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Electrical properties of extended defects in III-nitrides

Albert Minj,[a,1] Daniela Cavalcoli,[a,*] Geeta Rani Mutta Popuri,[a,b] Arantxa Vilalta-Clemente,[b,2] Pierre Ruterana[b] and Anna Cavallini[a]

[a]*Department of Physics and Astronomy, University of Bologna, Italy*
[b]*CIMAP, UMR 6252, CNRS-ENSICAEN-CEA-UCBN, 6, Bd Maréchal Juin, 14050 Caen Cedex 4, France*

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Abstract—Electrical and structural properties of extended defects including threading dislocations/V-defects and nanopipes in unintentionally doped GaN, InGaN (50 nm)/GaN and AlInN (33 nm)/AlN(1 nm)/GaN heterostructures have been investigated by means of various scanning probe (Kelvin probe and conductive-Atomic Force Microscopy) and electron beam (electron beam induced current and transmission electron microscopy) microscopy methods. Due to low energy measurements of Kelvin probe force microscopy, charge state of the dislocations have been correctly identified where threading dislocations (TDs) with screw-component are negatively charged, while pure-edge type TDs are neutral in InGaN/GaN. It is explained how various factors such as indium segregation, surface termination, presence of vacancies and/or impurities affect the electrical charge, conductivity and recombination properties of the extended defects. They are found to be strongly correlated to the type of dislocations as identified from TEM.

Keywords: III-Nitrides; Dislocations; V-defects; Electrical activity of extended defects; AFM

1. Introduction

The III–V nitrides constitute a unique semiconductor material family with outstanding properties [1–4], however, this goes along with the possible structural and chemical problems due to the differences in their intrinsic physical properties [5–7]. As the fabrication of devices needs the deposition of heterostructures, extended defects may play a major role in their performance. They have been identified as threading dislocations (TDs) [8–12], inversion domains [13–16], stacking faults [17–20], nanopipes [21,22] and V-defects [23,24]. They form during the epitaxial growth process in order to accommodate lattice and thermal mismatch, and they are detrimental to III-nitride based devices [25,26]. In particular, TDs often terminate on the surface as inverted pyramidal structures, which are also known as V-defects [27]. It has been shown that only those TDs that have a screw-component in AlInN and InGaN form V-defects, while pure edge-type TDs do not form V-defects [28]. Notwithstanding many studies published in the last years on dislocations and V-defects, a general consensus on their electrical and optical properties has not been achieved. In references [29–30], it is experimentally demonstrated that dislocations are negatively charged using the Coulomb scattering model to justify the temperature dependence of mobility (μ) in n-type GaN (10^{16}–10^{20} cm^{-3}). Look et al. [31] modeled the scattering mechanism of free carriers with charged dislocations in GaN and obtained a very good agreement with experimental data of free carrier concentration and mobility over a wide range of temperatures. In a previous report [32], we demonstrated that this effect is particularly important in AlInN/GaN based heterostructures when the threading dislocation density (TDD) exceeds 10^8 cm^{-2}. In their Density Functional Theory (DFT) theoretical calculations, Elsner et al. [33] reported that TDs, both edge and screw, may be electrically inactive and do not form deep gap states in wurtzite GaN, while screw dislocations in full-core configuration could form deep gap states due to the associated large bond distortions. Using atomic resolution Z-contrast imaging Xin et al. reported that pure edge and mixed dislocations in GaN exhibited an eightfold ring core structure and pointed out that screw dislocations could exist both as full core and open core structures [34]. Apart from theoretical and indirect determination of the charge state of dislocations, direct experimental observations were also performed, such as Electron Holography (EH) and Ballistic Electron Emission Microscopy (BEEM). However, these techniques have given contradictory results, as fixed negative and positive charges were reported by EH and BEEM, respectively [35,36]. From the above mentioned reports, it is

* Corresponding author.
[1] Present address: Institute of Materials Science, University of Valencia, Spain.
[2] Present address: Department of Materials, University of Oxford, Oxford OX1 3PH, United Kingdom.

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worth noting that: (i) the origin of charge states at TDs in wurtzite GaN and GaN alloys is still unclear; (ii) indeed, theory predicts that pure edge and screw dislocations are electrically inactive with no deep states in the gap, and the experimental reports suggest an electrical activity and deep level states for TDs in wurtzite GaN. Of course one point may be that the studied configurations are not completely identical which may explain the data difference. It is certain that in these materials, not only for binary but also for ternary and quaternary alloys, atomic structure and impurity segregation would play a strong role and this will probably depend on the growth conditions, which may have been different from one report to the other.

The present study aims at clarifying the role of electrical charge, indium segregation and presence of impurities at extended defects in GaN, InGaN and AlInN layers. In particular, electrical properties of V-defects and TDs are studied by mapping the electrical properties of III-N based alloys at the nanoscale by scanning Kelvin probe microscopy (SKPM), conductive AFM (C-AFM) and electron beam induced current (EBIC). The results are correlated to structural analyses carried out by Transmission Electron Microscopy (TEM).

In Section 2, the experimental details on samples and experimental methods are reported. In Section 3 the results are presented and discussed for each sample set examined and finally in Section 4 the conclusions are drawn.

2. Experimental methods

Three sets of samples have been investigated: unintentionally doped *n* type-GaN (3-μm) samples (set 1), InGaN/(50 nm)/GaN (3 μm) heterostructures with varying In% (set 2) and AlInN (33 nm)/AlN(1 nm)/GaN(3 μm) heterostructures (set 3). The details on the investigated AlInN/GaN heterostructures can be found in reference [37]. All the heterostructures were grown by Metalorganic Chemical Vapor Deposition (MOCVD). The GaN substrate was grown at 1050 °C on sapphire using a low temperature (~530 °C) GaN nucleation layer. In AlInN/AlN/GaN heterostructures, AlN and AlInN were both grown at ~780 °C and InGaN/GaN layers were grown between ~720 °C and ~730 °C. Indium percentage in InGaN/GaN samples as assessed by High resolution X ray diffraction is around 17%.

The electrical conductivity, surface potential and recombination properties of extended defects have been investigated by Scanning Probe methods including C-AFM, SKPM and EBIC. AFM measurements were performed with NT-MDT Solver-Pro 47 instrument in ambient conditions. The C-AFM maps are obtained in contact mode by measuring simultaneously the current while a constant bias is applied between the conductive tip and the grounded sample. SKPM measurements were performed in two-pass modes. In the first pass, topography is obtained in constant-Amplitude tapping mode. In the second pass, Kelvin potential is obtained where the AFM tip is AC biased with 1.5 V. Doped-diamond tips with resonance frequency = 210 kHz, force constant = 72 N/m were used. Due to the moderate value of force constant, the same tip was used both in static and dynamic modes, allowing for simultaneous SKPM and C-AFM mapping. Ohmic contacts were obtained by indium soldering. EBIC studies were carried out on Schottky contact fabricated by vacuum evaporation of a 50 nm thick gold layer. In order to avoid electrodiffusion phenomena no annealing procedure has been applied to obtain the junction and the EBIC measurements were carried out without any applied bias.

The heterostructures were examined in cross sections by TEM to determine the nature and the type of dislocations, along with their relationship with V-defects. To this end, the TEM specimens were prepared using conventional mechanical polishing followed by Ar+ milling at 5.0 keV with the beam angle at 5° incidence. In order to minimize the ion beam damage, the samples were maintained at the liquid nitrogen temperature during ion milling and a final cleaning step of 0.7 keV was applied. The specimens were observed in conventional TEM using JEOL-2010 microscope operated at 200 keV.

3. Results and discussion

3.1. Scanning probe studies of GaN layers

Fig. 1(a) shows a 60 × 60 μm^2 AFM topography map of GaN surface. The surface is composed by step terraces and nano-dimension TDs (not visible in topography map Fig. 1a), and up to micro-size V-shape defects. Step terraces are typical of smooth MOCVD Ga-polar GaN surface [38]. The root mean square (rms) roughness, as measured over a scan area of 5 × 5 μm^2, was ~0.35 nm. Fig. 1(b) is the corresponding C-AFM map obtained at a bias of +6 volts applied to AFM tip. Here, it can be noted that at every single V-shaped defect, a higher local conductivity is measured. In certain cases, the one-to-one correlation with the map in Fig. 1(a) is not very clear, which is only due to lack of contrast in the topography image. Local current at the V-defect facets increases up to a maximum of 500 pA, while the background current at the same applied bias in defect-free area is lower than 56 pA (close to the minimum detectable current). Since the AFM tip-surface contact can be considered as metal–semiconductor Schottky junction with barrier height ϕ_B the current I in forward bias V depends exponentially on ϕ_B by the relation:

$$I = AA^{*} \exp\left(-\frac{q\phi_B}{kT}\right)\left[exp(\frac{qV}{nkT}) - 1\right] \quad (1)$$

with A contact area, A^* Richardson's constant, n diode ideality factor, k Boltzman constant, T temperature and q the electronic charge.

The current increment at the V-defects can be explained by the reduction of the surface barrier height at V-defect facets in comparison to the defect-free surface, as seen in the surface potential map in Fig. 1c, obtained on the same area of Fig. 1a. A significant reduction in the surface potential at each V defects, even up to a maximum of 1.2 V, is clearly visible. This value is 6–12 times higher than the change in surface potential due to charged dislocations in GaN reported in the literature [38]. This local decrease in the surface potential is directly related to the surface downward band bending and cannot be associated to negatively charged dislocations emerging at the V-defects, as negatively charged dislocations typically induce an upward band bending in *n*-type material [36]. Two observations can be pointed out. First, the reduction in Kelvin potential is independent of V-defect dimensions, which rules out any