate). Recently, Nayak et al. [13] showed that Mg-doped GaN growth process with a high hole density can be completed by imposing Ga-poor growth conditions rather than Ga-rich conditions. For this reason, we developed a set of Mg-doped GaN layers that differ only in the Cp_2Mg flow rate; i.e., for different Mg concentrations in Ga-poor growth conditions.

For the sustainable success of optoelectronic devices, the understanding of fundamental properties of Mg-doped GaN layers is of paramount importance. Besides to probing the optical and electrical properties, the mechanical properties are also of special concern for functional elements in optoelectronic devices. For instance, Jian et al. [14] investigated the effect of Mg doping on the conventional mechanical properties of GaN films using the nanoindentation technique. However, the derived values for hardness and Young's modulus are likely prone to the indentation size effect, given the low applied loading forces. Besides, mechanical characterization, such as the creep behavior, was not performed.

To the best of our knowledge, scarce studies in literature report on the creep behavior of semiconductor thin films and have not been investigated in detail [15]. In addition, the study of nanomechanical properties of Mg-doped GaN epilayers at elevated loads has been seldom reported. In particular, the nanoindentation creep behavior of Mg-doped GaN layers have not been tackled yet in the literature. Hence, conducting a thorough assessment of the mechanical behavior of Mg-doped GaN layers in relation to their physical properties is of substantial importance. In this work, the effect of Mg-doping on GaN layers' physical properties, nanomechanical characteristics and creep behavior was systematically investigated. The obtained results demonstrate that the incorporation of Mg defects in GaN films has influenced their physical properties, which in turn affects their nanomechanical characteristics.

Presumably, the current study opens new scientific perspectives for thorough investigations of creep behavior for semiconductor thin films for fostering targeted combinations of functional and mechanical properties for better benefits from their characteristics.

2. Experiments

A set of Mg-doped GaN epilayers with different acceptor concentrations was grown on (0001) *c*-plane sapphire substrates by MOCVD. The sapphire substrate was nitrided at 1100 °C. Then, a buffer layer of GaN (30 nm) has been deposited at 600 °C. The Mg-doped GaN layer was deposited on GaN buffer layer after being annealed in a mixture of H_2 , N_2 and NH_3 gases. The thickness achieved for Mg doped GaN samples is about 1 μm . Mg doped GaN films were prepared at 1100 °C with 3 slm of ammonia (NH_3) and 3.6 slm of nitrogen (N_2). The Metal-organic precursors for Ga, and Mg were trimethylgallium (TMG) and bis(cyclo-pentadienyl) magnesium (Cp_2Mg), respectively. These precursors were maintained in a thermostatic bath to control their vapor pressure. TMG flow rate is fixed at 20 $\mu\text{mol}/\text{min}$, while Mg flow rate was controlled by tuning the temperature of the thermostatic bath of Cp_2Mg in order to obtain adjustable Cp_2Mg flow rate with only 0.4 slm of hydrogen (H_2) as carrier gas. The Cp_2Mg flow rate is about 2 $\mu\text{mol}/\text{min}$ for sample A, 7 $\mu\text{mol}/\text{min}$ for sample B and 9 $\mu\text{mol}/\text{min}$ for sample C. The Mg concentration ($[\text{Mg}]$), which was measured using SIMS by means of EVANS EAST facility, is about $1.1 \times 10^{18} \text{ atoms}/\text{cm}^3$, $2.1 \times 10^{19} \text{ atoms}/\text{cm}^3$ and $6.1 \times 10^{19} \text{ atoms}/\text{cm}^3$ for sample A, sample B and sample C, respectively. In order to activate the Mg acceptors, Mg doped GaN layers were annealed in N_2 ambient at 900 °C during 30 min. Room-temperature Hall effect measurements with the standard Van der Pauw configuration were performed to determine hole density. The surface morphology and its root-mean square roughness (RMS) were evaluated by AFM (Bruker-Model Nanoscope IIIA Multimode). Structural properties were studied and the dislocation density was computed using a high resolution XRD system-BRUKER D8 Discover. PL measurements were performed

using an argon laser doubled in frequency (244 nm) as an excitation source with a power of 15 mW. It was used to deduce the activation energy of Mg atoms by varying the temperature and laser excitation intensity. Room-temperature Raman spectroscopy using a Labram HR EVOLUTION instrument with a red laser emitting at 633 nm and a power of 100 mW, was carried out. The purpose of the spectroscopy was to probe the residual stresses within Mg-doped Ga samples. Nanoindentation tests were carried out at room-temperature using “NanoTest NT1, Nano Materials, Ltd” instrument equipped with a diamond Berkovich-type indenter. The three-sided pyramidal Berkovich shape has a half angle of 65.3°. The Berkovich indenter radius is equal to 100 nm. 20 nanoindentation tests were performed with a maximum load of 10 mN on each sample. The indentation imprints were separated by 30 µm to avoid any interactions between them that may alter the measurement results. Besides, the indentation depths were carried out at less than 10% of the film thickness to eschew the sapphire substrate effect. Moreover, SEM technique (Zeiss Merlin Gemini II equipment) was used to observe and analysis the indentation imprints for Mg doped GaN samples.

3. Results and discussion

- 3.1 Study of the surface morphology
- 3.2 Study of the structural properties
- 3.3 Study of the electrical properties
- 3.4 Study of the optical properties
- 3.5 Study of the lattice vibrational properties
- 3.6 Investigation of the anomechanical behavior

3.6.1. Determination of nanomechanical properties

3.6.2. Assessment of the creep behavior

4. Conclusion

This paper analyzes the physical and nanomechanical properties of Mg-doped GaN layers deposited on sapphire substrates using the MOCVD technique. The effect of increasing the Cp2Mg flow rate on the structural, morphological, electrical, optical, and lattice vibrational properties using several probing techniques was observed. The studied layers showed a degradation of surface morphology, an increase in defects, and a rise in resistivity and stresses as the Mg concentration increased. Moreover, the conductivity transitions from n-type to p-type was obtained with the generation of blue luminescence owing to Mg defect complexes as deep donors. This was observed for the Cp2Mg flow rate equal to 7 µmol/min, which achieved the highest hole concentration of $(4.5 \pm 0.2) \times 10^{17} \text{ cm}^{-3}$ and suggests potential use of Mg-doped GaN layers in optoelectronic devices. On the other hand, an increase in the Cp2Mg flow rate was also observed to affect the nanomechanical properties by increasing the hardness and Young’s modulus (hardening effect), and the H/E ratio, which could be advantageous for certain applications requiring high-strength materials. This evolution is grounded in the increase of the compressive stress, pre-existing dislocation density along with the introduction of point defects during the incorporation of Mg doping into GaN layers. This leads to the decrease in creep stress exponent and the maximum creep depth, which are attributed to the inherent creep mechanism governed by dislocation glides and climbs. Subsequently, our findings underscore the significance of grasping the correlation between the physical properties and mechanical characteristics of Mg-doped GaN thin films, which have potential implications for a wide range of technological applications.

CRediT authorship contribution statement

Z. Benzarti: Conceptualization, Formal analysis, Methodology, Investigation, Writing – original draft, Writing – review & editing. A. Khalfallah: Conceptualization, Formal analysis, Methodology, Writing– original draft, Writing – review & editing. Z. Bougrioua: Resources, Methodology. M. Evaristo: Resources, Methodology. A. Cavaleiro: Resources, Methodology.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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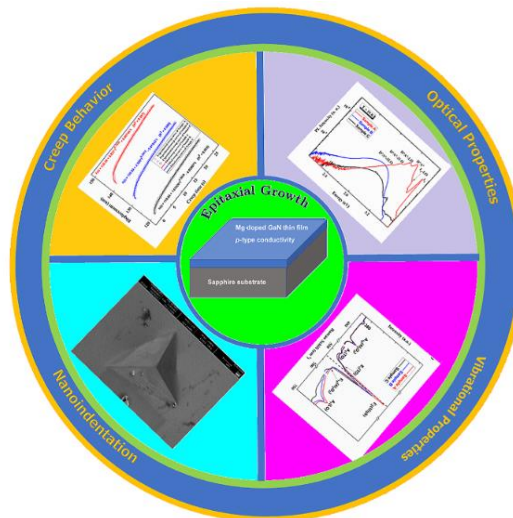
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Graphical Abstract



Highlights

- Mg-doped GaN films on sapphire were deposited using MOCVD technique.
- Mg-doping effects on physical and mechanical properties of GaN films were systematically studied.
- Cp₂Mg flow rate was found for n to p-type conductivity transition, enhancing LED efficiency.
- Mg incorporation impacts Young's modulus, hardness, and creep exponent.
- Creep behavior suggests dislocation glides and climbs as governing deformation mechanism.