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Observation of dislocation etch pits in epitaxial lateral overgrowth GaN by wet etching

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Abstract

This work investigates dislocation etch pits in epitaxial lateral overgrowth (ELO) GaN by wet etching. A mixture of H_2SO_4 and H_3PO_4 was used as an etching solution. SEM and AFM were employed to observe the surface topography. For the as-grown sample, SEM images show the flat, smooth surface without any pits or hillocks. After the chemical etching, hexagonal shaped etch pits were observed at the edge of ELO GaN. AFM observation of etched ELO GaN displayed high densities of etch pits clustered in the "window" region and the coalescent line of two growing fronts. In contrast, the overgrowth region was nearly free of etch pits. Moreover, we observed that different sizes of etch pits dominated in "window" region and coalescent region. This implied different types dislocations dominated in these regions. © 2002 Elsevier Science Ltd. All rights reserved.

1. Introduction

Owing to a significant lattice mismatch between GaN and sapphire, GaN films grown on sapphire contain dislocation densities of 10^8–10^{10} cm^{-2} [1]. Dislocations are believed to limit the fabrication of high quality nitride-based devices with a longer lifetime and high performance. Lester et al. indicated that nitride-based light emitting diodes have great tolerance of high dislocation densities [1]. This implies that dislocations in these diodes do not act as efficient non-radiative recombination centers. However, high dislocations densities still influence the device characteristic and performance. Kozodoy et al. found that reverse bias leakage current of p–n junction diodes was three orders of magnitude larger than that in the high dislocation density region [2]. Shiojima et al. revealed that dislocations caused a small area with low Schottky barrier height at the interface [3].

Selective area growth and epitaxial lateral overgrowth (ELO) are effective methods for the reduction of dislocation densities in GaN films. Furthermore, this reduction improves the device performance. Nakamura et al. increased the lifetime of their laser diode from 35 to 1150 h by the ELO method [4]. There are many reports investigating defects in ELO GaN by transmission electron microscopy (TEM) [5,6]. However, there have been few reports which investigated dislocations in ELO GaN by wet chemical etching.

In this work dislocation etch pits of ELO GaN are observed by wet chemical etching. Wet chemical etching is a conventional method for evaluating dislocations in an III–V compound semiconductor. There have been many observations of dislocation etch pits on GaN surface [7–9]. For example, Shiojima et al. [9] identified that the dislocation etch pit densities observed by atomic force microscope (AFM) were almost the same as the mixed dislocation densities observed by TEM. They concluded that a better approach for evolution of mixed dislocation is the combination of wet chemical etching and AFM. In this study, the surfaces of ELO GaN were etched with hot H_2SO_4/H_3PO_4 mixture and then observed by scanning electron microscopy (SEM) and AFM.

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2. Experiments

The ELO GaN was grown on 2 μm GaN "template substrate" by MOCVD in an EMCORE D75 reactor system. The growth pressure was carried out at 100 Torr, and the temperatures were in the range of 1010–1050 °C. Trimethylgallium (TMGa), and ammonia (NH_3) were used as Ga and N sources, respectively. Hydrogen was used as a carrier gas. The GaN "template substrates" were grown by MOCVD on a (0 0 0 1) sapphire substrate. Then a 100 nm thick SiO_2 film was deposited on the GaN "template substrate" by plasma enhancement chemical vapor deposition. Standard photolithography and wet chemical etching were applied to define the stripe patterns. The stripes were oriented perpendicular to the direction of [11–13]. The fill factor (ratio of open width to pattern period) of this pattern was 0.5. To reveal the dislocation etch pits, the chemical etching was carried out in a mixture of H_2SO_4 and H_3PO_4 with a ratio of 1:3 at 250 °C for 15 min. The shape and dislocation of etch pits were observed by SEM and AFM.

3. Results and discussion

Fig. 1(a) illustrates the SEM image of the as-grown ELO GaN surface. Flat and smooth surfaces without any pits or hillocks are observed in the as-grown ELO GaN. Fig. 1(b) displays the SEM image of the etched sample. After chemical etching, some well defined hexagonal etch pits around the edge of the ELO GaN wafer were discovered (Fig. 1(b)). While in the other area, nearly pits free was presented, as shown in Fig. 1(c). The etch pits are crowded in the coalescent line between the two growing fronts. This crowding indicates that incomplete coalescence of the two growing fronts causes poor chemical hardness. Therefore the etchant would etch the crystal in this region. Furthermore, no etch pits were observed in the other regions. This could be tentatively suggested that the size of etch pits in the areas is too small to be detected. The diameter of the hexagonal etch pits at the edge is approximately 1.3 μm. The densities of etch pits in the overgrowth region (coalescent line) are approximately $5 \times 10^{3}\ \mathrm{cm}^{-1}$ (with a linear density).

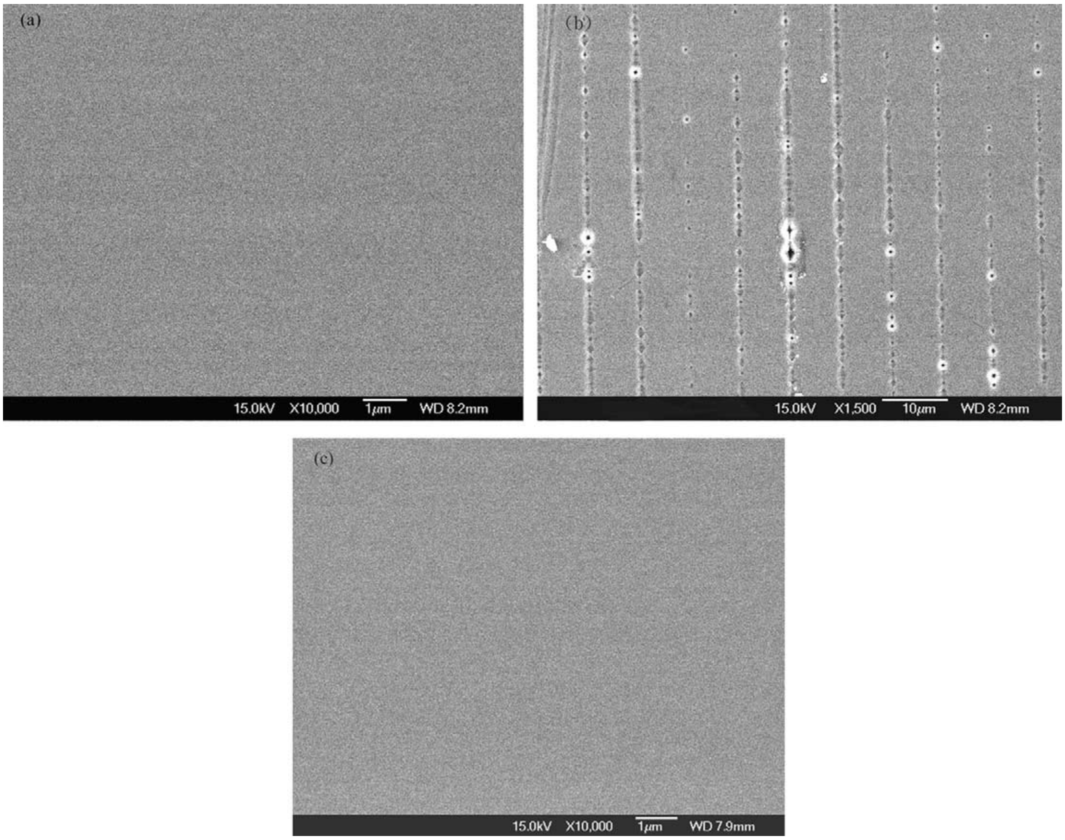

Fig. 1. SEM images of (a) as-grown, (b) the edge of chemical etched ELO GaN and (c) the center region of chemical etched ELO GaN. Some hexagonal etch pits are observed around the edge of ELO GaN surface.

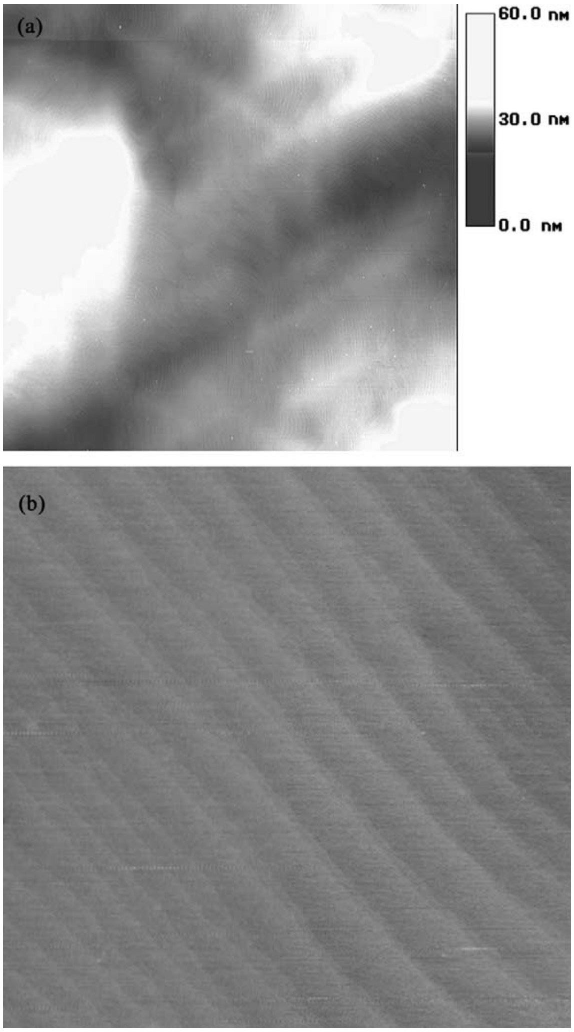

Fig. 2. Typical AFM images of (a) 30 × 30 μm^2 and (b) 2 × 2 μm^2 areas for as-grown ELO GaN. Wavy structures without step terminations or dark spot are seen.

Fig. 2(a) illustrates the typical AFM image of the sample shown in Fig. 1 over 30 × 30 μm^2 areas. The AFM observation over 30 × 30 μm^2 displays irregular undulations. The roughness of this undulation is approximately 70–90 nm. Fig. 2(b) illustrates the AFM image of the same sample in Fig. 1 over 2 × 2 μm^2 areas. A stepped and wavy structure with an average step height of 0.3 nm is observed in AFM image. No dark spots or step terminations in AFM observations can be detected. This indicates that the ELO GaN is free of mixed dislocation [5,10].

Fig. 3 shows an AFM image revealing etch pits on the ELO GaN surface after wet chemical etching. Two types of etch pits exist in 30 × 30 μm^2 areas. These pits are concentrated in the "window" region and the coalescent line. When the AFM observed areas are enlarged to 2 × 2 μm^2 two kinds of hexagonal pits are observed with diameters of approximately 90–115 nm, as shown in Fig. 4. The densities of the larger pits and the smaller ones are approximately 7×10^7 cm^{-2} and 9×10^8 cm^{-2}, respectively. We find that the densities of

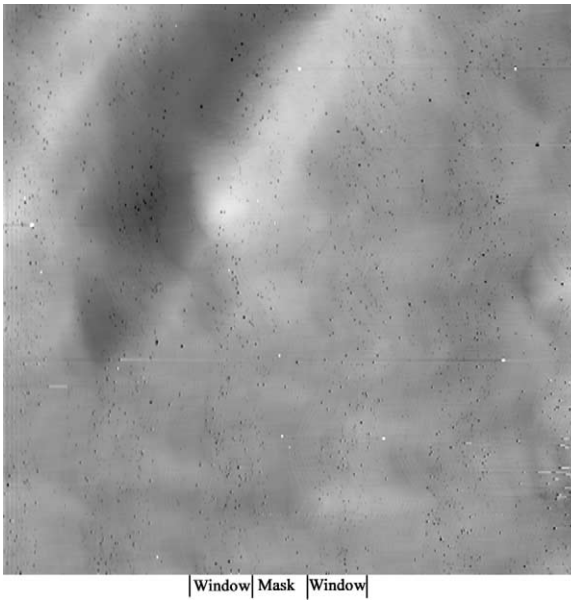

Fig. 3. Typical AFM images of 30 × 30 μm^2 areas for chemical etched ELO GaN. Etch pits clustered at the "window" region and along the coalescent line. In the overgrowth region was nearly free of etch pits.

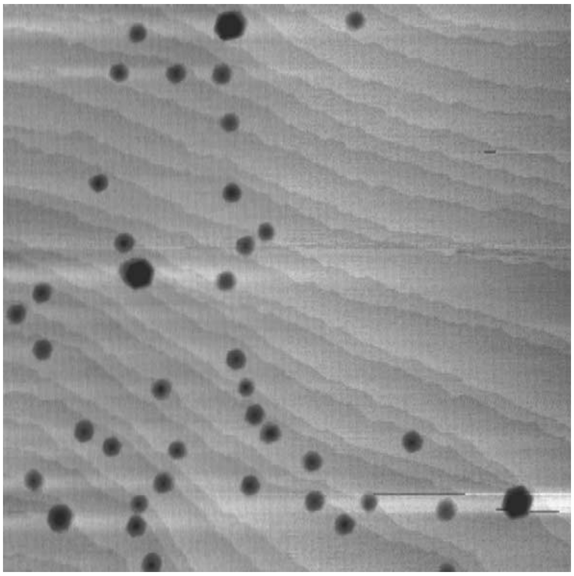

Fig. 4. Typical AFM images of 2 × 2 μm^2 areas for chemical etched ELO GaN surface. Two kinds of hexagonal pits were observed with diameters of approximately 115 and 90 nm.

the small pits are one order higher than that of the large pits. A high-density etch pits is observed in the "window" region as shown in AFM images. These etch pits might be produced by threading dislocations propagating to the top surface of ELO GaN, which originate from the GaN template substrate [6,8]. In contrast, the overgrowth region contains fewer etch pits. Only a few etch pits are observed along the coalescent line between the two growing fronts in the overgrowth region. Liliental-Weber et al. [6] reported that the dislocations in the homoepitaxial area (window region) would bend toward the overgrowth region. They also discovered that the lateral overgrowth region has planar defects and some helical and bent dislocation, but some of the bent dislocations are terminated near the SiO_2 mask. However, these defects (including the planar defects, helical and bent dislocation) are parallel to the surface, and cannot be identified by wet chemical etching. Furthermore, etch pits along the coalescent line also correspond to the plane-view and cross-section TEM images reported by Liliental-Weber et al. [6]. Misorientation between the two overgrowth fronts meeting cause a high density of mixed and edge types of dislocation in these areas. The etch pit distributions in our study also correspond to early reported TEM images [6,12].

Shiojima et al. demonstrated three different size pits on MOCVD grown GaN by AFM in their studies [9]. They concluded that the large and medium sized etch pits were created by the same dislocation. Hino et al. also revealed three types etch pits in their report [11]. However, they determined these pits correspond to three different types of threading dislocations. Our investigation presents only one kind of etch pits at the edge of the ELO GaN in SEM images. By comparing the pit sizes and pit shapes, these etch pits at the edge are similarly to the "β" type etch pits reported by Hino et al. That is, incomplete coalescence of the two growing fronts might result in mixed dislocations [11]. Moreover, the AFM observations show two kinds of pits in the "window" region, but fewer small size pits are observed in the coalescent region. This therefore implies that different kinds of dislocation dominate in the window and coalescent regions. Hansen et al. suggested the origin of large size pits were both pure edge and edge-screw mixed dislocations, and small size pits were open-core dislocations (or nano-pipes) [13]. Shiojima et al. reported that etch pits are a mixed dislocation. The different sizes of etch pits might be due to either the difference of Burgers vectors or dislocation bunching [9]. Hino et al. demonstrated three different size of etch pits correspond to three types of threading dislocations (i.e. edge, mixed and screw dislocations). As discussed previously, the densities of large and small pits in our investigation are similar to the "β " and "γ " type pits reported by Hino et al. [11]. Therefore, we suggest that both mixed and edge dislocations dominate in the "window" region and mixed dislocations dominate along the coalescent line. The origin of these etch pits, in our study, in particular areas is still unclear. Further studies are required to identify these etch pits.

In summary, this work has investigated dislocations in ELO GaN, by wet chemical etching. For the as-grown sample, SEM images reveal flat and smooth surfaces with no pits or hillocks. After chemical etching, a few hexagonal pits were observed along the coalescent line in the overgrowth region at the edge of the ELO GaN. AFM observations of etched ELO GaN displayed high densities of etch pits clustered in the "window" region and the coalescent line. Furthermore, different sizes of etch pits dominated in "window" region and coalescent region, implying different types of dislocations are dominant in these regions.

Acknowledgements

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