Mechanical response of GaN film and micropillar under nanoindentation and microcompression

T. H. Sung, J. C. Huang, J. H. Hsu, and S. R. Jian

1Department of Materials and Optoelectronic Science, Center for Nanoscience and Nanotechnology, National Sun Yat-Sen University, Kaohsiung 804, Taiwan
2Department of Materials Science and Engineering, I-Shou University, Kaohsiung 840, Taiwan

(Received 7 August 2010; accepted 6 October 2010; published online 26 October 2010)

The mechanical properties of GaN are examined by microcompression. The Young’s modulus and compressive yield stress in microscale are directly measured to be ~226 and 10 GPa, comparable to the modulus (~272 GPa) and hardness (15 GPa) measured by nanoindentation. The Raman spectrum measurements and transmission electron microscopy observations reveal that the residual stress in deposited film can be largely released in the form of micropillar. Upon microcompression, the strain energy is basically stored by dislocation and defect accumulation, with minimum residual stress regeneration. The small bending of the c-axis of the GaN micropillar upon compression would affect its optical performance. © 2010 American Institute of Physics. [doi:10.1063/1.3506498]

The wurtzite GaN has become a potential material due to its strong chemical and mechanical stability and high band gap of 3.2–3.4 eV. Defectless epitaxy single crystal GaN can be used as the core material on the high power laser. One gap of 3.2–3.4 eV. Defectless epitaxy single crystal GaN can be used as the core material on the high power laser. One gap of 3.2–3.4 eV. Defectless epitaxy single crystal GaN can be used as the core material on the high power laser. One gap of 3.2–3.4 eV. Defectless epitaxy single crystal GaN can be used as the core material on the high power laser. One gap of 3.2–3.4 eV. Defectless epitaxy single crystal GaN can be used as the core material on the high power laser. One gap of 3.2–3.4 eV. Defectless epitaxy single crystal GaN can be used as the core material on the high power laser. One gap of 3.2–3.4 eV. Defectless epitaxy single crystal GaN can be used as the core material on the high power laser. One gap of 3.2–3.4 eV.

So far, there have been some limited reports addressing the macroscopic, microscopic, or nanoscaled mechanical responses of the bulk or thin film crystalline GaN by using macroscaled or nanoscaled indentation testing. But due to the difficulty in fabricating high quality bulk single crystal GaN, the direct uniaxial compression results are still seldom reported. The micropillars prepared in this study are measured 1 μm in diameter and around 2.5 μm in pillar height, or an aspect ratio of about 2.5. Due to the FIB beam profile, there is a taper angle of the micropillar, typically around 2.8°. GaN damaged layers in the current FIB-milled 1 μm micropillars from the perpendicular and side radiation are estimated to be ~25 and 3 nm, and considered to be negligible. The micropillars are loaded in uniaxial compression by using the flat-punch Berkovich tip in a commercially available nanoindentation system (MTS Nano indenter XP) with the continuous stiffness measurement (CSM) function. The tests are run under the constant displacement rate mode at strain rates from 10⁻² to 10⁻⁴ s⁻¹. For tracing the mechanical response of the pillar at different straining stages, the preset stop displacement was set to be 100, 150, and 200 nm, representing the mostly elastic, starting plastic, and fully plastic conditions. The Young’s modulus and hardness of the tapered micropillars are corrected by the method described previously. Nanoindentation was also preformed on the same GaN film to reduce Ga ion damage effect on the GaN pillar surface.

The micropillars prepared in this study are measured 1 μm in diameter and around 2.5 μm in pillar height, or an aspect ratio of about 2.5. Due to the FIB beam profile, there is a taper angle of the micropillar, typically around 2.8°. GaN damaged layers in the current FIB-milled 1 μm micropillars from the perpendicular and side radiation are estimated to be ~25 and 3 nm, and considered to be negligible. The micropillars are loaded in uniaxial compression by using the flat-punch Berkovich tip in a commercially available nanoindentation system (MTS Nano indenter XP) with the continuous stiffness measurement (CSM) function. The tests are run under the constant displacement rate mode at strain rates from 10⁻² to 10⁻⁴ s⁻¹. For tracing the mechanical response of the pillar at different straining stages, the preset stop displacement was set to be 100, 150, and 200 nm, representing the mostly elastic, starting plastic, and fully plastic conditions. The Young’s modulus and hardness of the tapered micropillars are corrected by the method described previously. Nanoindentation was also preformed on the same GaN film to reduce Ga ion damage effect on the GaN pillar surface.
by using the Berkovich tip under the CSM constant displacement rate mode at a strain rate of $10^{-3}$ s$^{-1}$, from which the elastic modulus and hardness at the steady region within 400–600 nm in depth can be extracted for comparison.

Micro-Raman was conducted by a mode-built confocal microspectroscopic system. The micro-Raman spectra are then fitted by Gaussian distribution curve. The $E_2$ peak shifting can be indexed for characterizing the residual stress, and the full width at half maximum (FWHM) of the $E_2$ peak can be used to indicate the crystal quality and defect concentration. The microstructure of the GaN epitaxial layer is examined by using the Tecnai F20G2 field emission gun TEM operated at 200 kV. The high quality TEM foil samples are coated with carbon layer as a supporter, and then milled to foil thickness less than 100 nm by FIB.

Before microcompression, the basic properties of the as-deposited GaN film are measured by nanoindentation. The modulus and hardness are measured to be $\sim$272 GPa and 15 GPa, respectively.

Figure 1 depicts the load-displacement and transformed engineering stress-strain curves of the compressed 1 $\mu$m micropillars at a strain rate of $10^{-3}$ s$^{-1}$. The initial part of the stress-strain curve appears to be bended, or not fully elastic. This is a typical result from the uneven pillar top surface and pillar taper effect by the FIB milling. After deformation over 100 nm displacement, the micropillar would deform in its normal elastic behavior. There is an apparent strain burst occurring at $7.4 \pm 1$ mN in load. By using previous results, when the taper angle is small and $\sin \theta \sim \theta$, the extracted elastic modulus $E$ of 1 $\mu$m micropillars is $\sim$$226 \pm 17$ GPa. And the engineering yield stress for the first strain burst is calculated to be $\sim$$10 \pm 1$ GPa. Parallel compression tests were also conducted at $10^{-2}$ and $10^{-4}$ s$^{-1}$, there seems no significant influence from the strain rate over this range.

The micropillar yield stress can be compared with the maximum shear stress ($\tau_{\text{max}}$) extracted from nanoindentation testing. The maximum shear stress can be estimated by

$$\tau_{\text{max}} = 0.12 \left( \frac{P_{\text{crit}} E_s^2}{R^2} \right)^{1/3},$$

where $P_{\text{crit}}$ is the first pop-in load in nanoindentation, $R$ is the tip radius, $E_s$ is the actual modulus of the GaN material and it can be estimated from

$$\frac{1}{E_R} = \left( \frac{1 - \nu_m^2}{E_m} + \frac{1 - \nu_i^2}{E_i} \right),$$

where $E_R$ is the measured modulus by nanoindentation including machine and indenter effect (272 GPa for GaN nanoindentation), $E_m$ (1141 GPa) is the diamond tip modulus, $\nu_m$ (0.07) and $\nu_i$ (0.352) is the Poisson ratio for diamond indenter tip and GaN, respectively. The pop-in load in this study is $P_{\text{crit}}$$=1.2 \pm 0.3$ mN, corresponding to $\tau_{\text{max}}$ $\sim$6 GPa. This maximum shear stress can be transferred to a normal yield stress $\sim$12 GPa, which seems to be larger (by $\sim$20%) than the 10 GPa recorded by the 1 $\mu$m micropillars. This is acceptable since nanoindentation usually yield higher modulus and stress than microscale or bulk specimens due to the small volume constraint effect. Thus, the crystalline GaN appears to exhibit a yield stress in the neighborhood of 10 GPa in the microscale, and a even higher yield stress around 12 GPa in the nanoscale. There seems to be a pronounced size effect for GaN yield stress.

Raman spectroscopy can be one of tools to confirm if there is any significant residual stress before and after the first strain burst effect. There are three characteristic peaks giving the information of the lattice and optic relationship, namely, $E_2$, $A_1$(LO), and $E_1$(LO). The $E_2$ peak can be seen as a standard peak which is not sensitive to the incident optic axis. $A_1$(LO) and $E_1$(LO) represent two longitudinal optic (LO) phonon modes vibrating along the [0001] direction, and are forbidden while optical beam propagating perpendicular to the c-plane.

According to the calculation of Puech et al., the $E_2$ frequency would exhibit a blue shift of 2.43 cm$^{-1}$/GPa under hydrostatic stress. Figure 2 shows the representative Raman spectra for the as-deposited thin film, as-FIB-milled micropillars (not yet compressed), and micropillars compressed at $10^{-3}$ s$^{-1}$ to 100 and 200 nm preset displacement (before and after the first strain burst).

In comparison with the Raman $E_2$ peak center positions for the free standing (567.0 cm$^{-1}$) and as-deposited GaN (570.6 cm$^{-1}$) in Table I, the 3.6 cm$^{-1}$ blue shift indicates the compressive residual stress of 1508 MPa retained within the sputtered GaN thin film, using the results by Puech et al. After FIB milling into a GaN micropillar, from Fig. 2 and Table I, the Raman $E_2$ peak center position shifts back to 567.7 cm$^{-1}$, closer to the free standing GaN, reflecting that most compressive residual stress has been released and the retained residual stress drops down to less than 300 MPa. Upon compression loading on the micropillar to 100 and 200

<table>
<thead>
<tr>
<th>Specimen condition</th>
<th>$E_2$ peak (cm$^{-1}$)</th>
<th>$E_2$ FWHM (cm$^{-1}$)</th>
<th>Residual stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Free standing GaN*</td>
<td>567.0</td>
<td>-</td>
<td>0</td>
</tr>
<tr>
<td>As-deposited film</td>
<td>570.6</td>
<td>2.1</td>
<td>$\sim$1508</td>
</tr>
<tr>
<td>As-FIB-milled pillar</td>
<td>567.7</td>
<td>2.6</td>
<td>$\sim$280</td>
</tr>
<tr>
<td>Pillar compressed to preset 100 nm</td>
<td>567.5</td>
<td>5.7</td>
<td>$\sim$198</td>
</tr>
<tr>
<td>Pillar compressed to preset 200 nm</td>
<td>567.0</td>
<td>6.2</td>
<td>24</td>
</tr>
</tbody>
</table>

The pyramidal planes GaN micropillar reveals massive dislocation slip traces on shown in Fig. 3, the longitudinal section of the compressed GaN film, there is minimum defect with clean image. As thin film and the compressed micropillar. In the as-deposited micropillar, FWHM values are in the low range around 2.0 to 2.5 cm\(^{-1}\), reflecting the low defect contents at these stages. As the micropillars are deformed mostly elastically or fully plastically, FWHM values increase significantly to 5.7 and 6.2 cm\(^{-1}\). It means that, under compression testing, although the micropillars could release most of the residual stress from their surfaces, it still accumulate enormous defect concentrations. The increased defect amount is consistent with the TEM observations, shown in Fig. 3.

In addition to the E\(_2\) peak, information can be extracted from the A\(_1\)(LO) and E\(_1\)(LO) peaks. It can be seen from Fig. 2 that the as-deposited GaN film contains separate A\(_1\)(LO) (727 cm\(^{-1}\)) and E\(_1\)(LO) (735 cm\(^{-1}\)) peaks, while the deformed pillars exhibit merged single peak at 732 cm\(^{-1}\), termed as the quasi-longitudinal optic phonon (QLO) peak. This is usually related to the distortion of the c-axis of the GaN lattice, as seen by TEM.

TEM characterization has been done on the as-deposited thin film and the compressed micropillar. In the as-deposited GaN film, there is minimum defect with clean image. As shown in Fig. 3, the longitudinal section of the compressed GaN micropillar reveals massive dislocation slip traces on the pyramidal planes \(\{10\bar{1}\}\), oriented at an acute angle of \(\sim 62^\circ\) to the basal plane (0001), consistent with previous finding. \(^2\)

The selected area diffraction pattern in Fig. 3 shows there is a small bending angle (a few degrees) inside the pillars (especially for the top portion) after microcompression. The local bending of the GaN lattice lead to the appearance of the QLO peak at 732 cm\(^{-1}\), as a result of the small distortion of incident optic axis of backward scattering and in-turn the combination of A\(_1\)(LO) and E\(_1\)(LO) peaks. It also leads to the appearance of E\(_1\)(LO) (at 561 cm\(^{-1}\)) where TO stands for the transverse optic mode peak, which should be forbidden according to the selection rule from group theory. \(^1\) Finally, throughout the Raman and TEM characterizations, there is no evidence of recrystallization, cracking, or phase transformation in the compressed GaN micropillars.

In summary, this study presents the first data on the micro-scaled compressive elastic modulus (226 ± 17 GPa) and microscaled compressive yield stress of the (0001) GaN micropillars (10 ± 1 GPa). These values can be compared with the modulus (~272 GPa) and hardness (15 GPa) measured by nanoindentation. The Raman spectrum measurements and TEM observations reveal that the residual stress induced in the sputtered GaN film can be largely released upon FIB-milling into 1 \(\mu\)m micropillar. Upon microcompression, the strain energy is basically stored by dislocation and defect accumulation, with minimum residual stress regeneration. With increasing compressive plastic strain, the Raman E\(_2\) FWHM keeps increasing from 2.5 to 6.2 cm\(^{-1}\), consistent with the increasing defect concentration seen in longitudinal TEM micropillar samples. The small bending of the c-axis of the GaN micropillar upon compression loading would also affect its optical performance via the merge of the A\(_1\)(LO) and E\(_1\)(LO) peaks into a single QLO peak.

The authors would like to acknowledge the sponsorship from National Science Council of Taiwan under the Project No. NSC 98-2221-E-110-035-MY3 and the Nano Center of NSYSU.

---