Epitaxial Growth of AlN on Sapphire by ECR Plasma Assisted MOCVD under Mirror Field Conditions

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Wurtzite-type aluminum nitride (w-AlN) films were epitaxially grown on sapphire substrates by electron-cyclotron-resonance plasma-assisted metalorganic chemical vapor deposition (ECRMOCVD) without low-temperature (LT) buffer layer. The epitaxial growth of AlN films was performed under a mirror field condition. Even without the low-temperature buffer layer, AlN epitaxial films with excellent crystallinity and surface flatness were grown at 1000 C. The variation in the ratio of the volume of epitaxial domains to that of non-epitaxial domains was also investigated.

I. INTRODUCTION

W-AlN is a wide band-gap semiconductor (Eg=6.2eV at 300 K), and is considered to be a very promising opto-electronic material for applications such as light-emitting diodes and lasers in the deep UV region [1, 2]. AlN is also used as a buffer layer for the GaN based laser fabricated on sapphire substrate [3]. Therefore, the detailed investigation of epitaxial growth of AlN on sapphire is very important subject even now. The temperature of epitaxial growth can be widely varied by using ECR plasma assisted MOCVD method, because stable nitrogen source gas such as NH_3 can be easily decomposed at low temperature [4]. In the present study, w-AlN was grown on sapphire substrates under a mirror field condition after thermal nitridation, using TMA and NH_3 as source gases.

II. EXPERIMENTAL

The ECR plasma CVD apparatus used in this study is comprised of an ECR plasma chamber, a growth chamber, a microwave circuit, a gas supplying system and an evacuating system. In addition to the two magnetic coils around the plasma chamber for the ECR field, a magnetic sub-coil was wrapped around the growth chamber. Details concerning the structure of the apparatus used in the present study have been reported in a previous paper [5]. The magnetic field gradient from the plasma chamber to the growth chamber was controlled by the current flow in the sub-coil. The mirror field was obtained by the sub-coil current flows in the same (I_{sc}>0) directions to the magnetic coils for ECR field. Under mirror field condition, the plasma space potential at 10mm from the substrate surface in the growth chamber decreased. Under low space potential, the degradation of the crystal structure of the epitaxial films is believed to be suppressed, due to the depression of the impingement of positive ions with high energy. Therefore, the epitaxial growth was performed under a mirror field condition (I_{sc}=40A).

AlN growth was carried out via the following three processes. (a) thermal cleaning of substrates in H_2 at 1050 C for 10min. (b) thermal nitridation of substrate surface with NH_3 at 1050 C for 10min. (c) epitaxial growth of AlN for various temperatures for 300min. Film thickness was approximately the same (0.8~1.0μm), except for the film grown at 1200 C (0.56 μm). In order to investigate the influence of the insertion of the LT buffer layer, thin AlN films were grown at 600 C for 5-20min before the epitaxial growth. The growth conditions of w-AlN are as follows: substrate sapphire (0001), pressure during the growth 2mTorr, NH_3 gas flow rate 15sccm, microwave power 340W. The V/III (NH_3/TMA) gas feed ratio during the growth was 200. The obtained AlN films were characterized by an atomic force microscopy (AFM: DI Nanoscope III) and by an X-ray diffractometer (RIGAKU RAD-IIR with a Ni filter).

III. RESULTS AND DISCUSSION

Figure 1 shows the dependence of the FWHM values of (0002) diffraction peak of θ-2θ scan (DS 1/6 , RS 0.15mm, SS 1/6) and of ω-rocking curve (DS 0.05mm, RS 0.6mm, SS open) on the growth temperature. The instrumental broadening has been subtracted by the deconvolution procedure using the diffraction spectra of sapphire substrates and Si wafers. The substrate temperature at 1000 C was best for the growth of AlN with excellent crystallinity and crystal orientation, as can be seen from the figure ($\Delta(2\theta)$=1.5min, $\Delta\omega$=7.1min). The $\Delta\omega$ of AlN films grown over LT buffer layer at 1000 C was about the same that grown at 600 C without LT buffer layer. By the insertion of LT buffer layer, it is considered that the crystal orientation of epitaxial films scatters due to the variation in the crystal orientation of AlN layer grown at the low temperature.

The small diffraction peaks from (10-10) and (10-11) planes, together with large peak from (0002), were also observed. The appearance of the (10-10) and (10-11) diffraction peaks indicates the existence of the domain whose c-axis is not parallel to that of the substrate (non-epitaxial domain). The ratio of the integrated intensity of (0002) to those of (10-10) and (10-11) was very large ($I_{(0002)}/\{(I_{(10-10)}+I_{(10-11)}\}$) at the growth temperature of 900~1000 C, as shown in Fig. 2. The inclusion of the non-epitaxial domains could be suppressed together with the improvement of the crystallinity and crystal orientation at appropriate growth temperatures.

The surface flatness varied with growth temperature as shown in Fig. 3. Average roughness measured by AFM was only 0.45nm at the growth temperature of 1000 C. Two-dimensional (2D) growth was enhanced when AlN was grown on sapphire substrate at 1000 C after thermal nitridation processes. On the other hand, three-dimensional (3D) growth was enhanced by the growth at high temperature (≧1200 C). The growth rate of AlN grown at 600~1100 C was almost the same (2.6~3.0nm/min). On the other hand, the rate of AlN grown at 1200 C was 30% smaller than that of the other films. This means that the reaction at 1200 C is governed by the thermodynamics between the epitaxial film and precursors. This rate limiting step may be responsible for the enhancement of the surface roughness.

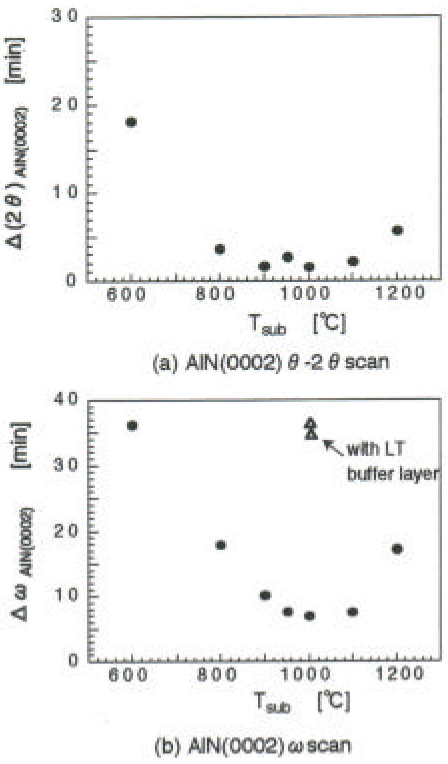

Fig.1 Variation in the FWHM values of (a) θ-2θ scan and (b) ω rocking curve as a function of substrate temperature.

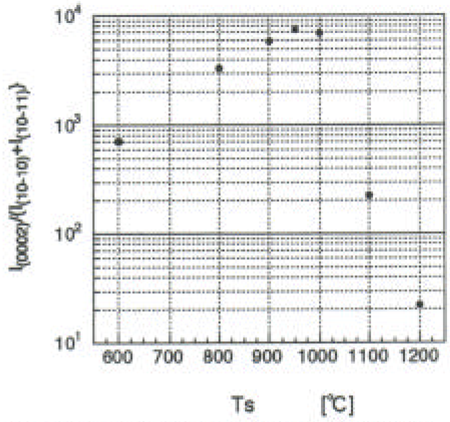

Fig.2 Dependence of the ratios of integrated diffraction intensity $I_{(0002)}/\{I_{(10-10)}+I_{(10-11)}\}$ of AlN on the substrate temperature.

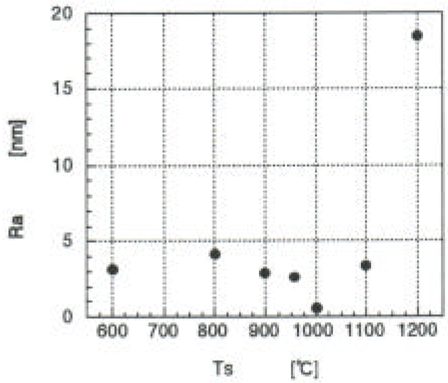

Fig.3 Variation in the average roughness of AlN films as a function of the substrate temperature.

IV. CONCLUSION

W-AlN films were epitaxially grown on sapphire substrates by ECRMOCVD method under a mirror field condition, using TMA and NH_3 as source gases. Even without the LT buffer layer, AlN epitaxial films with excellent crystallinity, crystal orientation and surface flatness were grown at 1000 C after thermal nitridation. The ratio of the volume of epitaxial domains to that of non-epitaxial domains was very large at the growth temperature of 900~1000 C.

V. REFERENCES

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