Studies of Si Doped AlN Layers for n-type Electrical Conductivity

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We have grown high quality AlN layers on c-plane sapphire substrates in our LP MOVPE system. We doped the AlN layers with Si to obtain n-type electrical conductivity. We observe that the Si incorporation leads to the degradation of surface and crystal quality as well as in-plane tensile stress of the AlN layer. Finally, we have obtained a fair electrical conductivity for a Si doped AlN layer which has a Si concentration of $1.5 \cdot 10^{18}$ cm^{-3}. By room temperature Van der Pauw Hall measurements, we have found: electron carrier concentration of $4 \cdot 10^{14}$ cm^{-3}, carrier mobility of 30 cm^2 V^{-1} s^{-1}, and resistivity of 530 Ω cm. By varying the temperature, an activation energy of about 200 meV could be estimated. Meantime, we observe that the electrical conductivity is correlated with the intensity ratio between the deep level transition at 3 eV and the near-band-edge luminescence of low temperature CL spectrum.

1. Introduction

Recently, aluminum nitride (AlN) has attracted much attention due to its extremely large direct bandgap (approx. 6.0 eV) and outstanding thermal and chemical stability. AlN and $Al_xGa_{1-x}N$ ternary alloys have a wide application perspective especially in the area of high-power high-temperature electronic and UV region optoelectronic devices [1–3]. For such device applications, intentional doping is essential to achieve sufficient electrical conductivity and control carrier concentrations. However, very limited reports have been so far published on the transport phenomena and properties of intentionally doped pure AlN and high Al content $Al_xGa_{1-x}N$ ternary alloys compared to GaN or InGaN, due primarily to the difficulties on the epitaxial growth of high quality layers of these materials [4–7]. Especially the effective and efficient doping of such materials to obtain the required electrical conductivity for practical AlN based devices remains a key issue for the nitride researchers. The major issues associated with poor electrical conductivity of AlN are the high ionization energy of dopants, solubility, and compensation by native defects and unintentionally incorporated background impurities. In this report, we present the results of a high quality undoped AlN bulk layer and the effect of Si doping on structural and spectroscopic properties. We also report the effect of [Si] on the intensity of deep level transitions in low temperature (10 K) cathodoluminescence (CL) spectra, which are presumably due to Al vacancies and/or of related complexes [7], and the electrical conductivity of the corresponding sample.

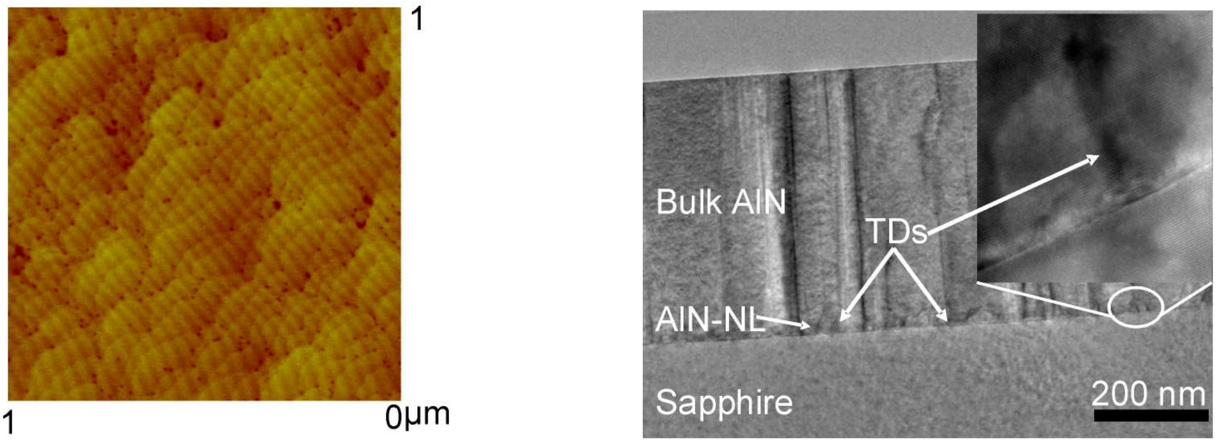


Fig. 1: AFM image of the optimized AlN layer grown at 1190 °C (left). Cross sectional bright field TEM image illustrating threading dislocations (TDs) (right).

2. Experimental

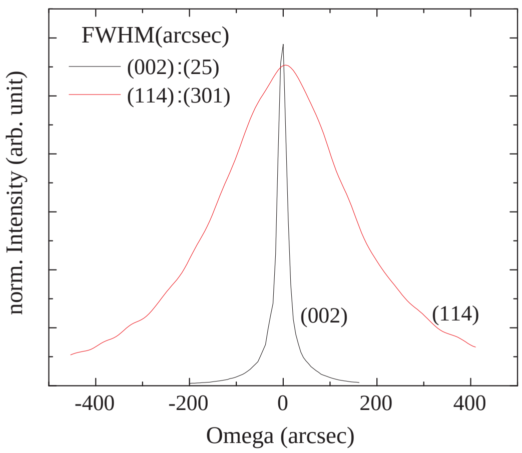
We deposited approx. 500 nm to 1 µm thick AlN layers on *c*-plane sapphire substrates in an AIXTRON AIX 200 RF LP–MOVPE system at a growth temperature of 1190°C and a pressure of 35 mbar in N_2 and H_2 ambient. Trimethylaluminum (TMAl) and NH_3 were used as group III and V precursors, respectively and H_2 was used as a carrier gas. The details of the growth process are described elsewhere [8, 9].

Si doping, by using silane (SiH_4), was performed by typically depositing a 350 nm thick Si-doped AlN layer on a 250 nm thick optimized undoped AlN buffer layer. The basic growth conditions of the Si-doped layers were similar to those of the undoped buffer layers.

The surface quality was analyzed by using atomic force microscopy (AFM). High-resolution X-ray diffraction (HRXRD) rocking curve measurements (ω scan with open detector) were carried out to examine the crystal quality of AlN epitaxial layers. Low temperature (T=10 K) CL provided information about the spectroscopic properties. Varying temperature two and four point I–V, and room temperature Van der Pauw Hall measurements were carried out to measure electrical properties. The Si concentration [Si] was extrapolated from our growth parameters using other AlN samples as reference which had been measured by secondary ion mass spectroscopy (SIMS) [10].

3. Results and Discussion

We obtained an atomically flat surface of the undoped AlN layer with a measured rms surface roughness of 0.2 nm as shown in Fig. 1 (left). A large number of networks of small pits is also visible on the surface which can be related with the threading dislocations as seen on the cross sectional TEM image (Fig. 1 (right)). The density of such small pits is approximately $1 \cdot 10^{10}\,\mathrm{cm}^{-2}$. These small pits may reflect the contribution of all types of threading dislocations present in the layer as confirmed by plan-view TEM. However, the density of large hexagonal pits (diameter more than 50 nm) on the surface was significantly reduced to less than $10^4\,\mathrm{cm}^{-2}$. The FWHM of the X-ray rocking curve for symmetric (002)



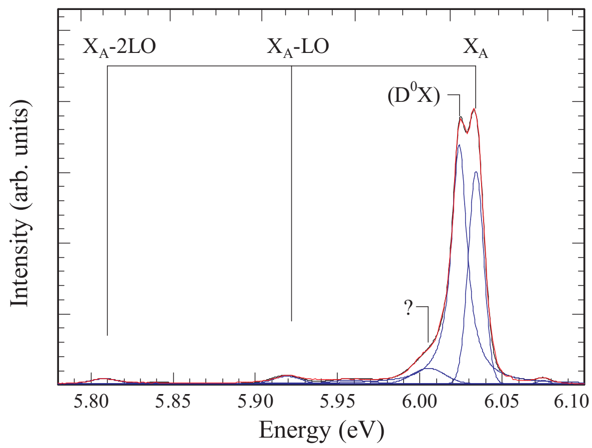


Fig. 2: HRXRD rocking curve measurements of an 1 µm thick undoped AlN layer illustrating FWHM of symmetric (002) and asymmetric (114) reflections (left). Low temperature (10 K) CL spectra of the undoped AlN layer (right) illustrating the FWHM of the near band-edge CL peaks to be 10 meV.

and asymmetric (114) reflections are 37 and 338 arcsec, respectively. Similar as described by others [11], we observed a further improvement of our undoped AlN layer by growing a thicker layer of about 1 µm. The FWHM of the X-ray rocking curve for (002) and (114) reflections of this sample is 25 arcsec and 300 arcsec, respectively (Fig. 2 (left)). The high quality of our AlN layer was further confirmed by the observation of a very narrow near band-edge excitonic emission. The two main contributions exhibit only a FWHM of about 10 meV determined from a lineshape analysis of the emission peak shown in Fig. 2 (right). The two lines at 6.024 and 6.034 eV are assigned to a donor bound (D_0X) and the A free exciton (X_A) transition, respectively. This assignment was proven by analysing temperature dependent CL measurements as shown for similar samples in [12]. The position of the A free exciton transition (X_A) indicates that our AlN layer is almost unstrained.

We studied the effect of Si doping on the structural and spectroscopic properties of the AlN layer. Similar to our previous report [9], we again found an adverse effect of Si doping on the surface and crystal quality of our lastly optimized AlN layers. The degradation of the surface and crystal quality may be related to the evolution of new defect networks in the Si doped area. TEM investigations show the emergence of pure screw and/or mixed type threading dislocations with Si introduction. However, the edge type threading dislocations, which run throughout the layer thickness, are indifferent to the Si incorporation. Figure 3 (left) shows a narrow near band-edge excitonic emission having a FWHM of about 20 meV of a Si doped sample having [Si] of $1.5 \cdot 10^{18}\,\mathrm{cm}^{-3}$ by low temperature (10 K) CL measurement. The luminescence peak is resolved into two transitions at 5.962 eV and 5.986 eV for a donor bound exciton, (D_0X), due to Si donors and the A free exciton (X_A), respectively. The position of the A free exciton transition (X_A) indicates that our AlN layer is strained. Furthermore, as reported in [9], the HRXRD evaluations and the red shift of the near band-edge CL peaks of samples having different [Si] show an in-plane tensile strain up to [Si] of $2 \cdot 10^{19}\,\mathrm{cm}^{-3}$. The appearance of tensile strain in these samples was also observed in Raman studies of the E_2- mode, which showed a shift towards lower wavenumbers (see e.g. [13]). Hence, it is confirmed that Si doping up to the concentration of approx. $2 \cdot 10^{19}\,\mathrm{cm}^{-3}$ leads to tensile strain within the AlN layer. Additionally, in CL measurements, we observe deep level transitions at about 3 eV and 3.5 eV which presumably are due to Al vacancies and of respective complexes [7,14]. The intensity ratio between the deep level transition at 3 eV and the near band-edge luminescence is changing with [Si] as shown in Fig. 3 (right). We observe a minimum intensity ratio for the sample having a Si concentration of $1.5 \cdot 10^{18}\,\mathrm{cm}^{-3}$. By room temperature Van der Pauw Hall measurements, we have found fair electrical conductivity for this sample: electron carrier concentration of $4 \cdot 10^{14}\,\mathrm{cm}^{-3}$, carrier mobility of $30\,\mathrm{cm}^2\mathrm{V}^{-1}\mathrm{s}^{-1}$, and resistivity of $530\,\Omega\,\mathrm{cm}$. By varying the temperature, an activation energy of about 200 meV could be estimated. Therefore, we observed that best electrical conductivity is correlated with a minimum intensity ratio between the deep level transition at 3 eV and the near band-edge luminescence. Higher Si doped samples having a larger intensity ratio are highly resistive. This is basically due to the acceptor-like type of the Al vacancies which are compensating the Si donors. Hence, it is imperative to reduce Al vacancies and the respective complexes to obtain desirable electrical conductivity in AlN. Reducing, especially, the edge type threading dislocations could help to improve the lateral conductivity.

4. Conclusion

We obtained high quality AlN layers having rms surface roughness of 0.2 nm, FWHM of HRXRD for (002) and (114) reflections of 25 and 300 arcsec, respectively. This excellent quality is further confirmed by low temperature CL spectra with a FWHM of the donor bound exciton peak of 10 meV. We found that the surface and the crystal quality is degraded by Si doping. HRXRD and low temperature CL measurements show increasing in-plane tensile stress up to Si concentrations of approx. $2 \cdot 10^{19}\,\mathrm{cm}^{-3}$. From low temperature CL measurements, we observe that the intensity ratio between the deep level transition at 3 eV and near-band-edge luminescence is changing with [Si] and it is minimum for the sample having [Si] of $1.5 \cdot 10^{18}\,\mathrm{cm}^{-3}$. This sample shows a fair n-type conductivity at room temperature: electron carrier concentration of $4 \cdot 10^{14}\,\mathrm{cm}^{-3}$, carrier mobility of $30\,\mathrm{cm}^2\mathrm{V}^{-1}\mathrm{s}^{-1}$, resistivity of $530\,\Omega\mathrm{cm}$, and activation energy of about 200 meV for increasing temperature.

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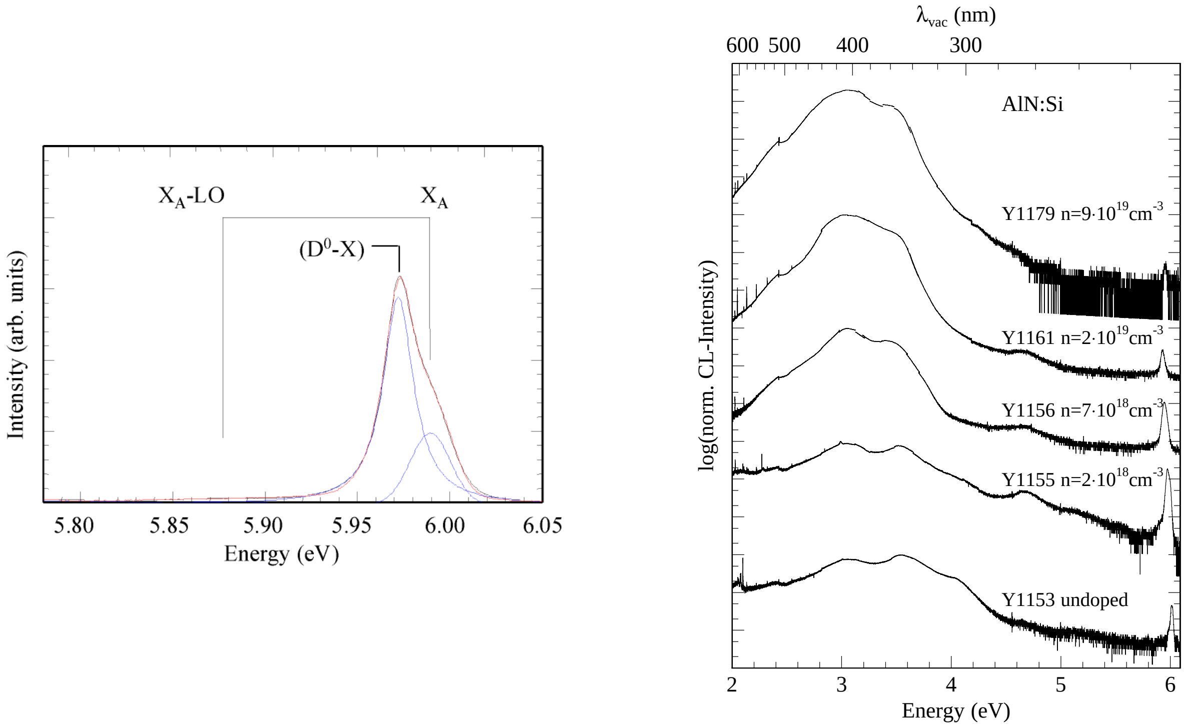

Fig. 3: Low temperature (10 K) CL spectra of a Si doped layer illustrating the FWHM of the CL peak approx. to be 20 meV (left). Overview of low temperature (10 K) CL spectra for the Si doped and the undoped samples (right). The peak at 3 eV increases in intensity for higher doped samples. The intensity ratio between the peaks at 3 eV and near band-edge luminescence is minimum for [Si] $1.5 \cdot 10^{18}\,\mathrm{cm}^{-3}$

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