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Comparative study of threading dislocations in GaN epitaxial layers by nondestructive methods

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A B S T R A C T

An investigation of the threading dislocations (TDs) density in two high quality GaN epitaxial layers grown on sapphire (Al_2O_3) substrate, with the thickness of 300 and 1000 nm, respectively, is presented in this paper. In the case of GaN_{300} sample, taking into account that the finite size effect exerts an increased influence, further advanced methods are necessary to find the correct results besides the standard analysis of the Full Width at Half Maximum (FWHM) of the X-ray rocking curves peaks as in the case of the thicker sample, GaN_{1000}. Thus, we explored the tails of the rocking curves using a universal power law fit and subsequently, we used modified version of the Williamson-Hall plots. Analysis of the XRD profiles allowed us to avoid the overestimation of the threading dislocations density determined by the influence of the correlation between dislocations. Moreover, we successfully separate the contribution of the strain and size broadening based on the analysis of refined Williamson Hall plots and demonstrate the nearly uniform dislocation density depth distribution of the GaN studied films, with no supplementary nucleation or annihilation centers are present inside the epitaxial film using, both grazing incidence and grazing exit configurations of the asymmetric geometry.

1. Introduction

The unique properties of III-nitride based compounds compared to other semiconductor materials (e.g. wide direct band gap, high breakdown electric field, chemical physical and thermal stability) made them a legitimate opportunity for modern electronic and optoelectronic devices [1]. Accordingly, intensive efforts were devoted to explore their feasibility and to bring to the market environmentally friendly GaN-based devices, including high-power and high-frequency electronics [2], photodetectors [3], or light emitting diodes (LEDs) [4]. The heteroepitaxial growth of GaN epilayers on foreign substrates, with different lattice parameters, such as sapphire, silicon carbide or silicon lead to inherent issues related to the heterointerfacial threading dislocation density and the large residual strain.

Threading dislocations are the dominant defects in GaN films and they appear at the interface between substrate and epitaxial layer. In general, two types of threading dislocations (TDs) can be distinguished [5]. The majority of dislocations are edge type dislocations and their density is normally 10^8–10^{10} cm^{-2} [6], while the screw dislocations density are one to two orders of magnitude lower than the edge dislocations density [7]. These structural defects induce negative effects on optical and electrical properties of GaN-based devices and can also influence device reliability and lifetime, although they might be responsible for useful additional properties when their type and density are controlled [8,9]. In this context, it is important to balance correctly the required optical and electrical properties of such materials with their growth processes.

Alternative approaches were proposed to measure the dislocation density, combining the experimental techniques with numerical image analysis and theoretical calculations, each of them showing both advantages and disadvantages. In essence, the Hirsch group firstly reported the direct observation of dislocations by transmission electron microscopy (TEM) in 1956 [10] and this method is still the most effective. With TEM one can directly determine the character of dislocations and their distribution in thin films, but given the small size of the investigated area, their density has to be high enough to be observable in the field of view. TEM sample preparation represents another drawback, because it is difficult to prepare and time-consuming, especially when sapphire or silicon carbide are used as substrates. This method also lacks the statistical relevance because only a small volume of sample can be analyzed.

Scanning electron microscopy (SEM) and atomic force microscopy

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(AFM) are two non-destructive microscopic techniques providing lower resolution but allowing instead superior statistic information when compared to TEM. Even if the AFM analysis does not require laborious sample preparation as the TEM ones, it was shown that a preliminary surface treatment is necessary to ensure that all the dislocation pits are imaged [11,12]. Unlike the previous mentioned methods, with X-ray diffraction (XRD) method, a large area is probed, and thus point-sampling errors inherent to all microscopy-based methods are eliminated. Additionally, it does not require a special sample preparation and the matching of X-Ray wavelength to the atomic scale of the semiconductors is an important advantage. Consequently, the XRD measurements of threading dislocation density are increasingly used to characterize GaN epilayers grown on different substrates, supporting the further development of GaN-related devices.

The threading dislocation density is directly proportional to the square of the Full Width at Half Maximum (FWHM) of the rocking curve [13,14]. This is only an approximation, because at the FWHM contribute also the intrinsic rocking curve, width of the instrumental broadening, the strain broadening, the mosaicity and the sample curvature. If for high dislocation density films the ω-scan broadening sources can be overlooked, when low dislocation density films are analyzed, they become significant [15]. A different model was proposed by Kaganer et al., where the diffuse scattering from XRD diffraction line tails are taken into consideration, instead of FWHMs of the peaks [16–18]. Consequently, since they are not influenced by the correlations between dislocations, they follow the universal power laws and the density of TDs is calculated from the tail slopes (q^{-2}, q^{-3}, q^{-4}).

In this paper, we report a comparative analysis of the TD density in two high quality GaN epilayers on sapphire, with low density of dislocations and different thicknesses, using nondestructive characterization methods, like AFM and HR-XRD techniques. Since we analyzed the untreated GaN surfaces, the AFM data represent only a qualitative view of the investigated samples, being a preliminary step to the X-ray based analyses. The X-ray rocking curve data allowed us to obtain the TDs density using the tail analysis, and, moreover, to find a correlation between the components of TDs. The strain and the size broadening of FWHMs, due the finite size of the layer, were separated using the modified Williamson-Hall plots, a powerful method where all available asymmetric reflections are taken into account. Moreover, the effect of sample curvature was circumvent using narrow slit at X-ray source (1 mm) for the X-ray measurements. Also, using two special configurations of the asymmetric skew geometry, such as through Grazing Incidence - XRD at small incident angle and through Grazing Exit – XRD, respectively, we demonstrate that the dislocation density is constant in the depth of the GaN films, indicating that no supplementary nucleation or annihilation centers are present inside the epitaxial film. These results consist in a step further towards a better understanding of the relationship between generation of edge and screw components of TDs, their dislocation density depth profile of the GaN epilayers on foreign substrates and how to separate the strain and the finite size effect in FWHMs of the rocking curves.

2. Experimental details

Two commercial substrates with epitaxially grown GaN layers on sapphire substrates with different thicknesses – 300 nm (Sample 1 – GaN_{300}) and 1000 nm (Sample 2 – GaN_{1000}), respectively – were investigated in this paper by both XRD and AFM techniques.

The AFM images of the GaN surface morphology have been acquired using Ntegra Aura (Nt-MDT) equipment, operated in semi-contact (intermittent-contact) mode. A fresh silicon probe (*HA_NC, Nt-MDT*) with 12 N m^{-1} nominal elastic constant, 235 kHz resonance frequency and < 10 nm tip radius was used in the measurements. Detection of dislocation pits on the GaN surface were performed using a threshold-based grains & pores analysis algorithm included in the *Nova* image analysis software of the SPM microscope.

"Rocking curve" diffraction profiles (omega scans at fixed 2theta Bragg angle) were collected for several (hk.l) lattice plane orientation in both symmetrical and/or asymmetrical skew/grazing incidence GI-XRD/ grazing exit GE-XRD configuration using a 9 kW rotating anode SmarLab diffraction system (Rigaku Corporation, Japan). The diffraction system was configured in the parallel beam mode using a multilayer X-ray mirror (Cross Beam Optics – CBO module) with an axial divergence of 0.05 degrees in the vertical diffraction plane of the goniometer. Although the absolute lattice parameter measurements (high resolution triple axis multiple reflection method) can be also performed, we used the double axis method (a four bounce Ge(220) monochromator at the X-ray source and an open detector), which is a relative lattice parameter low resolution XRD method. The double axis XRD method offers a very high diffracted X-ray beam intensity at the detector side and thus a reduced measurement time, particularly on a 9 kW rotating anode system (fast X-ray scans, typically below 10 min) with the disadvantage of low resolution beam (at the detector side-open detector, all diffuse scattering from the surface is accounted). Moreover, the asymmetric diffraction peaks are easier to measure because of the higher intensity collected at the detector side. Another important fact is that for highly distorted epitaxial thin films the triple axis method did not usually work, particularly for asymmetric (low structure factor) lattice planes. Kaganer demonstrate that the two methods give a different power law of $I_{diff}(\omega)$ – triple axis method gives a $I \sim q^{-4}$ while the double axis method gives $I \sim q^{-3}$, and that the double axis method has certain theoretical and practical advantages (higher diffuse scattering intensity collected at the detector side).

For our diffraction experiments we used $2 \cdot 10^{-4}$ degrees omega steps, with the speed ranging from 0.1 to 12 degrees/min. The X-ray generator operating voltage and current nominal values of the SmartLab diffraction system were fixed at U=45 kV and I_{tube}=200 mA. Besides the four bounce Ge(220) monochromator, we use a multilayer mirror (Rigaku Cross Beam Optics module) to define a parallel beam (PB) optical mode, standard Soller slits of 5° at the detector side and an open detector (receiving & incident slits RS1, RS2=IS=1 mm).

3. Results and discussion

Since the threading dislocations cross the layer along its normal, from the layer-substrate interface to the surface, the termination of TDs at surface gives rise to small pits, and these topographic changes are imaged by AFM technique. The AFM image of the GaN_{300} surface topography is presented in Fig. 1. As it can be observed, pits with different sizes are revealed on the GaN surface, which can be related to the character of dislocations. The character of the dislocations might be

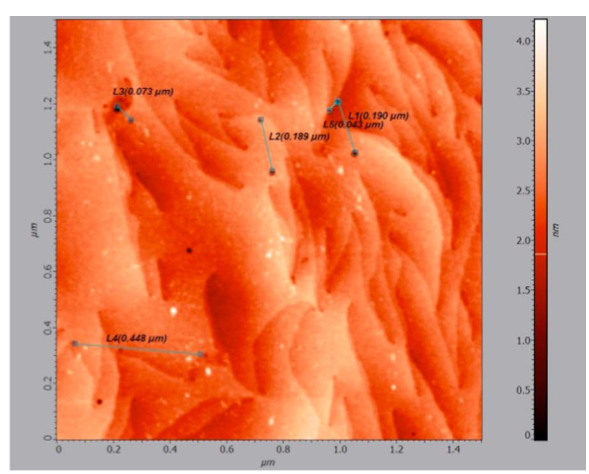

Fig. 1. AFM image of Sample 1 and the corresponding distance between dislocations. The analyzed surface area is 1.5×1.5 μm^2.