Erbium-doped AlN epilayers synthesized by metal-organic chemical vapor deposition

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Abstract: Erbium doped AlN epilayers (AlN:Er) have been grown by metal organic chemical vapor deposition. The 1.54 µm emission properties were probed by photoluminescence (PL) emission spectroscopy and compared with those of GaN:Er. Optimum intensity of the 1.54 µm emission from AlN:Er was obtained for growth temperature at 1050 °C. It was found that the emission intensity from AlN:Er is higher than that from GaN:Er for above and below energy gap, as well as resonant excitation. A significant narrowing of the infrared Er^{3+} PL lines was observed when pumping resonantly into an intra-4f transition. The integrated intensity of the 1.54 µm emission shows a decrease by a factor of only 1.2 between 10 K and 300 K.

References and links

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1. Introduction

Growth of semiconductor hosts doped with rare-earth (RE) atoms stimulated great interest due to the potential applications of RE doped semiconductors as light emitters and optical amplifiers. Rare earth elements have partially filled 4f shells which are screened by 5s and 5p electronic states. Due to their shielded 4f levels, the spectral line shape of rare earth ion emission is insensitive to the host semiconductor. Out of the many RE elements, erbium (Er) is the most widely investigated element in semiconductor doping, because of its $^4I_{13/2}$ to $^4I_{15/2}$ transition at 1.54 μm. This transition is of great interest in the field of communications because it lies in the minimum loss region of silica fibers used in optical communications [1–6].

However, it is well established that the emission intensity at room temperature of the Er^{3+} ions depends strongly on the band gap of the host semiconductor. Er emission from smaller band gap semiconductors has low efficiency at room temperature due to a strong thermal quenching effect [7,8]. Furthermore, it has also been suggested that the environment created by more ionic hosts enhances the Er emission efficiency [9,10]. Therefore, the III-nitride wide band gap appears to be excellent host for Er. Optical emission at 1.54 μm in Er-doped GaN epilayers has been observed. Er doped GaN has demonstrated a highly reduced thermal quenching of the Er luminescence intensity as compared to other semiconductor host materials such as Si and GaAs [11,12]. AlN has a bandgap (6.1 eV) compared to (3.4 eV) of GaN and AlN has a larger electronegativity difference (−1.4 compared to −1.2 for GaN) [13]. Therefore, it is expected that AlN as a host material will result in lower thermal quenching of Er emission. AlN is of great technological significance because of its physical and chemical properties such as low dielectric constant, excellent thermal conductivity, electrical resistivity, excellent mechanical strength and chemical stability [14]. These reasons make AlN an excellent host material for device applications.

The luminescence from Er doped AlN has been investigated by several groups in the past few years. Different growth methods have been used to incorporate Er into AlN such as ion implantation [15], metal-organic molecular beam epitaxy [16], reactive radio frequency magnetron sputtering [17]. Er doped AlN obtained by *in situ* incorporation by metal-organic chemical vapor deposition (MOCVD) has not yet been experimentally realized, although AlN based deep ultraviolet light emitting diodes and photodetectors grown by MOCVD are of high quality [18,19]. In this work, we report on the MOCVD growth of Er doped AlN epilayers and their structural and optical properties.

2. Experimental details

Er doped AlN epilayers were deposited on sapphire substrates using MOCVD system. The aluminum source was trimethylaluminum (TMA) and the nitrogen source was ammonia (NH_3).Trisisopropylcyclopentadienylerbium (TRIPEr) was used for the *in situ* Er doping. Hydrogen was the carrier gas and was kept constant at 2 standard liters per minute. The growth started with double buffer layers consisting of a thin (30 nm) AlN buffer layer (buffer 1) grown at 950 °C and 30 mbar followed by a second 100 nm AlN buffer layer (buffer 2) at 1100 °C grown at 30 mbar [20], and a 1.0 µm AlN template grown at 1300 °C and 30 mbar followed by a 0.5 µm Er-doped AlN epilayers grown at 1050 °C and 30 mbar. In situ spectroscopic reflectance was used to determine the growth rate and the thickness of epilayers. X-ray diffraction (XRD) was used to determine crystalline quality, while atomic force microscopy (AFM) was used to study the surfac. A photoluminescence (PL) spectroscopy was employed to study the optical properties of the Er-doped AlN epilayers under above and below bandgap excitation. The PL system consists of a frequency doubled, tripled, and quadruple 100 femtosecond Ti: Sapphire laser with an average power of 150 mW at 263 and 395 nm, 1 mW at 196 nm, and a repetition rate of 76 MHz. Infrared detection was accomplished by an InGaAs detector. For resonant excitation, a 980 nm laser diode was used as the excitation source.

3. Results and discussion

Figure 1(a) shows a schematic diagram for the general layer structure of Er doped AlN epilayer grown on 1 µm undoped AlN template on sapphire substrate. Figure 1(b) shows the in situ optical reflectance curve of optimized AlN:Er epilayer grown on AlN template. There is nearly no change in the intensity of in situ optical reflectivity during the growth of the undoped AlN template epilayer. However, there is a little attenuation of the intensity during the growth of the AlN:Er epilayer. This indicates a quasi-two-dimensional growth mode and a smooth surface epilayers. The thickness of the Er-doped AlN epilayer is 0.5 µm as determined from the optical reflectance curve.
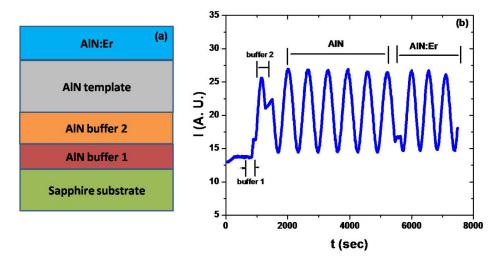

Fig. 1. (a) Schematic layer structure and (b) *in situ* optical reflection curve of the optimized Er doped AlN epilayer (AlN:Er) grown on AlN/sapphire template.

To determine the optimum growth temperature for AlN:Er films the growth temperature was varied from 950 °C to 1125 °C . The Er source temperature was fixed at 70 °C. Interestingly, all films exhibited PL emission at 1.54 µm regardless of growth temperature. Figure 2(a) shows the growth temperature dependence of the 1.54 µm PL spectra measured at

300 K for λ_{exc} = 375 nm of AlN:Er. It can be seen that the 1.54 µm emission line shape is independent of growth temperature. Figure 2(b) shows the PL intensity at 1.54 µm as a function of the growth temperature of AlN:Er. The PL intensity, which is proportional to the active Er concentration [21], increases for growth temperatures from 950 °C to 1050 °C and then starts to decrease for growth temperatures from 1075°C to 1125 °C.

XRD measurements were used to analyze the structural quality of the AlN:Er films. The XRD spectra of the films showed a peak position at 2θ ~36.0° for all growth temperatures. The variation of the full width at half maximum (FWHM) of the (0002) rocking curve is plotted in Fig. 2 (c), the figure indicates that the crystalline quality of AlN:Er films is nearly identical for different growth temperatures. Similar behavior was seen for the (102) rocking curves. AlN:Er epilayers grown by MOCVD show much better crystalline quality compared to AlN:Er films grown by magnetron sputtering[17]. This could be explained by higher growth temperatures used in MOCVD. This is also consistent with the fact that crystalline quality of undoped AlN epilayers grown by MOCVD is much better than those grown by magnetron sputtering. The targeted Er doping concentrations in all samples are the same, around 2×10^{20} atoms/cm³. Therefore, the 1.54 µm emission intensity variation is not related to the crystalline quality or the Er concentration with the growth temperature. However, the reason for the variation of the 1.54 µm emission intensity as a function of growth temperature can be partially explained by the variation of the concentration of the optically active Er ions. This indicates that the number of incorporated active Er ions in AlN:Er is a strong function of the growth temperature based on the proportional relation between PL intensity and the number of active Er ions.
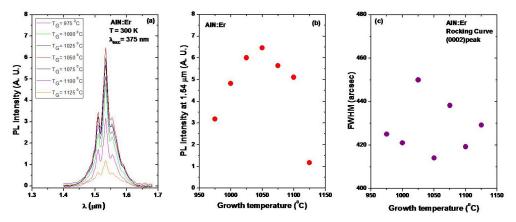

Fig. 2. (a) Room temperature (300 K) PL spectra near 1.54 µm of AlN:Er grown at different growth temperatures (975, 1000, 1025, 1050, 1075, 1100, and 1125 °C). The excitation wavelength used was 375 nm. (b) The PL intensity at 1.54 µm as a function of the growth temperature. (c) The full width at half maximum (FWHM) of the XRD (0002) rocking curve of AlN:Er as a function of the growth temperature.

Figure 3 shows the room temperature PL spectra of Er-doped AlN and the best Er-doped GaN. Both films have the same thickness and grown at the same Er molar flow rate. The PL in Fig. 3(a) was excited using 375 nm (λ_{exc} = 375 nm) laser, which corresponds to below-gap pumping for both AlN:Er and GaN:Er. Both films exhibited two emission peaks at wavelengths 1.0 and 1.54 µm, corresponding to the intra-4f Er^{3+} transitions from the ${}^{4}I_{11/2}$ (second excited state) and ${}^{4}I_{13/2}$ (first excited state) to ${}^{4}I_{15/2}$ (ground state), respectively. The PL intensity of the 1.54 µm peak from AlN:Er is slightly higher than that from GaN:Er. The full width at half maximum (FWHM) of the 1.54 µm peak is 53 and 30 nm for AlN:Er and GaN:Er, respectively. The PL intensity of the 1.0 µm from AlN:Er is sharply decreased compared to that from GaN:Er. The PL in Fig. 3(b) was excited using 195 nm (λ_{exc} = 195 nm) laser, which corresponds to above-gap pumping for both AlN:Er and GaN:Er. The PL

intensity of the 1.54 μm peak from AlN:Er is 1.45 times higher than that from GaN:Er. The FWHM of the 1.54 μm peak is 47 and 31 nm for AlN:Er and GaN:Er, respectively. The PL intensity of the 1.0 μm from AlN:Er is hardly seen compared to a strong peak from GaN:Er.
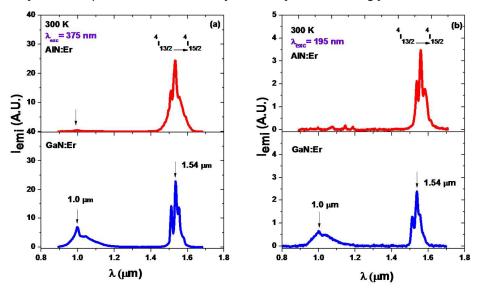

Fig. 3. (a) 300 K PL spectra near 1.54 μm of AlN:Er (above) and GaN:Er (below). The excitation wavelength used was 375 nm. (b) 300 K PL spectra near 1.54 μm of AlN:Er (top) and GaN:Er (bottom). The excitation wavelength used was 195 nm.

Due to the wide availability of high power semiconductor laser sources (λ = 980 nm) most of the commercial Er-doped fiber amplifiers (EDFAs) are pumped by diode lasers in this wavelength window. Furthermore, to use Er doped III-nitrides as a gain medium of 1.5 μm lasers, resonant pump using 980 nm lasers significantly reduces the quantum defect compared to using non-resonant pump. Practical application of optical amplifiers based on erbium-doped III-nitride will thus conceivably rely on 980 nm pumping [22]. Figure 4 compares the room temperature PL emission intensity probed at 1.54 μm of AlN:Er and the best GaN:Er epilayers under excitation with 980 nm laser (λ_{exc} = 980 nm), which corresponds to resonant pumping for Er in both AlN:Er and GaN:Er. AlN:Er exhibited an intra-4f transition at 1.54 μm two times stronger than that from GaN:Er. The FWHM of the 1.54 μm peak is 24 nm compared to 47 nm for AlN:Er excited with λ_{exc} = 195 nm. The narrowing of the 1.54 μm related emission PL lines when pumping resonantly into an intra-4f transition suggests that a specific class of Er^{3+} ions was selectively excited [23].

Figure 5 compares the PL spectra of the IR emission near 1.5 μm of AlN:Er for λ_{exc} = 263 nm at room temperature (above) and at 10 K (below). The 1.54 μm emission peak is dominant at low temperatures with its emission intensity decreases with an increase in temperature. The transition located at 1.51 μm is not visible at 10 K and its emission intensity increases with temperature following a different trend compared to the 1.54 μm peak. The total absence of the high energy contribution of the PL at 1.51 μm at 10 K has been observed before [24, 25]. The integrated intensity in the range 1.45 to 1.625 μm of the PL spectra at 10 K and 300 K shows a decrease from 1 to 0.83. This thermal quenching is smaller than the reported thermal quenching (20%) for GaN:Er [11, 12]. This indicates that Er emission at 1.54 μm in AlN:Er has a higher degree of thermal stability, attributing to its larger bandgap.
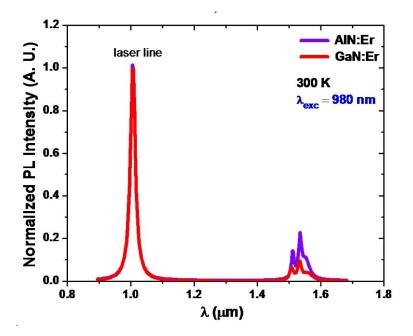
Fig. 4. Comparison of room temperature Er-related PL spectra near 1.54 µm between AlN:Er and GaN:Er epilayers under resonant pump by a 980 nm laser diode..
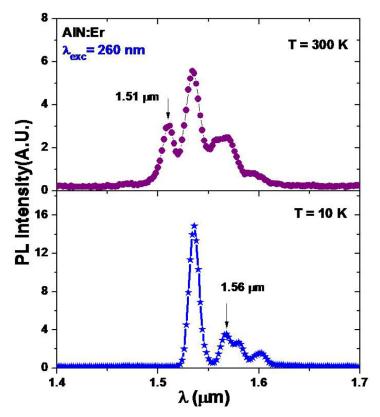

Fig. 5. PL spectra of the 1.54 µm emission of AlN:Er measured at different temperatures 300 K (top) and 10 K (bottom).The excitation wavelength used was 260 nm.

4. Conclusion

In summary, Er-doped AlN epilayers were synthesized with in situ doping by MOCVD. The influence of the growth temperature on the optical and structural properties of AlN:Er was investigated. The growth temperature of 1050 °C is the optimal temperature to obtain the highest PL intensity at 1.54 µm. PL results showed that both above and below band gap excitation produces emission at 1.54 µm from AlN:Er higher than that from GaN:Er. A significant narrowing of the infrared Er^{3+} PL lines was observed when pumping resonantly into an intra-4f transition. The Er emission at 1.54 µm in AlN has a high degree of thermal stability. These results demonstrate that Er-doped AlN has very high potential for applications in optoelectronic devices operating at the main telecommunication wavelength of 1.54 µm.

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