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Nanoindentation characterization of GaN epilayers on A-plane sapphire substrates

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ABSTRACT

Gallium nitride (GaN) epilayers was deposited on *a*-axis sapphire substrate by means of metal-organic chemical vapor deposition (MOCVD) method. The GaN epilayers has been investigated in their repetition pressure-induced impairment events from nanoindentation technique and, the relative deformation effect was observed from atomic force microscopy (AFM). From the morphological studies, it is revealed that none of crack and particle was found even after the indentation beyond the critical depth on the residual indentation impression. The 'pop-in' event was explained by the interaction of the deformed region, produced by the indenter tip, with the inner threading dislocations in the GaN films. Pop-in events indicate the generation and motion of individual dislocation, which is measured under critical depth and, no residual deformation of the GaN films is observed.

1. Introduction

Gallium nitride (GaN) is a highly attractive material of groups III–V nitride semiconductors, because it is great potential for the development of optoelectronic devices in blue/green light emitting diodes (LED), semiconductor lasers, and optical detectors. The fabrication of optoelectronic devices based on GaN epilayers requires to be understood in the mechanical properties as well as its optical and electrical properties [1–4], that is to say, the mechanical damages of GaN epilayers, such as film cracking and interface delamination caused by thermal stresses, chemical–mechanical polishing, usually suppress the processing yield and application reliability of microelectronic devices.

The heteroepitaxy using typical substrates (*e.g.*, sapphire) with high lattice mismatch is discussed in early report. GaN epilayers on sapphire substrates exhibits large lattice mismatch (about 14.5%) causing in-plane tensile strain in the GaN layers. In fact, the most common orientation of sapphire used for GaN is the *c*-axis sapphire. And, the lattice mismatch of the GaN films on *a*-axis $(1\,1\,\bar{2}\,0)$ sapphire is less (2%) than that on *c*-axis $(0\,0\,0\,1)$ sapphire (13.9%), which promises for excellent quality of GaN growth with improved surface morphology [5]. Here, compared to bulk single crystals, the deformation properties of thin films can serve strongly correlate to the geometrical dimensions and the materials defect-structure. The misfit dislocations at the interface play an important role such as carrier mobility, and luminescence efficiency. In this respect, nanoindentation has proven to be a powerful technique for probing the information on the mechanical properties of GaN epilayers with characteristic dimensions in the sub-micron regime, such as hardness and Young's modulus [6,7]. During indentation load of GaN epilayers, the nanoindentation-induced discontinuity (so-called 'pop-in') in the load–displacement curve has been observed in elsewhere investigation. The slip band movement [8,9] and dislocation nucleation mechanism [6,10] have been proposed to explain this 'pop-in' event. Most of the studies were carried out on *c*-axis GaN epilayers or bulk single crystals [11]. Comparatively, the GaN epilayers was observed in their mechanical damages, however the detail work in the *a*-axis GaN epilayers is still not yet unclear.

In this article, significant investigation of the mechanical characterizations on GaN epilayers is motivated. The pressure-induced impairment of GaN epilayers/*a*-axis sapphire substrate has been investigated by using nanoindentation technique and atomic force microscope (AFM).

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2. Experimental details

GaN epilayers was grown on a-axis $(1\,1\,\bar{2}\,0)$ sapphire substrates by using the metal-organic chemical vapor deposition (MOCVD) method. To fabricate the GaN epilayers, a 10-nm-think AlN-buffer layer was first grown on $(1\,1\,\bar{2}\,0)$ sapphire substrate. Then, GaN epilayers with a thickness of nearly 2 μm was grown on top of the buffer layer by MOCVD at 1100 °C, using Triethylgallium (TEGa), trimethylaluminium (TMAl), and ammonia (NH_3) as the gallium, aluminium, and nitrogen, respectively.

The mechanical properties of GaN epilayers performed by a Nanoindentation system (Nano Indenter XP instrument, MTS Corporation, Nano Instruments Innovation Center, TN, USA) with Berkvich indenter tip, whose radius of curvature is 50 nm, were conducted under continuous contact stiffness measurement (CSM) technique. In CSM measurement, a series of continuous load–unload indents were carried out at the penetration depth of 15, 40, and 300 nm. Each indentation was separated by 30 μm to avoid possible interference between neighboring indents. Here, all indents were performed at room temperature. The analytic method developed by Oliver and Pharr was adopted to determine the hardness (H) and Young's modulus (E) of the GaN films from the load–displacement curves [12]. The thermal drift was kept below $\pm$0.05 nm/s for all indentations considered in this work. The nanoindentation-induced crack morphology of the GaN epilayers was examined by atomic force microscopy (AFM, Veeco D5000).

3. Results and discussion

The load–indentation depth data for the grown GaN thin film with 2 μm thickness on a-axis sapphire substrate is discussed, the multiple discontinuities during loading were clearly revealed in Fig. 1(a). The hardness and elastic modulus as a function of indentation depth can be obtained from the CSM measurements, as illustrated in Fig. 1(b) and (c), respectively. The hardness and elastic modulus of the GaN films/a-axis sapphire substrate are determined to be 15.9 $\pm$ 0.8 and 394.6 $\pm$ 12 GPa, respectively. Table 1 displays some of relevant data for comparison [8,13,18]. We believe that the discrepancies among the mechanical parameters obtained by various indentation methods are mainly due to the specific tip–surface contact configuration and stress distribution inherent in the different plane sapphire substrate. At beginning, we observe that the 'pop-in' event took place at a depth of 16 nm (at a critical load of 0.16 mN) and, the contact stress has been given to be 25.45 GPa (see the inset in Fig. 1(a)). The critical contact stress of the penetration depth in 16 nm is required to cause the initial 'pop-in' event in the a-axis GaN film. This case is relatively large with that of GaN film on c-axis sapphire substrates (23 nm; at a load of 0.48 mN; contact stress 22.5 GPa) [14]. The phenomenon is suggested that the low indentation load (0.45 mN) serves the Berkovich tip in the increasing stress on the film's surface than the Berkovich tip on the GaN film's surface. In fact, the data obtained from Berkovich diamond indenter in our result were also very large with that of spherical tip (140 nm; at a load of 30 mN; contact stress 9 GPa) as reported in the other study [13]. Therefore, the initial sudden displacement discontinuity generally accompany with maximum shear stress generated under the indenter and, is more than that of the remaining other 'pop-ins'. We also observe that the corresponding indentation depth versus hardness data from the hardness value increased highly about 36 GPa before the 'pop-in' event at 16 nm (see the inset in Fig. 1(a)). The fact was attributed from the sharper tip that induces plasticity at a shallow indentation depth. That is to say, the hardness values can be calculated accurately [8]. It is suggested that the 'pop-in' events partially induce from the heteroepitaxial thin film, which can also be attributed to the inner-defects of GaN films.

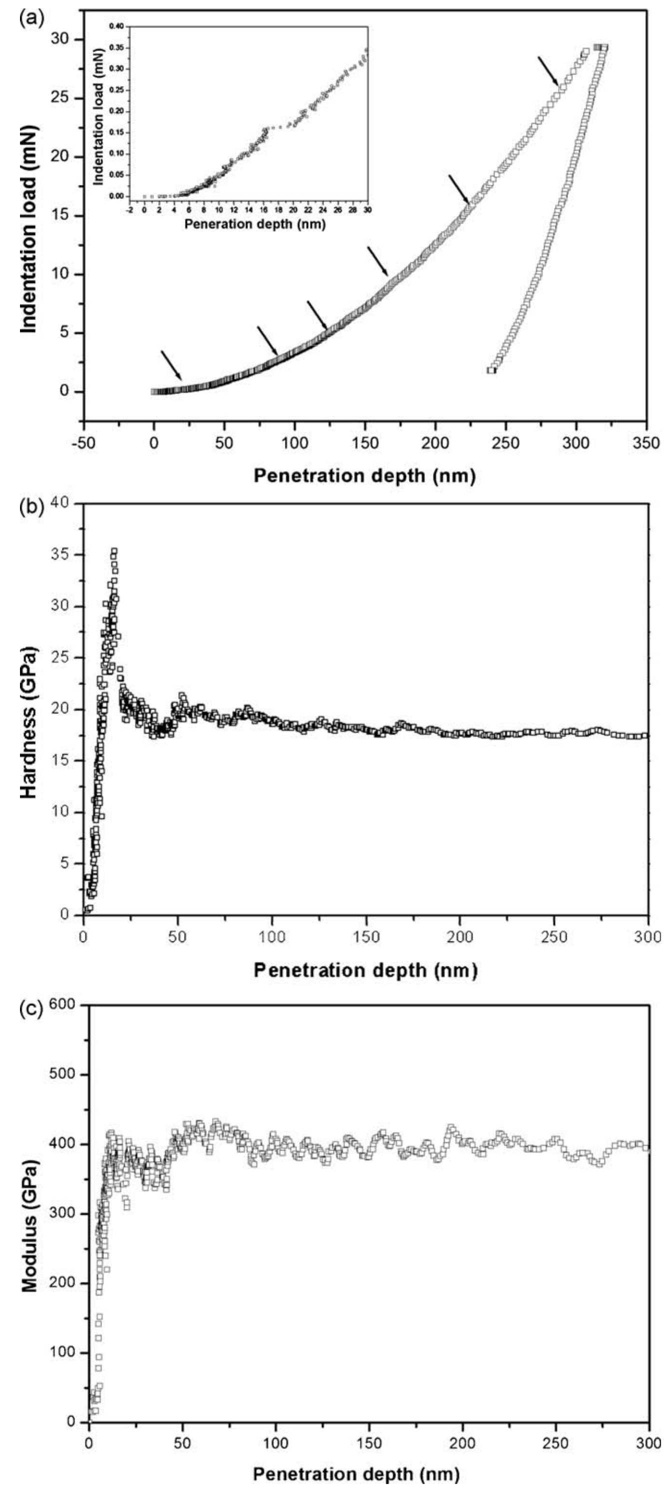

Fig. 1. Nanoindentation test results: (a) a typical load–displacement curve (inset: the typical load–displacement curve illustrates the drastic variation at the critical depth of first 'pop-in' at 16 nm); (b) hardness–displacement curve; (c) modulus–displacement curve for GaN films/a-axis sapphire substrate.

Evidently, due to the soft GaN film grown epitaxially on a hard sapphire substrate, it is easy to find that the differences in lattice parameter and thermal expansion coefficients between the film and substrate. This may introduce a misfit strain at the interface, and then the strain is released by generating threading dislocations in the GaN epilayers [15,16]. In the experiment of indentation loading, the deformed region was punched out on the top edge from the indenter tip; the threading dislocation may be to cause a

Table 1
Hardness and elastic modulus of GaN films obtained from various measurement methods.

	Average hardness (GPa)	Average modulus (GPa)	Indenter tip
A-plane GaN[a]	15.9 ± 0.8	394.6 ± 12	Berkvich
C-plane GaN [13]	22.5		Berkvich
C-plane GaN [18]	19 ± 11	286 ± 125	Berkvich
C-plane GaN [8]	13.4	233	Spherical

[a] This study.

sudden increase in plasticity, therefore the sudden discontinuity in the load–indentation depth curve is showed. Navamathavan et al. [14] conducted that the threading dislocation in the GaN films/*c*-axis sapphire substrate can suddenly propagate after acquiring threshold energy from the deformed region. This is the reason why the multi-'pop-ins' of the GaN epilayers is occurred. However, some of the threading dislocations terminate as they yet to reach the film surface [17]. At the same time, the GaN films/*a*-axis sapphire substrate can be interacted by indenter tip to appear deformed zone, the threading dislocations then excursion into films through certain depths, afterward causing the 'pop-in' event. It is conjectured that the mechanism of the event is caused by slip bands [8,9,13], and/or dislocation nucleation [6].

In Fig. 2(a), the AFM image of the residual indentation mark is revealed that none of crack and particle even occurred after the indentation depth. From the section view of the indentation mark, Fig. 2(b) shows the residual volume from the edge of indentation. Dislocation-induced 'pop-in' event tends to associated with two distinct deformation behaviors before (pure elastic behavior) and after (elastoplastic behavior) the phenomenon. It is speculated that the multiple 'pop-in' events are revealable over the indentation load and penetration depth. We suggested that the initial 'pop-in' event is owing to the role of dislocation. Therefore, this could be due to the occurrences of a discontinuity in the load–indentation depth curve. For this case, the investigation of initial deformation is measured under critical depth, which films deformed elastically and no residual deformation is observed (Fig. 3(a)). At the same time, while the indentation is stopped after the exactly 'pop-in'

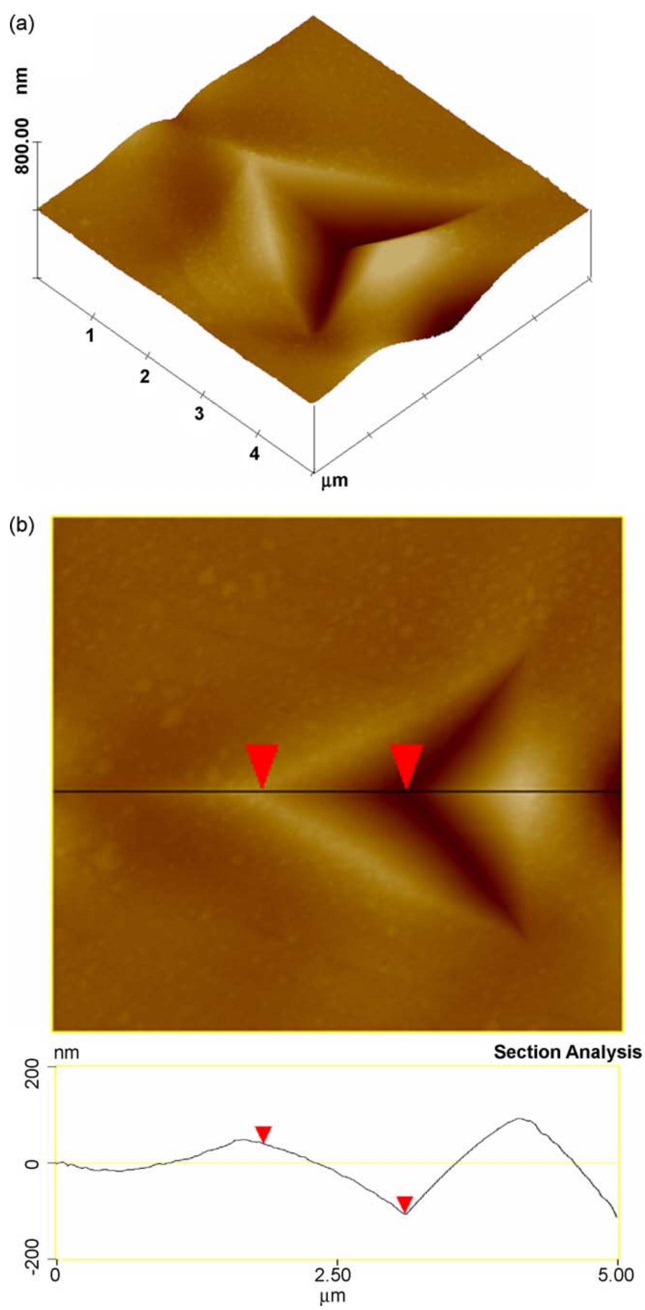

Fig. 2. (a) The AFM image of the residual indentation mark is revealed that none of crack and particle even occurred after the indentation depth; (b) the residual volume from the edge of indentation.

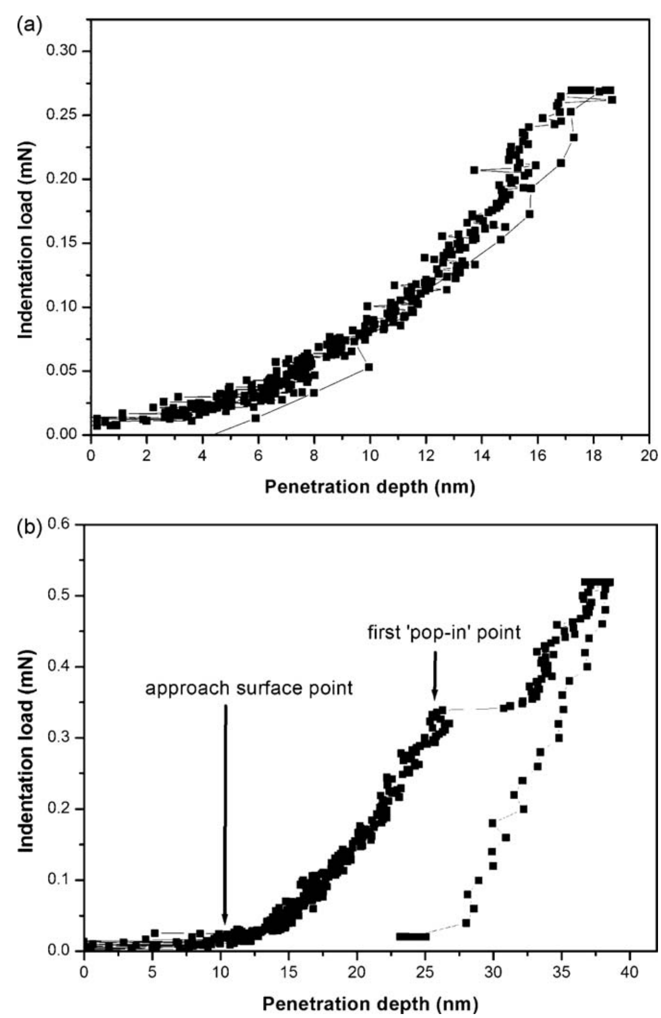

Fig. 3. (a) A typical load–displacement curve for just before the critical 'pop-in' depth (14 nm) shows the elastic recovery; (b) a typical load–displacement curve for just after the critical 'pop-in' depth shows sudden discontinuity and produced residual impression of about 26 nm.

distance, the residual mark depth is observed as shown in Fig. 3(b). This leads to larger deviations in the penetration depth versus indentation load curves. We suggested that the 'pop-in' event of the GaN films could be contributed from an entirely plastic deformation case. The present results are similar with the work that the GaN films can be elastically flexure before the 'pop-in' event [14].

Above-mentioned results and discussion, the primary deformation mechanism of the GaN films is due to dislocation nucleation and propagation along the easy slip systems. The mechanisms of responsible for the dislocation recovery appears to be associated with the activation of dislocations sources bring by loading-unloading cycle of the GaN films. The plastic deformation prior to loading–unloading cycle is associated with the individual movement of a small number of new nucleation, large shear stress is quickly accumulated underneath the indenter tip. When the local stress underneath the tip reaches at high level cycles, a burst of collective dislocation movement on the slip systems is activated, resulting in a release of local stress [14]. The extensive interactions between the dislocations slipping along the GaN surface, therefore, confined the slip bands resulted in a 'pop-in' event due to the deformed and strain-hardened lattice structure.

4. Conclusion

We employed a combination of nanoindentation and AFM techniques to investigate the contact-induced deformation behaviors of the GaN films/*a*-axis sapphire substrate. The deformation mechanisms of the GaN films result in a 'pop-in' event during loading-unloading cycle, especially lead to deviations in the penetration depth versus indentation load curves. The punching out of the threading dislocations beneath the deformed region may contribute to the 'pop-in' event, which has been shown as one of the mechanisms responsible for the plastic deformation of the GaN films.

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