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Effect of nitridation on structure, electrical and optical properties of InN epilayers grown on sapphire by PAMBE

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ABSTRACT

InN epilayers were prepared on c-plane (0001) sapphire substrates by plasma-assisted molecular beam epitaxy (PAMBE). We studied the effect of nitridation on structure, surface morphology, electrical and optical properties of InN films using X-ray diffraction (XRD), Reflection high-energy electron diffraction (RHEED), scanning electron microscopy (SEM), atomic force microscopy (AFM), Hall and photoluminescence (PL) measurement. The results showed a significant improvement of the crystalline qualities and surface morphologies and enhancement of the electrical and optical properties for InN films grown after nitridation. Moreover, the energy band-gap of InN films were also determined by optical absorption and photoluminescence (PL) measurement.

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Extensive attention has been paid to the indium nitride (InN) epitaxial layers and devices because of its promising properties such as lowest effective mass, highest electron mobility and narrowest band gap energy (~0.7 eV) among the III-nitride semiconductors $[1-3]$ $[1-3]$ $[1-3]$, which make it ideal for high efficiency solar cells, infrared light-emitting diodes (LEDs) and laser diodes (LDs) [\[4,5\].](#page--1-0) Moreover, the narrow band-gap of InN allows the group III-nitrides alloy system, such as (AlGaIn)N-based light emission devices, to extend their spectral range from near-infrared to deep ultraviolet. Initially, the band-gap of InN film was predominantly found at around 1.9 eV by the optical absorption of InN films deposited by sputtering techniques $[6]$. The variations in the band-gap measurements were mainly due to the presence of oxide precipitates [\[7,8\]](#page--1-0), the formation of indium (In) clusters $[9]$ and the Burstein-Moss energy shift [\[10\]](#page--1-0). In order to grow pure InN layers and get its more accurate band-gap energy, the main difficulty lies with its thermal decomposition, which becomes excessive above 600 \degree C and its impurity incorporation problem, especially by oxygen [\[11,12\].](#page--1-0) Even though the InN films grown by metal organic vapour phase epitaxy (MOVPE) techniques exhibit a significant improvement of the crystalline quality as well as a much narrower direct band-gap. Higher growth temperature is required for effectively cracking ammonia, which conflicts with the low growth temperature required to prevent InN decomposition [\[13\]](#page--1-0). In comparison, plasma-assisted molecular beam epitaxy (PAMBE) is a suitable method for producing InN, in which low growth temperature is possible in combination and impurity incorporation is less under an ultra high vacuum (UHV) growth environment [\[1\].](#page--1-0) Currently, the sapphire substrates have been widely used for the heteroepitaxial growth of InN. However, a large lattice mismatch around 25% between InN and sapphire substrates exists. Therefore, nitridation of the substrates is considered to be employed prior to the growth in order to improve the qualities of the InN epilayers.

In this paper, we have grown the InN epilayers on sapphire substrates by PAMBE and studied the effect of nitridation on the crystallographic, morphological, electrical and optical properties of InN layers. Moreover, the energy band-gap of InN layers were also determined by optical absorption and photoluminescence (PL) measurement and found to be around 0.75-0.80 eV.

The growth system used in this study is a PAMBE system equipped with a radio frequency plasma source. The base pressure in the system is about 1×10^{-10} mbar. The sapphire substrates were thermally cleaned at 800 \degree C for 1 h in ultra-high vacuum. For Sorresponding author. Tel.: +86 183 4300 0484.

E-mail address: lwc9442@126.com (W Li) **Sample InN-s** (for InN on sapphire) the substrate was without

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Fig. 1. (a) ω – 2 θ XRD scans of InN films for samples InN-s and InN-ns. (b) ω -scans of the (0002) reflection of InN films for samples InN-s and InN-ns.

nitridation and for sample InN-ns (for InN on nitrided sapphire) the substrate was employed with nitridation by exposing the substrates surface to RF nitrogen plasma for 1 h under the Nitridation flow rate and plasma power of 0.2sccm and 400w, respectively. Afterwards, for both the samples, low temperature InN buffer layer of thickness of \sim 20 nm were grown at 400 °C followed by 160–180 nm thick InN epilayers at 450 °C. The nitrogen flow rate, In source temperature and plasma power were kept constant at 0.3 sccm, 650 \degree C and 400 w for both buffer layer and InN epilayers. The structural characterization and surface morphologies of the samples were carried out by XRD and Reed, SEM, AFM, respectively. The films'electrical properties were measured by Hall system and the optical properties were characterized by SHIMADZU UV-360 Spectrophotometer and 325 nm He-Cd laser. All the measurements were performed at room temperature (RT).

XRD ω – 2 θ and ω -scans of the InN (0002) reflection for samples InN-s and InN-ns were shown in Fig. 1. Both the samples showed the prominent peaks of InN film and sapphire substrate. The InN fims were preferentially oriented in the c-axis direction. In addition, the intensity of the (0002) peak of InN films for sample InN-ns was much stronger than that of sample InN-s and the full width at half maximum (FWHM) of w-scans of the InN (0002) reflection for sample InN-ns was smaller than that of sample InN-s, which were \sim 0.17 \degree and 0.28 \degree , respectively. This indicates that the crystalline qualities of InN films grown after nitridation are much better than that of InN films grown without nitridation. According to M. Losurdo et al. [\[14\]](#page--1-0) the mechanism of nitridation on c-plane sapphire is a conversion from the bare sapphire surface to AlN and/or AlN_xO_y layers, which indicates less lattice mismatch for the subsequent growth of InN layers. On the other hand, this is probably due to the increased surface mobility of the adatoms, which increases the probability of their finding lattice sites with minimum energy [\[15\]](#page--1-0). And this conforms to the enhancement of the electrical properties found in Hall results. Moreover, the FWHM of w-scans of the (0002) reflection for InN film grown after nitridation in our study is comparable to the reported value of InN film grown on GaN underlayer by PAMBE [\[16\]](#page--1-0).

Reflection high-energy electron diffraction (RHEED) patterns, which were taken at the end of the growth for samples InN-s and InN-ns were shown in Fig. 2. The dim streaky cum spotty RHEED pattern observed for sample InN-s reveals the signature of a rough surface, whereas the observed clear streaky pattern for sample InNns indicates a smooth surface. This also can be seen in the results found in SEM and AFM images as shown in [Fig. 3](#page--1-0).

SEM photographs showing top-down views and cross-sectional of InN films for samples InN-s and InN-ns were shown in [Fig. 3\(a\).](#page--1-0) The surface morphology of sample InN-s was obviously influenced by the large lattice mismatch, which was rough and showed coalesced islands. On the other hand, the film surface of sample InN-ns was smooth, had better uniformity, and illustrated twodimensional mode growth. This is probably due to the less lattice mismatch for the films of InN grown after nitridation and larger surface mobility of the adatoms. The surface morphologies of the InN films for samples InN-s and InN-ns were also carried out by atomic force microscopy (AFM) as shown in Fig. $3(b)$. The RMS roughness of the InN surface for samples InN-s and InN-ns, calculated from 1×1 um² AFM scans, were 26.5 and 5.37 nm, respectively.

Fig. 2. RHEED patterns of InN films for samples InN-s and InN-ns taken at the end of growth.

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