‘Pop-in’ phenomenon during nanoindentation in epitaxial GaN thin films on c-plane sapphire substrates

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Abstract

Nanoindentation studies have been carried out on undoped and doped epitaxial GaN thin films with different thickness (1–4 μm) were grown epitaxially on c-plane sapphire substrate by metalorganic chemical vapor deposition (MOCVD). Multiple discontinuities (so-called ‘pop-in’ events) were observed in the load–indentation depth curve irrespective of the thickness as well as the doping condition. Atomic force microscopy (AFM) studies on the residual indentation impression revealed no micro-cracks even after the indentation beyond the critical depth. The physical mechanism responsible for the ‘pop-in’ was explained by the interaction of the deformed region, produced by the indenter tip, with the pre-existing threading dislocation in the epitaxial GaN thin films.

Keywords: Nanoindentation; Pop-in; GaN film

1. Introduction

GaN and its related epitaxial thin films have received much attention in recent years for their proven and potential ability in optoelectronic and high power microwave device applications [1,2]. A successful fabrication of optoelectronic devices based on epitaxial thin film requires an understanding of the mechanical properties of this material as well as its optical and electrical properties. The formers are of special concern since heteroepitaxy using typical substrates (e.g., sapphire) involves high lattice mismatch. Additionally, compared to bulk single crystals, the deformation properties of thin films can differ because they strongly correlate to the geometrical dimensions and the materials defect-structure.

GaN epitaxial thin films on sapphire substrates exhibit large lattice mismatch (about 14.5%) causing in-plane tensile strain in the GaN layers. The difference in thermal expansion coefficients between the materials also cause wafer bowing when cooled down from the growth temperature. As misfit dislocations at the interface are detrimental to device performance (carrier mobility, luminescence efficiency, etc.), it is of interest to study the mechanical properties through the nanoindentation technique.

There have been some reports on the nanoindentation studies of bulk single crystal and epitaxial thin films of GaN by different groups [3–10]. During indentation loading of GaN film, a discontinuity (so called ‘pop-in’ or “burst”) in the load–displacement curve has been observed in some studies [5–10]. Moreover, twinning [5], slip band movement [7–9] and dislocation nucleation [6,10] mechanisms have been proposed to explain this ‘pop-in’ event. However, the occurrence of ‘pop-in’ event observed in some of the published work is still a matter of debate and remains unclear. So, the purpose of this article is to report how this ‘pop-in’ phenomenon occurred in these heterostructures and also correlate the possible reason for this physical mechanism.

2. Experimental

All GaN thin films were grown on c-plane sapphire substrate by MOCVD system (Emcore D125) with a vertical high-speed rotating disk reactor. Prior to
transferring the sapphire substrate to the growth chamber, the load lock chamber 
was evacuated up to $10^{-6}$ Torr to remove O$_2$ and H$_2$O. A thermal cleaning of 
the sapphire substrate was performed with high purity H$_2$ gas at 1040°C. The 
growth involved with deposition of InGaN/GaN buffer layer at low temperature 
(500–600°C) with a thickness of 300 Å followed by the growth of GaN thin 
films at a temperature of 1050°C. The GaN thin films with thickness ranging 
from 1 to 4 μm were grown. A detailed description of growth procedures and 
structural characterization was provided elsewhere [11].

The deformation behavior of the samples was performed using the Nanoin-
denter XP (MTS Nano Innovation Center, Oak Ridge, TN) with a dynamic 
contact module (DCM) head and Berkovich diamond indenter with a tip radius of 
approximately 80 nm tip. The nanoindentation system was fully calibrated using 
the corresponding plot of hardness as a function of indentation depth illustrates the drastic variation at the 
critical depth of first ‘pop-in’ at 23–26 nm.

Fig. 1 shows the load–indentation depth data for the undoped GaN thin film 
with 3 μm thickness on sapphire substrate. This Fig. 1 clearly reveals multiple discontinuities (so-called ‘pop-
in’ events) during loading. First ‘pop-in’ event took place at a depth of 23 nm (at a critical load of 0.48 mN). From the 
load–indentation depth data, the contact area at the beginning 
of the first ‘pop-in’ has been calculated to be 21,248 nm$^2$ for 
the Berkovich diamond indenter. The contact stress has conse-
quently been calculated to be 22.5 GPa. So, this critical contact 
stress is required to cause the first ‘pop-in’ event in the GaN 
film at a penetration depth of 23 nm, which is 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 [9]. We believe that the 
Berkovich tip produces much greater stress on the film’s surface 
even for the low indentation load (0.45 mN) than the spherical 
tip and hence the ‘pop-in’ event occur at very shallow depth of 
the GaN thin films. The first sudden displacement discontinuity 
generally occurs when maximum shear stress generated under 
the indenter is of the order of theoretical shear strength [12]. This high local stress ostensibly causes homogeneous nucleation of 
threading dislocations beneath the indenter surface, producing a sudden displacement discontinuity. However, the threshold 
stress required for occurrence of the first ‘pop-in’ event is more 
than that of the remaining other ‘pop-ins’. Evidently, in the cor-
responding indentation depth versus hardness data the hardness value is increased drastically (to about 37 GPa) before the ‘pop-
in’ event at 23 nm and after that decreased rapidly to a constant 
value for further increase in indentation depth (see the inset in 
Fig. 1). Generally, the indenter tip’s bluntness causes the depth of full plasticity to be too deep to discern any difference in the 
films. But, for the sharper tip induces plasticity at a shallow 
indentation depth and the hardness values can be determined 
more accurately. It is worth noting that further experiments have 
been carried out to confirm the reproducible behavior of this 
phenomenon in these epitaxial thin films. Interestingly, all 
other films irrespective of their thickness and doping condi-
tion showed this first ‘pop-in’ event at similar depths (namely 
23–26 nm) and so on. This critical depth of first ‘pop-in’ in 
GaN thin film is higher than that of epitaxial ZnO thin films 
on sapphire substrates (‘pop-in’ depth 13–16 nm) as we studied 
earlier [13]. We believe that this variation in the critical depth of 
‘pop-in’ event to occur is associated with the percentage of 
lattice mismatch with the substrate ($f_{\text{GaN}} = 15\% < f_{\text{ZnO}} = 18\%$). Further, we observed multiple ‘pop-in’ events at certain depths 
when the indentation depth was increased up to 300 nm and no ‘pop-out’ (reverse thrust) was observed during the unloading 
phase.

One likely explanation for the ‘pop-in’ events in the case 
of heteroepitaxial thin film can be attributed to the pre-existing 
defects concomitantly within these GaN films. It is well known 
that when soft film is grown epitaxially on a hard substrate, the 
differences in lattice parameter and thermal expansion coeffi-
cients between the film and substrate introduce a misfit strain 
at the interface. This strain is then released as threading dislo-
cations in the film [14,15]. During indentation loading, when 
the deformed region produced beneath the indenter tip punches 
out the top edge of the threading dislocation is likely to cause a sudden increase in plasticity, which could be expressed as 
a sudden discontinuity in the load–indentation depth curve. In 
other words, the threading dislocation in the film could sud-
denly propagate soon after acquiring threshold energy from 
the deformed region, showing a ‘pop-in’. To account for the 
multi ‘pop-ins’, some of the threading dislocations terminate 
before they reach the film surface [16] when the deformed zone 
beneath the indenter tip interact with these subsequent thread-
ing dislocations during the excursion into film, thus causing the 
‘pop-in’ events at certain depths in the thin film accordingly. 
We infer that this physical mechanism, rather than slip bands 
[7–9], dislocation nucleation [6,10] is the main criterion for the 
‘pop-in’ phenomenon in the epitaxial GaN thin film. In addi-
tion, amplitude-mode AFM image on the residual indentation 
impression revealed no micro cracks even after the indentation 
beyond the critical depth (Fig. 2). Therefore, the origin of the 
‘pop-in’ is due to dislocation based phenomenon.
Careful measurements are carried out below the critical depth showed that all the films deformed elastically and no residual deformation is observed, as shown in the load–indentation depth data in Fig. 3, the loading and unloading parts of the curve are perfectly superimposed. Our present results are closely matched with the studies carried on AlGaN thin films which elastically flexure prior to the ‘pop-in’ event [10]. Moreover, when the indentation is stopped just after the ‘pop-in’, the residual impression depth observed as shown in Fig. 4. The ‘pop-in’ event could thus be considered as an entirely plastic deformation process.

4. Conclusions

We have investigated the deformation behavior of the epitaxial GaN thin films using nanoindentation studies. Multi ‘pop-in’ events have been observed in the load–indentation depth curve. The critical stress produced beneath the Berkovich indenter was main criterion for the first ‘pop-in’ event at very shallow depths in the GaN thin film. The physical mechanism responsible for the multi ‘pop-in’ events were explained by the punching out of the threading dislocation beneath the deformed region produced by the indenter tip at corresponding depths in the GaN thin film. In addition, the critical depth for the first ‘pop-in’ to occur depends on the lattice mismatch of the epitaxial thin film. AFM studies on residual impression did not show any micro-cracks in and around the indentation region. Finally, indentation below the critical depth showed that the surface flexes elastically and beyond which interaction with pre-existing threading dislocation causing plastic deformation.

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References