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Epitaxial growth and its mechanism of GaN films on nitrided $LiGaO_2$(001) substrates by pulsed laser deposition

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Abstract: High-quality GaN films have been grown on nitrided $LiGaO_2$ substrates by pulsed laser deposition with an in-plane epitaxial relationship of GaN[11-20]//$LiGaO_2$[010]. The surface morphologies and structural properties for as-grown GaN films are studied by various characterizations in detail. These characterizations for the as-grown GaN films show excellent crystalline quality with a full-width at half-maximum value of 0.1° and a very smooth surface with a surface root-mean-square roughness of 1.1 nm. There is an interfacial layer existing between GaN films and $LiGaO_2$ substrates with a thickness of 0.9 nm. Furthermore, the nitridation effect on the properties of GaN films and the growth mechanism of GaN films on nitrided $LiGaO_2$ substrates by PLD have also been systemically studied. This work opens up a broad prospect for the growth of high-efficiency GaN-based devices on $LiGaO_2$(001) substrates.

Keywords: GaN; nitrided $LiGaO_2$ substrates; pulsed laser deposition; full-width at half-maximum; interfacial layer.

1. Introduction

In recent years, GaN and its related compound materials have attracted much attention due to their excellent properties, which make them suitable for the wide applications in solid state lighting, display, solar cells, and so on.[1-8] So far, GaN-based devices prepared on sapphire substrates have been commercialized. However, due to the large lattice and thermal expansion coefficient mismatches between GaN and sapphire, GaN-based devices on sapphire substrates could not meet the market demand for high-power lighting. Therefore, researchers have tried hard to fabricate GaN-based devices on novel substrates, such as $LiAlO_2$, $La_{0.3}Sr_{1.7}AlTaO_6$ (LSAT) and $LiGaO_2$[2, 6-8] which share relatively small lattice and thermal expansion coefficient mismatches with GaN. Among the foreign substrates for the epitaxial growth of GaN, $LiGaO_2$ is considered to be one of the best candidates. On the one hand, $LiGaO_2$ shows very small lattice and thermal expansion coefficient mismatches with GaN.[9-10] On the other hand, $LiGaO_2$ is easily etched by acid, which makes it possible for the fabrication of free-standing GaN-based devices.[9] What's more, it should be noted that Ga-face $LiGaO_2$ is suitable for the growth of high-quality GaN films at low temperature even without a nucleation layer.[11]

It has demonstrated that GaN films can be grown on $LiGaO_2$ by metal-organic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE).[12-13] However, due to the high growth temperature of MOCVD and MBE, GaN films grown on $LiGaO_2$ substrates usually suffer from the diffusion of Li atoms from the substrates and therefore lead to the formation of thick interfacial layer.[11-13] which hampers the growth of high-quality GaN films. One of the most effective approaches to overcome these problems is to reduce the growth temperature. Pulse laser deposition (PLD) is a suitable technology for the low temperature growth and has been demonstrated to be an excellent approach for the epitaxial growth of high-quality GaN films on $LiGaO_2$ substrates. [11] Additionally, nitridation on the substrate (eg. GaAs, InP) is believed to be an easy and effective way to suppress or stop the diffusion of active atoms.[14-17] Specially, it has been demonstrated that nitridation on $LiGaO_2$ helps to reduce or suppress the diffusion of atomic hydrogen into the $LiGaO_2$ bulk substrate in the hydrogen-rich MOCVD environments.[18] Thus, the combination of PLD and nitrided $LiGaO_2$ is an effective solution for the suppression the interfacial reactions between diffused Li atoms and GaN films, and is beneficial to the growth of high-quality GaN films.

PLD has been employed to grow GaN films on $LiGaO_2$ substrates for several years[11, 19-20] It is interesting to point out that high-quality GaN films can be directly grown on $LiGaO_2$ substrates by PLD without the formation of a nucleation layer.[11] So far, the procedure of nitridation has been successfully utilized in the synthesis of GaN films on $LiGaO_2$ substrates.[17-18] However, the nitridation effect of $LiGaO_2$ substrates on the properties of GaN films grown by PLD, as well as its growth mechanism, lacks a thorough study.

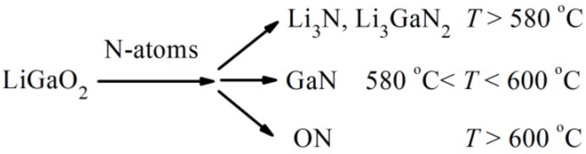
In this work, we report on the growth and characterization of high-quality GaN films on nitrided $LiGaO_2$ substrates by PLD. The underlying growth mechanism is discussed in detail as well. The surface morphologies and structural properties for as-grown GaN films measured by *in situ* reflection high energy electron diffraction (RHEED), scanning electron microscopy (SEM), atomic force microscopy (AFM), high-resolution X-ray diffraction (HRXRD), grazing incidence X-ray reflectivity (GIXR), and high-resolution transmission electron microscopy (HRTEM) are also reported.

2. Experiments

As-received $LiGaO_2(001)$ substrates were taken degassing treatment in the lock-load ultrahigh vacuum (UHV) chamber after cleaning by acetone and alcohol, and then dried by high-purity (7N) N_2. After degassing treatment,[21] $LiGaO_2(001)$ substrates were transferred into the UHV growth chamber with a background pressure of 5.0×10^{-10} Torr, and were annealed at 700 ^{o}C for 30 min to remove the residual carbon contaminants on the substrate surface. Afterwards, $LiGaO_2$ substrates were nitrided at 590 ^{o}C for 60 min with the pressure of 4×10^{-3} Torr in a nitrogen plasma atmosphere, which were provided by a radio frequency (RF) plasma radical generator operated at 500 W. During the GaN growth, the KrF excimer laser light was employed to ablate the high-purity (7N) metal Ga target with the energy of 250 mJ/pulse and laser pulse repetition of 20 Hz. The ablated species produced by the laser were mounted 5 cm away from the substrates, and the temperature were maintained at 700 ^{o}C. Meanwhile, nitrogen plasmas with the pressure of 1.0×10^{-2} Torr were supplied by RF plasma radical generator working at 500 W. During the whole growth, *in-situ* RHEED was utilized to monitor growth condition. The surface morphologies and structural properties of as-grown GaN films were measured by SEM (Nova Nano SEM 430 Holland), AFM (MFP-3D-S Asylum American), HRXRD (Bruker D8 X-ray diffractometer with Cu Kα1 X-ray source, λ=1.5406 Å), and cross-sectional HRTEM (JEOL 3000F field emission gun TEM, 300 kV, 0.17 nm), respectively.

3. Results and Discussion

The *in-situ* RHEED is employed to monitor the whole growth process. Fig. 1a illustrates the clear RHEED patterns of $LiGaO_2(001)$ substrate before the annealing process, which indicates that as-received $LiGaO_2(001)$ is rather rough. After annealing at 700 °C for 30 min, the sharp and streaky RHEED patterns of $LiGaO_2(001)$ can be found, suggesting that clean and flat $LiGaO_2(001)$ surface is achieved, as shown in Fig. 1b.[11] After nitridation, RHEED patterns become slightly dim with some bright points on them, as shown in Fig. 1c. This demonstrates that GaN has been formed on $LiGaO_2$ surface. It has been reported that the relationship between products and $LiGaO_2$ nitridation temperature can be described as:[18]



In this work, the nitridation temperature is set as 590 °C, which leads to the formation of GaN monolayer on the substrate surface by the nitridation process. This GaN monolayer is normally beneficial to the subsequent GaN growth. Furthermore, it is reasonable to assume that N-face GaN monolayer has been realized by nitridation of Ga-face $LiGaO_2$ substrates.[14] Fig. 1d displays the sharp and clear RHEED patterns for as-grown GaN films on nitrided $LiGaO_2$ substrates, indicating that single-crystalline GaN films with flat surface have been achieved on $LiGaO_2(001)$ substrates. Actually, all of the GaN, Li_3N, $LiGaN_3$, and ON influence the film quality. The formed Li_3N, $LiGaN_3$, and ON are usually polycrystalline or amorphous, and show different structures with GaN, which lead to easier formation of defects during the subsequent GaN growth, and therefore greatly deteriorates the properties of as-grown GaN films. Meanwhile, Fig. 1e shows the RHEED patterns for as-grown GaN films on non-nitrided $LiGaO_2$ substrates. Clearly, single-crystalline GaN films can also be grown on non-nitrided $LiGaO_2$ substrates. This result can be attributed to the merits of PLD, the flat and lattice-matched $LiGaO_2$ substrates and nitridation.[20-22] Furthermore, after carefully studying the RHEED patterns, the in-plane epitaxial relationship between GaN film and $LiGaO_2$ substrate of GaN[11-20]//$LiGaO_2$[010] can be deduced, and we find that the GaN film grown on nitrided $LiGaO_2$ substrate is Ga-face GaN, while the GaN film grown on non-nitrided $LiGaO_2$ substrate is N-face GaN.[23-25]

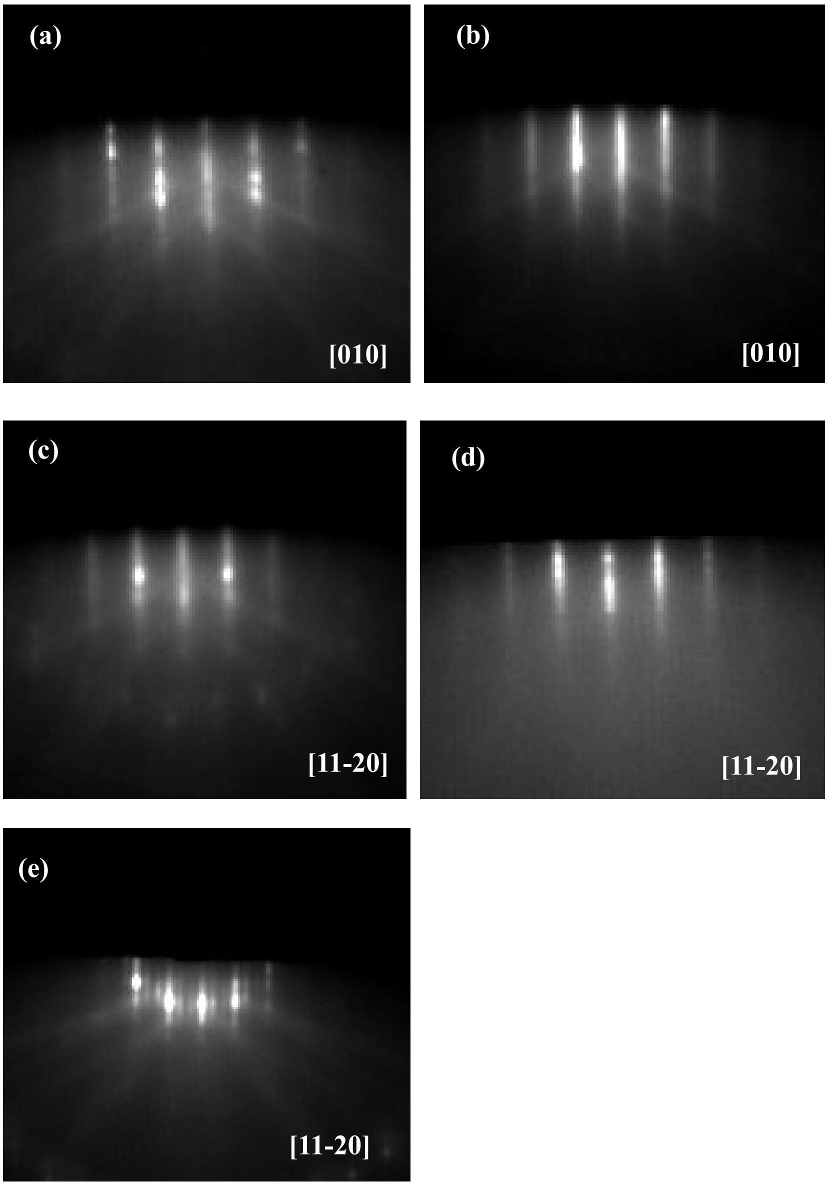
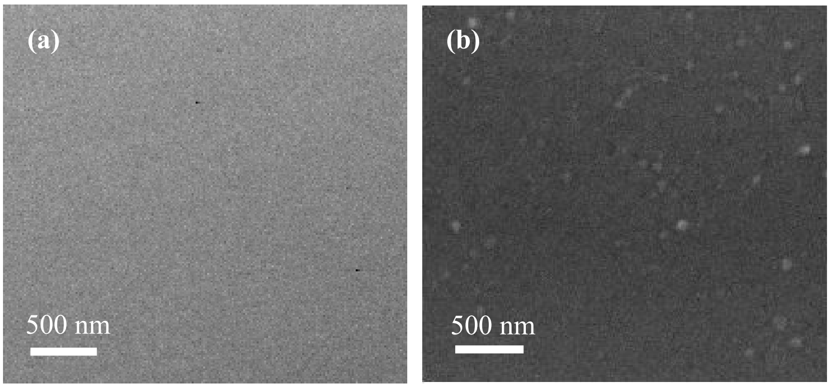


Fig. 1. RHEED patterns for $LiGaO_2$(001) substrates (a) before annealing; (b) after annealing; (c) after nitridation with the electron beam incidence along [010]. RHEED patterns for GaN films grown at (d) nitrided and (e) non-nitrided $LiGaO_2$ substrates with the electron beam incidence along [11-20].

Figs. 2a-d show the FESEM and AFM images for as-grown GaN films on nitrided and non-nitrided $LiGaO_2$(001) substrates, respectively. The GaN films grown on nitrided $LiGaO_2$(001) substrates show flatter nanostructure surface than that on non-nitrided $LiGaO_2$ substrates as observed in Fig. 2a and b, which is consistent well with the RHEED observation. Further study by

AFM measurement reveals that the as-grown GaN films on nitrided $LiGaO_2$(001) substrates show very smooth surface with the surface root-mean-square (RMS) roughness of 1.1 nm, as shown in Fig. 2c. This is a striking contrast to 3.5 nm obtained from the GaN films grown on the non-nitrided $LiGaO_2$ substrates, as shown in Fig. 2d. It is reported that up-to-date RMS roughness for device-ready GaN films is about 1.1 nm, which is achieved in the three most popular substrates cases, including sapphire, SiC, and Si substrates using conventional high-temperature growth techniques such as MOCVD, HVPE (hydride vapour phase epitaxy) or MBE.[26-33] Clearly, the up-to-date RMS roughness value is in the same level as of the GaN films grown on nitrided $LiGaO_2$ substrates in this work. Apparently, the GaN film on the nitrided $LiGaO_2$ substrate shows a very smooth and device-ready surface. We attribute this to two aspects. One is the nitridation of $LiGaO_2$ substrates,[14-17] and the other is the very small thermal expansion mismatch between the GaN films and the nitrided $LiGaO_2$ substrates.[34] The former leads to the formation of N-face GaN monolayer, which is possibly promising to form Ga-face GaN in the subsequent Ga-rich growth condition.[35] That is to say, in the subsequent PLD growth, the growth surface is the Ga-face GaN (or so-called Ga polarity GaN or GaN(0001)), which is good for the migration of GaN precursors and, thus, is beneficial to form the smooth GaN films.[36-38] The latter shows much smaller thermal expansion mismatch than that between GaN films and non-nitrided $LiGaO_2$ substrates, which indicates that the gallium adatom diffusion barrier and growth kinetics for GaN films grown on the nitrided $LiGaO_2$ substrates is much lower than that on the non-nitrided $LiGaO_2$ substrates.[39] This eventually results in a smoother surface. In contrast, the poorer surface morphologies of GaN films on non-nitrided $LiGaO_2$ substrates would be lied to the lattice and thermal expansion mismatches with GaN as well as N-terminated surface.[36-38]



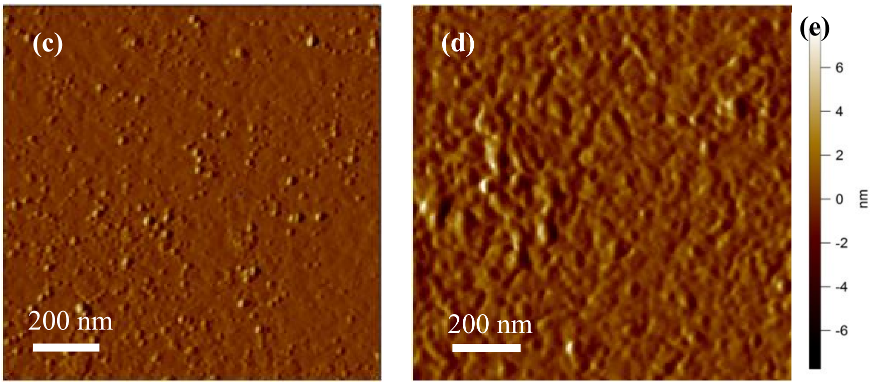


Fig. 2. FESEM images for as-grown GaN films on (a) nitrided and (b) non-nitrided $LiGaO_2$ substrates. AFM images for as-grown GaN films on (c) nitrided and (d) non-nitrided $LiGaO_2$ substrates. (e) is the color scale of AFM images.

The structural properties of as-grown GaN films are characterized by HRXRD. Fig. 3a shows the typical 2θ-ω scan for GaN films grown on $LiGaO_2$ substrates. Only diffraction peaks of GaN{0001} are detected except the diffraction peaks of $LiGaO_2${001}, which confirms that single-crystalline GaN films with the *c*-axis normal to $LiGaO_2$ substrates are obtained.[11] Fig. 3b is the φ scan of as-grown GaN (11-24), which exhibits six-fold rotational peaks with the interval of 60^o. It once again confirms that single-crystalline of hexagonal GaN films are obtained. Fig. 3b also shows the φ scan of the $LiGaO_2$(011) substrate. It shows two-fold rotational peaks with the interval of 180^o. These results once again confirm the in-plane epitaxial relationship of GaN[11-20]//$LiGaO_2$[010] obtained from RHEED measurement, indicating that the lattice mismatch between GaN and $LiGaO_2$ is as small as 0.2%.[40]

The crystalline quality of as-grown GaN films is studied by X-ray rocking curves (XRCs). Fig. 3c illustrates the XRCs for GaN films grown on nitrided and non-nitrided $LiGaO_2$ substrates. From Fig. 3c, the full-width of half-maximum (FWHM) values for GaN (0002) films grown on nitrided and non-nitrided $LiGaO_2$ substrates are 0.1° and 0.2°, respectively. These results indicate the higher-quality GaN films are grown on nitrided $LiGaO_2$ substrates. Meanwhile, the crystalline quality of GaN films on nitrided $LiGaO_2$ substrates achieved in this work is much better than that of the GaN films grown on sapphire by PLD,[22] and is comparable with the art-of-state GaN films grown by MOCVD and PLD on other substrates.[41-42] The N-face GaN monolayer has been formed by the high temperature nitridation before the growth of GaN films by PLD, which is beneficial to the nucleation of GaN and shows a reduction in the formation of threading dislocations when compared with that grown on non-nitrided $LiGaO_2$ substrates.[14, 43]

GIXR is introduced to examine the structural properties of GaN/$LiGaO_2$ interfaces. Fig. 3d displays the experimental reflectivity data and its fitted curve for the as-grown GaN films on nitrided $LiGaO_2$ substrates based on the Fresnel equation.[44-45] The fitting curve reveals that an interfacial layer with a maximum thickness of 1.0 nm exists between $LiGaO_2$ substrates. It also shows that the GaN surface RMS roughness is 1.2 nm, which is very close to the value measured by AFM. Similarly, GaN on non-nitrided $LiGaO_2$ has a much thicker interfacial layer with a value of 5.1 nm conducted at the same conditions. This clearly means that $LiGaO_2$ nitridation is of significance for the reduction in the thickness of interfacial layer. Meanwhile, the thickness of interfacial layer from GaN films on nitrided $LiGaO_2$ is much smaller than that between GaN and non-nitrided $LiGaO_2$ grown at 700 °C with a value of 16.6 nm.[11] We tentatively attribute this to the stable N-face GaN monolayer, which can effectively suppress the diffusion of Li atoms from the substrate and reduce the interfacial reactions.[11, 15]

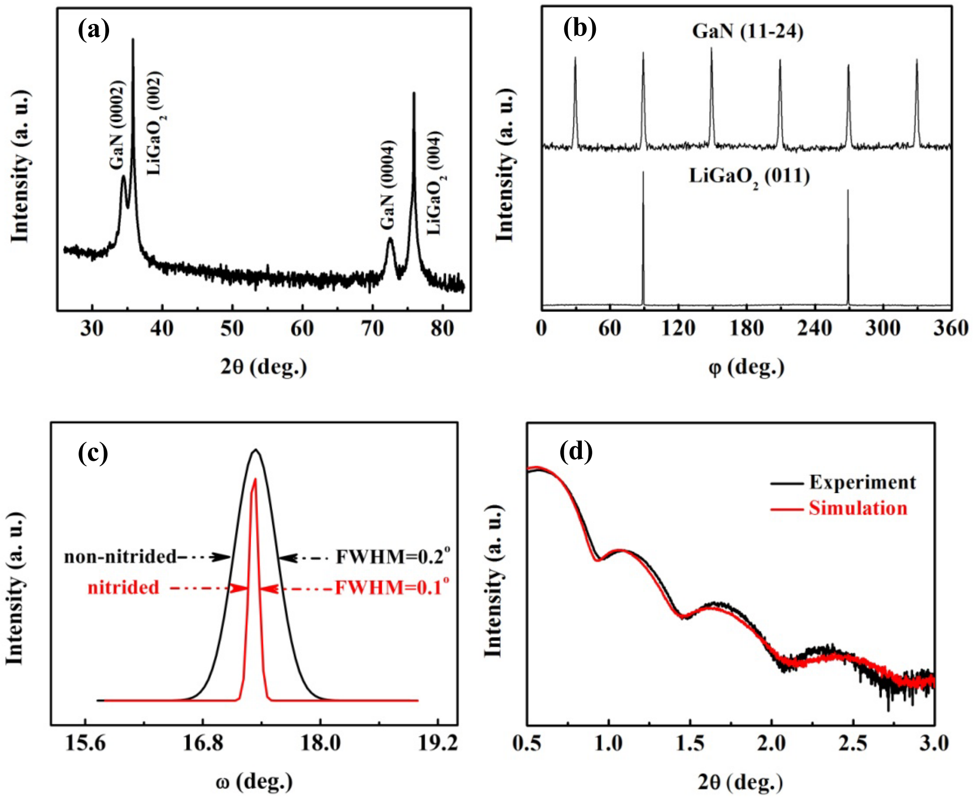


Fig. 3. HRXRD measurements of as-grown GaN films on nitrided $LiGaO_2$ substrates. (a) 2θ-ω scan; (b) φ scan of hexagonal GaN (11-24) and $LiGaO_2$ (011); (c) (0002) XRCs of as-grown GaN

films on nitrided and non-nitrided $LiGaO_2$; (d) Typical GIXR spectrum for the about 19 nm-thick GaN films on nitrided $LiGaO_2$ substrates.

In order to further study the interface between GaN films and $LiGaO_2$(001) substrates, cross-sectional TEM is introduced to evaluate GaN films grown on nitrided $LiGaO_2$. The cross-sectional TEM images of as-grown GaN films are shown in Figs.4. One can clearly see that the interface between GaN films and $LiGaO_2$(001) substrates is abrupt, as shown in Fig. 4a. This result is well consistent with GIXR measurement. The insert in Fig. 4 is a selected area electron diffraction (SAED) image of GaN films. According to the reciprocal lattice constants of GaN, the d-spacing of GaN in the [0001] and [10-10] directions are calculated to be 0.517 and 0.281 nm, respectively.[46] Evidently, this result once again confirms that single-crystalline of hexagonal GaN films have been grown on $LiGaO_2$. The excellent periodicity of lattice arrangement in the as-grown GaN films can be verified from the HRTEM image of the interface between GaN films and $LiGaO_2$(001) substrates, as illustrated in Fig. 4, which is attributed to the small lattice and thermal expansion coefficient mismatches between GaN films and $LiGaO_2$(001) substrates. Furthermore, the interface is quite bright and the interfacial layer thickness is verified to be 0.9 nm, which is in good agreement with GIXR measurements. In this case, the interfacial layer is GaN monolayer that formed by the nitridation of $LiGaO_2$ substrates, where the Ga atoms react with the nitrogen plasmas. The formed GaN monolayer can enhance the growth rate of GaN films provide as a good template for the subsequent growth of GaN films, and enhances the two-dimensional (2D) growth mode of GaN films.

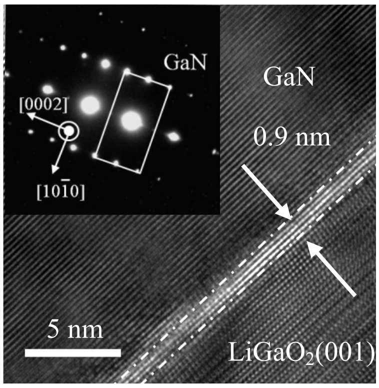


Fig. 4. cross-sectional TEM images of as-grown GaN films on $LiGaO_2$(001) substrates and inserted picture is a SAED image of GaN.

After studying the quality of GaN films carefully, the growth mechanism for GaN films grown on the nitrided $LiGaO_2$ substrates can be therefore deduced. As illustrated in Fig. 5, the growth process for GaN films deposited on the nitrided $LiGaO_2$ substrates is ascribed as follow. After thermal annealing at 700 ^{o}C for 30 min, clean and flat Ga-face $LiGaO_2$ substrates have been achieved.[11] Subsequently, the nitridation procedure of $LiGaO_2$ substrates is taken place by N plasmas supplied by RF plasma generator. During the nitridation process, the N plasmas could react with Ga atoms on the substrate surface with a rather slow rate and results in the formation of the atomically flat N-face GaN monolayer, proved by RHEED, Fig. 1c. Afterwards, high energy Ga plasmas produced by KrF laser are introduced to react with N plasmas to form GaN on the flat and ordered N-face GaN monolayer. This formed GaN monolayer can be found in Fig. 4, marked by two dashed lines, which is considered to be an excellent homo-template and is good for the adoption of Ga adatoms as well as the migration of GaN precursors on the surface. Furthermore, the formation of the table GaN monolayer can effectively eliminate the diffusion of Li atoms from the substrate, eventually stop the serious interfacial reactions between Li atoms and Ga (N) adatoms. Thereby, high crystalline quality Ga-face GaN films with flat surface morphologies are obtained on the nitrided $LiGaO_2$ substrates. As for the case of GaN films grown on non-nitrided $LiGaO_2$ substrates, due to the absence of the stable GaN barrier, the diffused Li atoms will react with Ga (or N) adatoms. This may lead to polycrystalline or amorphous materials, and then results in the thick interfacial layer between GaN films and non-nitrided $LiGaO_2$ substrates where many defects are formed, which greatly deteriorates the quality of subsequent growth GaN films. As a result, GaN films on non-nitrided $LiGaO_2$ substrates display poorer surface morphologies and structural properties due to heteroepitaxy, N-terminated surface, and the absence of the stable GaN barrier. Consequently, the nitridation of $LiGaO_2$ substrates plays a critical role in improving the surface morphologies and structural properties of as-grown GaN films by PLD.

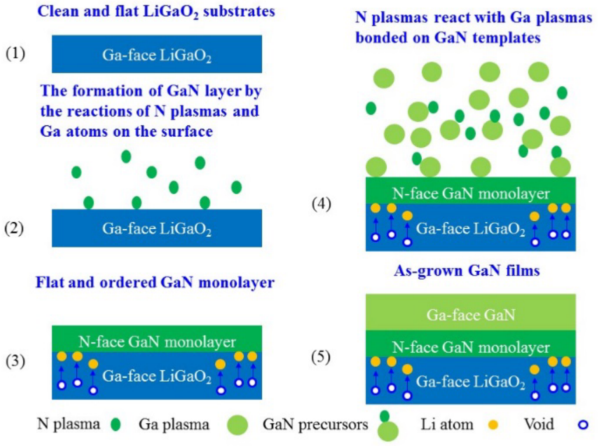


Fig. 5. Schematic diagrams for the GaN films grown on nitrided $LiGaO_2$ substrates.

4. Conclusions

In summary, high-quality GaN films grown on nitrided $LiGaO_2$(001) substrates by PLD as well as its growth mechanism have been carefully investigated. The FWHM of (0002) from a 19 nm-thick GaN films grown on nitrided $LiGaO_2$ is as small as 0.1^o, and the surface RSM roughness of the GaN film is as small as 1.1 nm. The interfaces between GaN films and $LiGaO_2$(001) substrates are atomically abrupt. Furthermore, the thickness of interfacial layer is measured to be 0.9 nm by HRTEM and is quite close to the result got from GIXR measurement. The successful growth of high-quality GaN on nitrided $LiGaO_2$(001) substrates can be mainly ascribed to two aspects. One is the small lattice and thermal expansion coefficient mismatches between GaN films and $LiGaO_2$ substrates. The other is the nitridation of $LiGaO_2$ substrates. The former is good for the nucleation of GaN films and is beneficial to the growth of high-quality GaN films. The latter is conductive to form a stable GaN monolayer that helps to reduce the interfacial reactions by suppressing the diffusion of Li atoms from the substrates. The study of the growth mechanism reveals that the nitridation of $LiGaO_2$ substrates plays an important role in achieving high-quality GaN films. The successful growth of high-quality GaN films on $LiGaO_2$(001) substrates provides an excellent solution for synthesizing of high-quality GaN-based devices on $LiGaO_2$ (001) substrates.

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