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Excimer laser annealing: An alternative route and its optimisation to effectively activate Si dopants in AlN films grown by plasma assisted molecular beam epitaxy

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ABSTRACT

Si doped AlN films were grown on GaN/Sapphire templates by plasma assisted molecular beam epitaxy (PAMBE) technique. We show that employing an excimer laser annealing with optimized power and frequency rather than the conventional thermal annealing could be a potential alternative route towards improving the structural and electrical properties of AlN layers. Upon optimized laser annealing of the Si-doped AlN layer, the electron concentration was achieved to be as high as $\sim 4.9 \times 10^{18}$ cm^{-3} which was measured by Raman spectroscopy measurement and was further cross checked by standard Hall measurement which estimated the same as $\sim 7.4 \times 10^{18}$ cm^{-3} with a mobility of 109 cm^2/V-sec. The improvement of free carrier concentration was leveraged by improvement of structural properties. The r.m.s surface roughness of the Si-doped AlN layers measured by atomic force microscopy was reduced to 0.76 nm and corresponding residual stress estimated by high resolution XRD and Raman measurement was found to be less than half compared to the in-situ Si-doped sample. Thus laser annealing is proposed to be a suitable method to achieve high electron concentration in Si doped AlN films without compromising the structural quality.

1. Introduction

AlN has the widest bandgap (~6.2 eV) among all the materials in the group III-Nitride family. Along with wide band gap, other properties of AlN viz. high thermal conductivity and stability, low thermal expansion coefficient, high dielectric breakdown field [1,2] etc. make it one of the most promising materials for application in deep UV light emitters [3], photo detectors [4] and heterojunction diodes [5] etc. However, in order to realize AlN based UV devices, one of the basic requirements is to achieve high n/p-type conductivity which has been a great challenge to the community due to the various fundamental reasons.

Si is primarily being used as the n-type donor for GaN or AlGaN with moderate percentage (~40%) of Al. Si forms substitutional shallow donor level at Ga or Al sites [6]. However, when it comes to pure AlN, irrespective of the growth method it has been widely found that the free carrier density in Si doped AlN layer has been significantly low ($< 10^{17}$ cm^{-3}) [7,8]. This is primarily attributed to the Si induced formation of DX centres or deep defects like nitrogen vacancies or impurity oxygen, that create deep donor states down to 300 meV from conduction band edge in AlN [9]. Further, large donor ionization energy in Si doped AlN has been another problem. For the layer grown using metalorganic chemical vapor deposition (MOCVD), the donor ionization energy is reported to be varying from 180 meV to 254 meV [7], whereas for molecular-beam epitaxy (MBE) technique the same has been reported to be 320meV [8]. The high donor ionization energy could be explained as the compensation of the shallow donor states Si_{Al} by triple acceptor Al vacancies whose formation energy has been estimated to be very low thus omnipresent in n-type AlN [10]. In contrary to the above results and previous theoretical predictions [8] which infer that it is impossible to produce semiconducting AlN, few groups across the globe were able to produce high quality epitaxial AlN film with n-carrier density as high as 10^{18} cm^{-3} [11,7].

Thus Si induced defects and also dislocation densities [12] act as recombination centres that decrease the free carrier concentration in AlN films. Annealing at sufficiently high temperature could be one of the methods to annihilate these defects and stress within the film so as to increase the availability of the free carriers in the film. In the conventional thermal annealing, dopant activation in AlN layer is usually carried out around a temperature of 1500 °C and above which is considered to be quite high with regard to its structural stability as in previous studies on thermal annealing it has been found that either of

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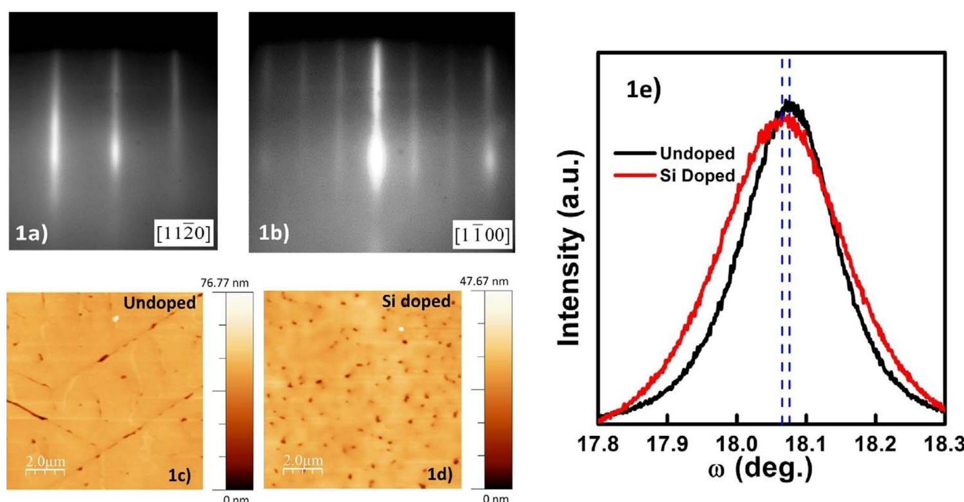

Fig. 1. a) Shows streaky RHEED pattern of AlN film (doped and undoped) along [11–20] azimuth and b) shows the same along [1-100] azimuth during growth interruption period. The superstructures confirm that the AlN surface exhibits (1 × 3) pattern. c) and d) show the 10 × 10 μm^2AFM images of surfaces of the undoped and doped samples respectively. e) shows the ω-scans around (0002) XRD peak of both the samples.

the surface roughness [13], crystal quality [13,14] or residual stress [15] deteriorates significantly with increasing annealing temperature. However, in the laser annealing process much higher temperature could be achieved instantaneously and locally by applying pulsed laser to the sample kept at room temperature. In this paper, we discuss the effect of laser annealing on the structural and electrical properties of Si doped AlN film. We show that an optimization of annealing condition is required in order to achieve maximum free carrier density in the film. Several AlN films were grown with varying Si cell temperature for different level of doping incorporation. However, for the interest of the present paper we will discuss only the growth and subsequent characterizations of an undoped AlN film and an AlN film grown with Si doping with Si cell temp of 1200 °C.

2. Experimental procedure

The Al-face AlN films were grown on c-plane oriented Ga-face GaN semi-insulating templates (grown on sapphire substrate by MOCVD, procured from Lumilog, France) in a RIBER C-21 MBE growth chamber (with a base pressure of 6×10^{-11}Torr) equipped with conventional effusion cells for group-III solid sources (Ga, Al, In) and dopant Si. A RIBER ADDON radio frequency plasma source was used for the active N species. The epi-ready substrates were at first thermally cleaned at 810 °C at UHV condition. Both the undoped and doped AlN films were grown at 800 °C wherein the RF power of the plasma cell was fixed at 400W and the N_2 flow was maintained at 0.41 sccm so that the chamber remained at 6.8×10^{-6} Torr background pressure throughout the growth duration. During the growth of the undoped sample the growth was initiated by opening the shutters of both Al and RF source simultaneously. The growths were done in slightly Al rich condition with beam equivalent pressure of Al fixed at 8.2×10^{-8}Torr. So after every 3 min of growth, Al beam was being interrupted for 50 s to allow the nitrogen to consume the extra Al at the growth front so that no metallic Al is segregated in the film. During the growth of the Si doped sample, the Si cell temperature was set at 1200 °C and the Si cell shutter was kept open only when the Al shutter used to be open. The thicknesses of the films were around ~500 nm while the growth rate was maintained at 2 nm/min in all the cases.

The Si doped sample was then characterized and compared with the undoped sample which is described in the Section 3.1. The doped sample was then annealed using an excimer laser at different conditions for optimizing the annealing process in order to achieve the best possible carrier concentration while retaining the crystal quality of the sample. The laser annealing chamber was initially pumped down to a low pressure (3×10^{-5}Torr) with the sample at the target position and then the chamber was brought to atmospheric pressure with 5N nitrogen in order to perform the annealing in nitrogen ambient. An ArF excimer laser with a wavelength of 193 nm was used for annealing. The laser spot size was adjusted to an area of 2.2 mm x 7.2 mm. The energy, voltage and frequency of the laser were set at 80 mJ/cm^2, 24 kV and 5 Hz respectively. The pulse duration of the laser was 10 ns. Three different pieces of the same doped sample (E16) were annealed with 300, 600 and 1500 shots of laser respectively. They will hereafter be designated as E16_300, E16_600 and E16_1500 respectively.

In-situ, real time reflection high energy electron diffraction (RHEED) characterization was performed with the STAIB system during the growth of all the samples under consideration. The samples were also characterized by several ex-situ diagnostic tools. In a RIGAKU smartlab high resolution x-ray diffraction (HRXRD) system (in which an x-ray source with Cu Kα radiation is provided with a Ge (220 × 2) 2-bounce monochromator to filter the Cu Kα1 (λ = 1.54083 Å) radiation only as the incident beam), ω-scans were performed around both the symmetric and asymmetric XRD peaks which elucidate the global structural quality of the doped as grown and annealed layers. Atomic force microscopy (AFM) was performed in contact mode in an Oxford Instrument Asylum system to obtain the surface morphology of the films. Finally the trend of the carrier concentration of the films was determined by a Lakeshore Model 8404 AC/DC Hall Effect Measurement system. The nature of variation of the carrier concentration obtained by the Hall measurement was further confirmed by Raman spectroscopy which was performed in a Horiba Scientific Raman spectrometer featuring an Ar^+ laser source of 514.5 nm for Raman excitation and a thermocouple cooled charge coupled device array attached to a spectrometer for detection with a spectral resolution of 0.2 cm^{-1}.

3. Results and discussion

3.1. Results of as grown undoped and doped sample

The as grown undoped and doped samples were at first compared to find out the effect of in situ Si doping in AlN films. During the growth, the RHEED pattern was (1 × 1) but during the growth interruption a (1 × 3) RHEED pattern was observed as shown in Fig. 1(a) and (b). It is due to a $(\sqrt{3}$x $\sqrt{3})$R30° reconstruction resulted from 1 or 2 monolayer thick Al at the growth front which confirms that the growth has taken place in slightly metal rich regime[16] as excess Al which causes metal segregation leads to different reconstruction. Same reconstruction (as in Fig. 1) was observed for both the films which infers that the chosen concentration of Si doesn't alter the growth front stoichiometry. Fig. 1(c) and (d) show the AFM images of the undoped and doped films respectively. Fig. 1(e) shows the ω-scans around (0002) XRD peak for

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