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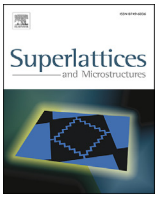

Effect of sputtering deposition process of indium tin oxynitride on surface damage of gallium nitride film

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ABSTRACT

The indium tin oxynitride (ITON) thin film was deposited on the gallium nitride (GaN) film/c-plane sapphire substrate using the magnetron sputtering method. The effects of ITON film sputtering deposition process on the surface damages of GaN film, i.e. the surface morphology, structure and component of GaN films, are investigated systematically. The surface root-mean-square roughness of the GaN film increases after direct current sputtering deposition process (DC-SDP), while that decreases after radio frequency sputtering deposition process (RF-SDP). The photoluminescence intensity ratios of GaN films related with DC-SDP and RF-SDP versus the raw GaN film are 0.46 and 0.98, respectively. It indicates that the raw GaN film surface is damaged seriously after DC-SDP, but almost has no damages after RF-SDP. The atomic concentration ratio of N and Ga in the raw GaN film reduces from 0.97 to 0.87 after DC-SDP, and from 0.97 to 0.96 after RF-SDP. It suggests that RF-SDP can compensate more N atoms to the GaN film than that of DC-SDP. From the high resolution transmission electron microscopy images, we can see that the nitrogen vacancy density at the ITON/GaN interface after RF-SDP is smaller than that after DC-SDP. The forward turn-on voltages (corresponding to the operating current 20 mA) of light emitting diode (LED) fabricated with the GaN films after RF-SDP and that after DC-SDP are 2.6 and 3.0 V, respectively. It can be concluded that the technology of RF-SDP is useful to reduce the surface structural damages of the GaN films and the forward turn-on voltage of the device, and improves the performance of GaN-based LEDs.

1. Introduction

Gallium nitride (GaN) is an essential light emitting material in the device of blue light emitting diode. A device of blue light emitting diode (LED) is usually constructed of sapphire substrate, GaN buffer layer, n-GaN layer, GaN multiple quantum well layer, p-GaN layer and indium tin oxide layer [1–4]. Indium tin oxynitride (ITON) is a novel significant transparent conductive oxide material used in the blue light emitting diode, similar as zinc oxide material [5–8]. The ITON layer is usually fabricated

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on the surface of GaN layer by the sputtering technology [9–14]. GaN film based on blue light emitting diodes play an important role in the applications of semiconductor lighting devices and liquid crystal displays [15–20].

There is a serious problem that the forward turn-on voltage of the blue light emitting diode is high, which makes the light output efficiency low. There are several possible reasons for this problem reported in the previous research [21–30], i.e. the weak optical and electrical properties of the ITO layer. However, the actual key reason is that the GaN film suffered surface damage during the ITO sputtering deposition process (SDP). Until to now, there are a few reports focused on the investigation of the surface damage of GaN film from the ITO sputtering deposition process. One report deduced that the surface damage of GaN film is directly due to the absence of nitrogen atoms in the surface [31]. And there are almost no previous studies involving the formation of the surface damage of GaN film from the ITON sputtering deposition process.

In this work we focus on the effect of sputtering deposition process of ITON film on the surface damage of GaN film. Direct current (DC) and radio frequency (RF) sputtering sources are adopted to deposit ITON film on GaN film/c-plane sapphire substrate. Different sputtering deposition processes can induce notable different influences on the surface damage of GaN film. The surface damage of GaN film, i.e. the surface morphology, photoluminescence, structures and component of GaN films, are determined systematically. The absence of nitrogen atoms in the surface of GaN film is analyzed by use of high resolution transmission electron microscopy.

2. Experimental

The ITON thin film was deposited on the GaN film/c-plane sapphire substrate by the magnetron sputtering method, in which direct current and radio frequency sputtering sources were adopted respectively. The sputtering power was 200 W. The argon gas flow was 80 sccm, and the nitrogen gas flow was 5 sccm. The weight composition of the ITO target was 90% In_2O_3 and 10% SnO_2. The substrate temperature was at room temperature. Firstly, the ITON film with thickness of 100 nm was deposited on the GaN film utilizing 340 s for DC-SDP and 1300 s for RF-SDP. Secondly, the ITON film was removed by etchant ($HCl:FeCl_3 = 1:3$, vol%) from the GaN film/c-plane sapphire substrate. Finally, the properties of GaN film were determined by various methods.

The thickness of the ITON thin film was measured using thin film thickness measuring instrument (Filmetrics F60-t). The surface morphology and roughness of the GaN films were analyzed by atomic force microscopy (AFM, Vecco Dimension Edge). Three-dimension images were gained with scanning area 1 μm × 1 μm by AFM. The photoluminescence properties of the GaN films were measured by the spectrofluorometer (JobinYvon Fluorolog Tau-3) with exciting wavelength of 290 nm and emission wavelength from 350 to 375 nm. The structures of the GaN films were analyzed by X-ray diffractometer (XRD, PANalytical X' Pert PRO MPDs) with Cu K_α radiation. The components and valence band spectra of the GaN films were determined by X-ray photoelectron spectroscopy (XPS, Thermofisher ESCALAB 250Xi) with Al K_α radiation (1486.6 eV, monochromatized), and calibration using C1s line (284.8 eV). The cross section images of GaN/ITON interface were determined by high resolution transmission electron microscope (HRTEM, FEI Tecnai G2 F20) in order to observe the nitrogen vacancies and verify the GaN surface damages.

3. Results and discussion

3.1. Surface morphologies

The surface morphologies of the GaN films corresponding to different sputtering deposition processes are shown in Fig. 1. Fig. 1(a)–(c) shows the two-dimension AFM images of the GaN films and Fig. 1(d)–(f) shows the three-dimension AFM images. From Fig. 1(a)–(f) we can see that the grain sizes on the surface of the GaN films are become bigger after DC-SDP, and the grain size of GaN film corresponding to RF-SDP is smaller than that corresponding to of DC-SDP.

The etching process of ITON films does not affect the grain size of the GaN films, because the etchant ($HCl:FeCl_3 = 1:3$, vol %) does not react with GaN material theoretically and the GaN raw films before and after etching process have the same surface root-mean-square roughness experimentally.

Fig. 1(g) shows that the surface root-mean-square roughness of the GaN film rises from 0.39 to 0.50 nm after DC-SDP, while declines from 0.39 to 0.22 nm after RF-SDP. It indicates that RF-SDP makes the surface of the GaN film smoother than DC-SDP.

3.2. Photoluminescence properties

Fig. 2 shows the photoluminescence spectra of the GaN films corresponding to different sputtering deposition processes. There is one peak at 362 nm in the spectrum of the raw GaN film, which is ascribed to the band edge emission and corresponding to the band gap of 3.425 eV. The peak at 362 nm has not shifts in the spectrum of the GaN film corresponding to DC-SDP, while it shifts to 361 nm (band gap of 3.435 eV) in the spectrum of the GaN film corresponding to RF-SDP. The wavelength of the peak has a blue shift. It indicates that the band gap of the GaN film corresponding to RF-SDP is bigger than the GaN film corresponding to DC-SDP.

The intensities of the peaks change obviously. The intensity of the GaN films decreases a lot after DC-SDP, while it decreases a little after RF-SDP. We calculated shows the peak intensity ratio of the different GaN films versus the raw GaN film at 362 nm peak. The peak intensity ratios related with DC-SDP and RF-SDP are 0.46 and 0.98, respectively. The reduction of the

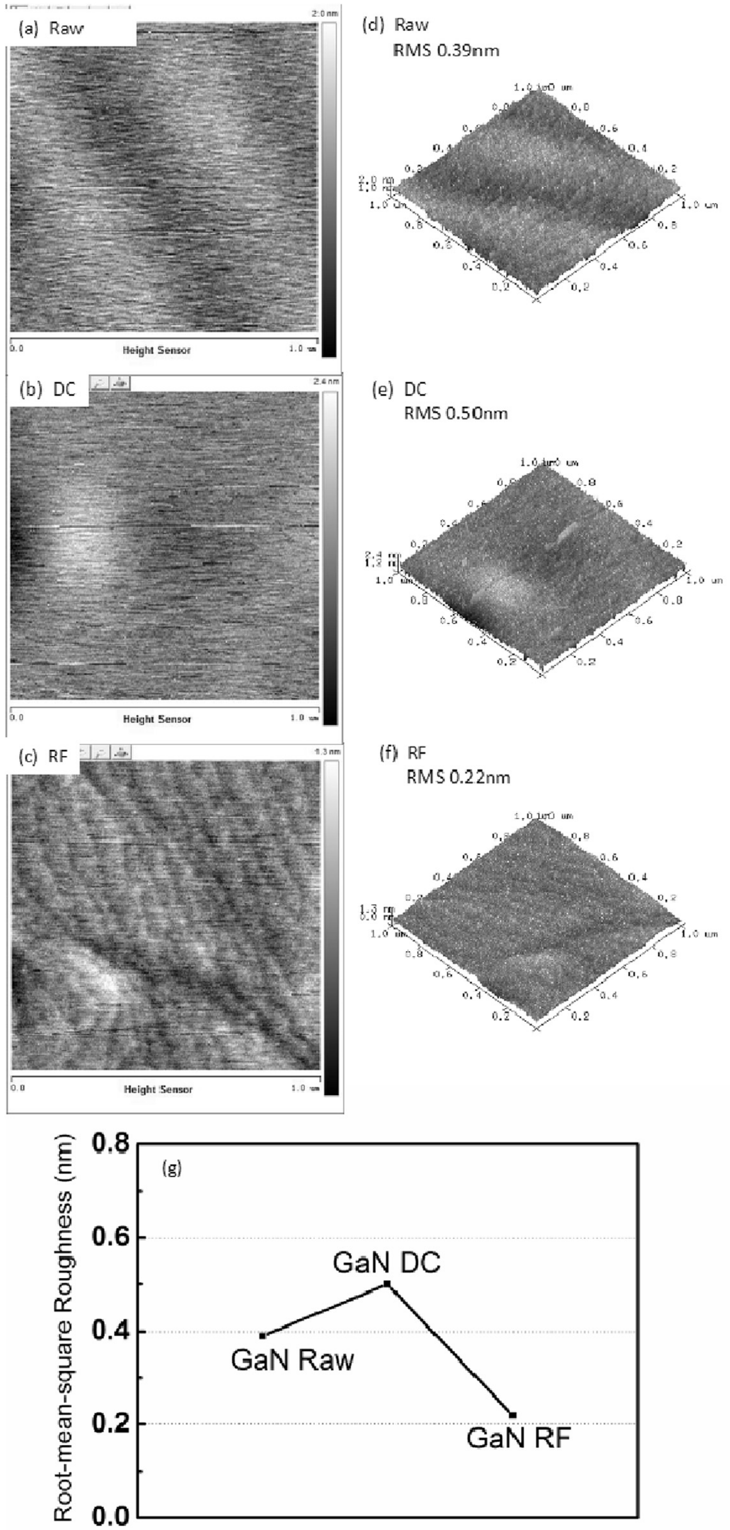

Fig. 1. Surface morphologies of the GaN films corresponding to different sputtering deposition processes: (a)–(c) two-dimension AFM images of raw GaN films, GaN films corresponding to DC-SDP and RF-SDP, respectively; (d)–(f) three-dimension AFM images; (g) surface root-mean-square roughnesses.

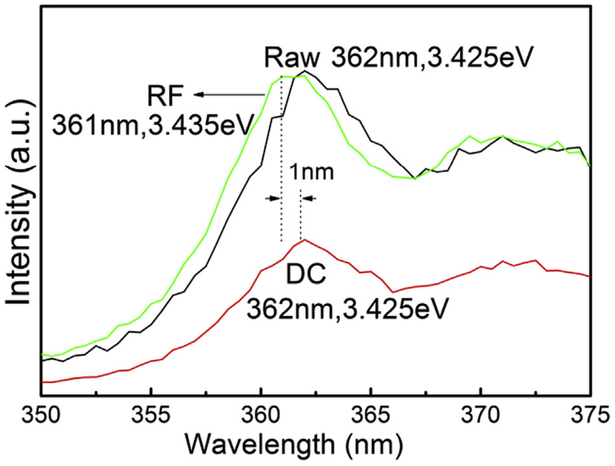

Fig. 2. Photoluminescence properties of the GaN films corresponding to different sputtering deposition processes.

photoluminescence intensity suggests the enlargement of surface damage on GaN films, according to the previous studies related with plasma reactive ion etching process [32,33]. The trend of intensity variation degree versus surface structural damage of GaN film in the sputtering deposition process is similar with that in plasma reactive ion etching process. It indicates that the surface structure of the GaN film is damaged seriously after DC-SDP, and almost has no damages after RF-SDP.

3.3. Structures of GaN films

The photoluminescence properties of the GaN films imply that different sputtering deposition processes have different effects on the surface damage of the GaN film. In order to interpret the formation of the surface damage in the GaN film, the structures are analyzed.

The XRD spectra of the GaN films corresponding to different sputtering deposition processes are shown in Fig. 3. From the XRD spectra of the GaN films, we can see that a main crystal phase can be observed, i.e. GaN structure. The crystal structure of GaN belongs to the crystal system of hexagonal (JCPDS Card No. 50–0792), and the crystal lattice constants are $a_0 = b_0 = 0.3189$ nm, $c_0 = 0.5186$ nm. The peak at 34.6° corresponds to GaN(002), which suggests that the GaN material is a single crystal. The GaN crystal films before and after the etching process of the ITON film, DC-SDP and RF-SDP have the same hexagonal crystal phase and growth orientation [002]. It indicates that the etching process of the ITON film, DC-SDP and RF-SDP do not affect the inner crystal phase structures of the GaN films.

3.4. Component of elements

The component in the GaN film is determined by XPS, as shown in Fig. 4. The atomic concentration ratios of N and Ga elements corresponding to different sputtering deposition processes are calculated through the XPS spectra, as

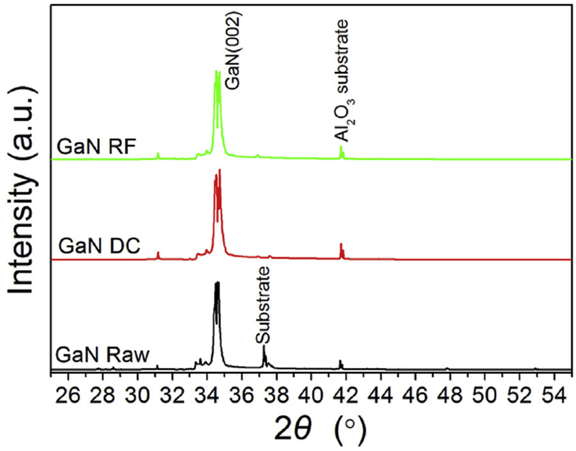

Fig. 3. XRD spectra of the GaN films corresponding to different sputtering deposition processes.

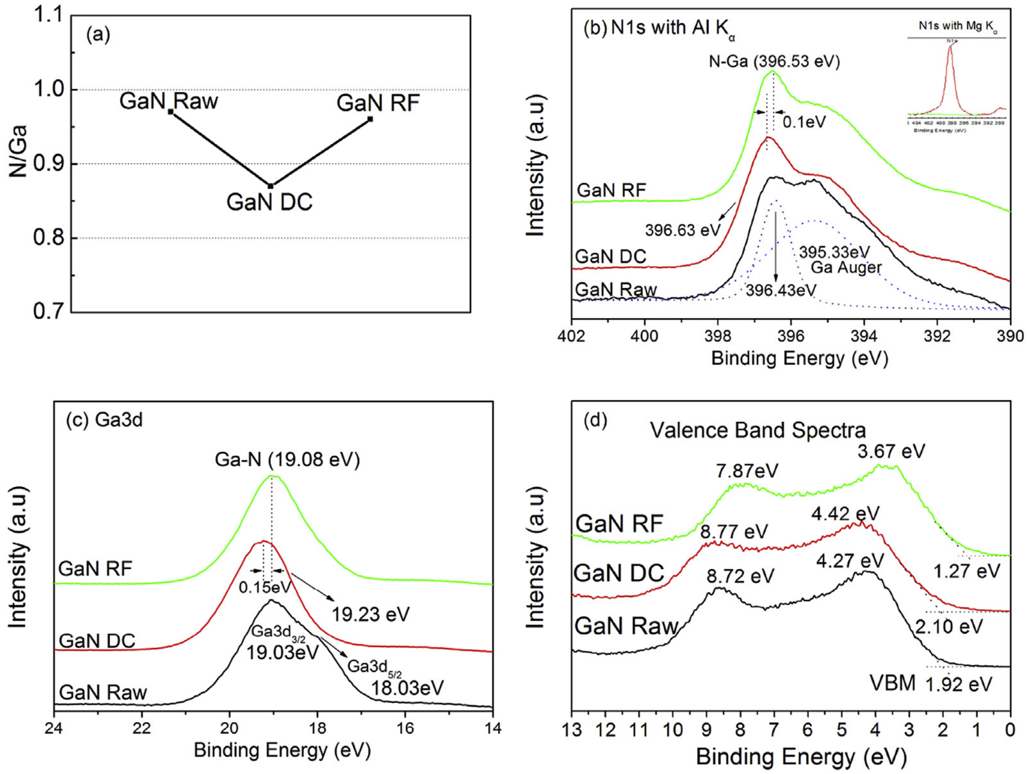

Fig. 4. Element components of the GaN films corresponding to different sputtering deposition processes: (a) N/Ga atomic ratio; (b) XPS spectra of N1s with Al K_α, and inset is that with Mg K_α; (c) XPS spectra of Ga3d; (d) XPS valence band spectra, and valence band maximum (VBM) energy.

shown in Fig. 4(a). The atomic concentration ratio of N and Ga in the GaN film reduces from 0.97 to 0.87 after DC-SDP, and from 0.97 to 0.96 after RF sputtering. It suggests that RF-SDP can compensate more N atoms to the GaN film than DC-SDP.

Fig. 4(b) shows the XPS spectra of N1s core level of the GaN films corresponding to different sputtering deposition processes. There are two peaks in the XPS spectrum of the raw GaN film and the main peak at the binding energy of 396.43 eV is ascribed to the N—Ga bonds. This peak shifts 0.2 eV to higher energy after DC -SDP, and 0.1 eV after RF-SDP. The peak shift to the higher binding energy is due to the introduction of donor-like states [34]. In addition, the secondary peak at 395.33 eV is ascribed to the Ga Auger peak when XPS spectrum is with Al K_α radiation. The inset shows the XPS spectrum of N1s core level with Mg K_α radiation, which verifies that the secondary peak is due to the Ga Auger peak. The atomic concentration of N is emended by de-convolution.

Fig. 4(c) shows the XPS spectra of Ga3d core level of the GaN films corresponding to different sputtering deposition processes. The peak at the binding energy of 19.03 eV of the raw GaN film is ascribed to the Ga—N bonds. This peak has a higher energy shift of 0.2 eV after DC-SDP, and 0.05 eV after RF-SDP. The peak shift to the higher binding energy is due to the introduction of donor-like states [34], for example, Ga interstitials and N vacancies. In our work, the atomic concentration ratio of N and Ga in the raw GaN film is 0.97 which approaches 1.00 and the interstitial formation energy is higher than vacancy formation energy, thus the main donor-like states is N vacancies. It suggests that RF-SDP can compensate more N atoms to the GaN film than DC-SDP.

Fig. 4(d) shows the XPS valence band spectra of the GaN films corresponding to different sputtering deposition processes. Two peaks at the binding energy of 4.27 and 8.72eV are observed, respectively. The peak at 4.27 eV may be attributed to the Ga4p-N2p interaction and correspond to the p-like valence band orbital state, which are similar with InN [30]. The peak at 8.72 eV may be due to the Ga4s-N2p interaction and correspond to the s-like valence band orbital state, which are similar with InN [35]. The peak at 4.27 eV shifts up to 4.42 eV after DC-SDP, and down to 3.67eV after RF-SDP. The valence band maximum (VBM) energy of the GaN film increases from 1.92 to 0.87 eV after DC-SDP, and declines from 1.92 to 1.27 eV after RF-SDP. The valence band maximum energy of the GaN film increases by 0.18 eV after DC-SDP, however declines by 0.65 eV after RF-SDP. It means that the Fermi energy level of the GaN film after RF-SDP is closer to the valence band maximum energy than that of the GaN film after DC-SDP, which can reduce the energy band bending of the GaN film and decrease the interface contact barrier between the ITON and GaN film.

3.5. The surface damage of the GaN film

The surface damage of the GaN film is investigated by high resolution transmission electron microscope (HRTEM). Fig. 5 shows the HRTEM images of the ITON/GaN interface corresponding to different sputtering deposition processes. From the left of the Fig. 5, we can see that there are some nitrogen vacancies V_N in the ITON/GaN interface. It indicates that the nitrogen vacancy density in the ITON/GaN interface after RF-SDP is smaller than that after DC-SDP. The insets of Fig. 5 show the selected area diffraction images of the ITON/GaN interface. The main crystal planes of GaN are nearly similar.

The crystal structure models of GaN are shown in the bottom insets of Fig. 5. The crystal structure models of GaN belong to the hexagonal crystal system, which is consistent with the result of XRD result. Fig. 5(a) shows the interplanar spacing of GaN(002) is 0.259 nm-nearly a half of crystal lattice constant c_0 (0.5186 nm). It is clearly seen from Fig. 5 that the growth orientation of ITON prepared by DC-SDP is [222], while that is [400] corresponding to RF-SDP. Fig. 5 shows the interplanar spacing of ITON(222) fabricated by DC-SDP is 0.293 nm and that of ITON(400) fabricated by RF-SDP is 0.254 nm. It suggests that the interplanar spacing of ITON(400) fabricated by RF-SDP is closer to the interplanar spacing of GaN(002) than that of ITON(222) fabricated by DC-SDP. As is obviously shown from the rectangles in the middle of Fig. 5, the lattice matching of the GaN and ITON fabricated by RF-SDP is better than that related with DC-SDP.

The surface damage of the GaN film is ascribed to the N vacancies on the GaN surface. The RF-SDP of the ITON film can compensate more N vacancies on the GaN surface than DC-SDP. It implies that the RF-SDP of the ITON film can reduce more surface damages of the GaN film than DC-SDP.

The intrinsical reason of the RF-SDP can replenish more N atoms to the GaN film than DC-SDP is that the RF-SDP can reduce more O^{2-} sputtering ions than DC-SDP. Fig. 6 shows the diagrammatic sketches of GaN crystal surface damage mechanism in different ITON film sputtering deposition processes. The DC-SDP applies only a negative voltage on the ITO target, generates some O^{2-} sputtering ions that have larger bombardment effect on the GaN crystal surface, and results in loss of more N atoms on the GaN crystal surface. However, the RF-SDP applies negative voltage and positive voltage to the ITO target alternately, generates less O^{2-} sputtering ions than DC-SDP, and lead to replenish more N atoms on the GaN crystal surface than DC-SDP.

3.6. Performance of the GaN-based LED

Fig. 7 shows the curves of current versus voltage for GaN-based LEDs fabricated with the GaN films corresponding to different sputtering deposition processes. From Fig. 7 we can see that, the forward turn-on voltages (corresponding to the

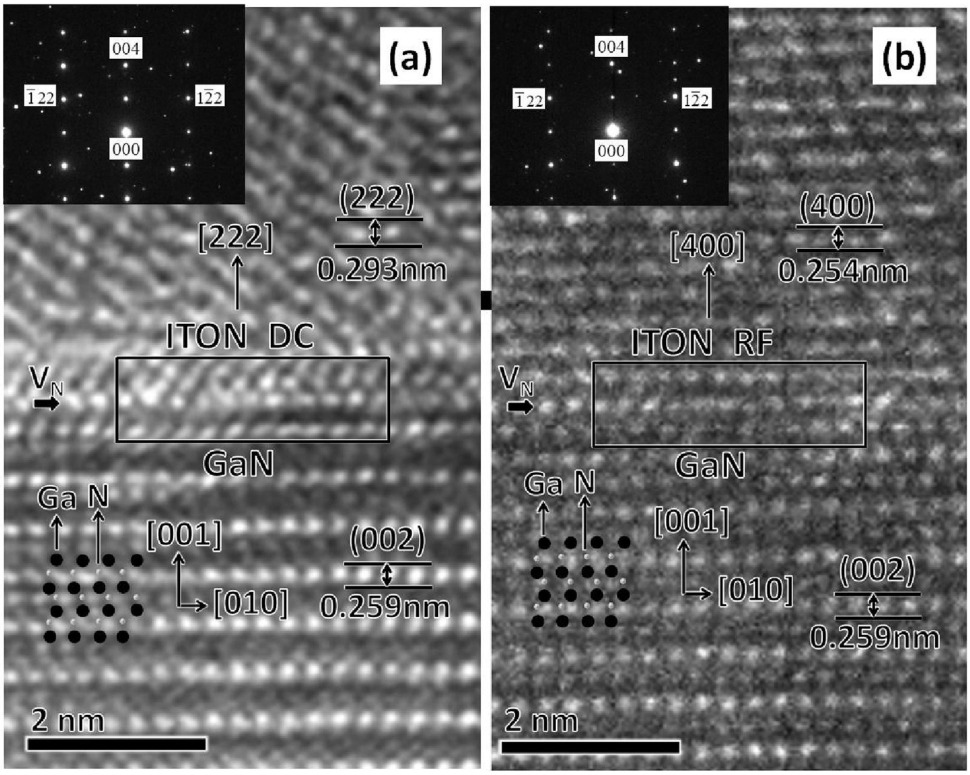

Fig. 5. HRTEM images of the ITON/GaN interface with the GaN films corresponding to different sputtering deposition processes. Insets are the selected area diffraction patterns and crystal structure models.

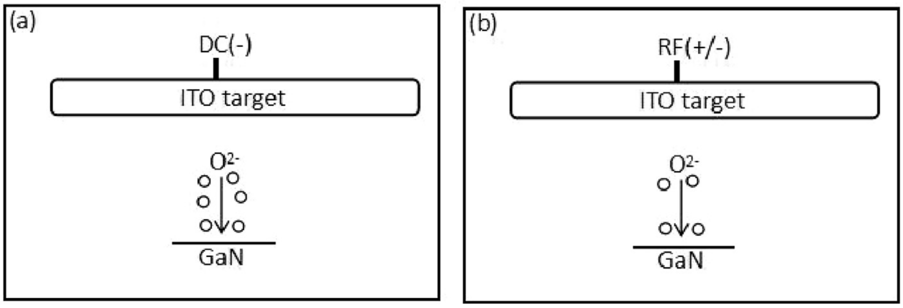

Fig. 6. The diagrammatic sketches of GaN crystal surface damage mechanism in different ITON film sputtering deposition processes: (a) DC-SDP; (b) RF-SDP.

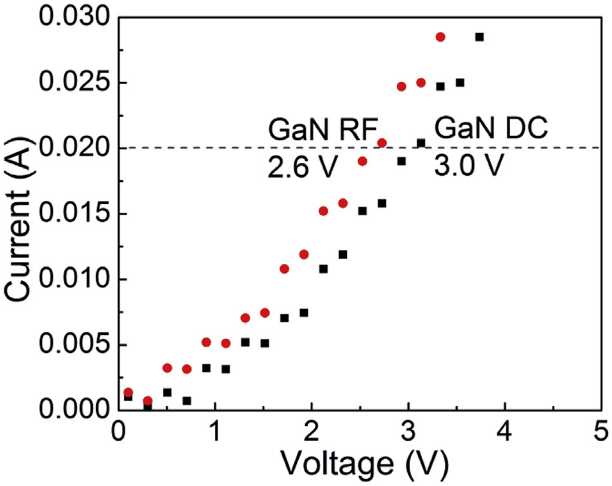

Fig. 7. The current-voltage curves of the GaN-based LEDs fabricated with the GaN films corresponding to different sputtering deposition processes.

current 20 mA) of LED fabricated with the GaN films after RF-SDP and DC-SDP are 2.6 and 3.0 V, respectively. The forward turn-on voltage of the GaN-based LEDs by RF-SDP reduces about 0.4 V than that by DC-SDP. It can be concluded that the technology of RF-SDP is useful to reduce the forward turn-on voltage and improve the performance of the GaN-based LEDs due to the modification of the ITON/GaN interface structure.

4. Conclusions

The ITON thin film was deposited on the GaN/c-plane sapphire substrate by the magnetron sputtering method. Investigation indicates that RF-SDP makes the surface morphology of the GaN film smoother than that of DC-SDP. The photoluminescence intensity ratios of the GaN films related with DC-SDP and RF-SDP versus the raw GaN film are 0.46 and 0.98, respectively. It indicates that the GaN film surface is damaged seriously after DC-SDP, and almost has no damages after RF-SDP. The XRD patterns indicate that the crystal structure of the GaN film after RF-SDP is closer to the raw GaN film than that after DC-SDP. The atomic concentration ratios of N and Ga in the GaN film reduces from 0.97 to 0.87 after DC-SDP and from 0.97 to 0.96 after RF-SDP. It suggests that RF-SDP can compensate more N atoms to the GaN film than DC-SDP. The valence band maximum energy of the GaN film increases about 0.18 eV after DC-SDP, and declines about 0.65 eV after RF-SDP. From HRTEM images, we can see that the nitrogen vacancy density in the ITON/GaN interface after RF-SDP is smaller than that after DC-SDP. The surface structural damage of the GaN film is ascribed to the N vacancies formed on the GaN surface. It implies that the RF-SDP of the ITON film can reduce more surface damages of the GaN film than that by DC-SDP. RF-SDP can minish the energetic atom bombardment effects on the GaN surface. The forward turn-on voltage of the GaN-based LEDs by RF-SDP reduces about 0.4 V than that by DC-SDP. It can be concluded that the technology of RF-SDP is useful to reduce the forward turn-on voltage and improve the performance of the GaN-based LEDs.

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