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Multiwafer atmospheric-pressure MOVPE reactor for nitride semiconductors and ex-situ dry cleaning of reactor components using chlorine gas for stable operation

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We describe an atmospheric-pressure MOVPE reactor with a capacity of 2 inch by 10 or 3 inch by 8. In this reactor, the parasitic reaction of particulate generation is suppressed by adopting a high-flow-speed design. As a result of suppressing of the parasitic reaction, we have also grown GaN at a growth rate of more than 28 µm/hr at atmospheric pressure. It is also important to grow a heterostructure of Al-containing alloys continuously to enable device applications, while maintaining the proper growth conditions for other layers such as GaN:Mg. An $Al_{0.09}Ga_{0.91}N$ layer has been grown at a growth rate of 1 µm/h at atmospheric pressure. In the production environment, it is also essential to operate a reactor under a stable condition. To this end, we have adopted ex-situ dry cleaning of all reactor components with deposits on them after every growth run. Dry cleaning was conducted using chemical etching of the deposit by chlorine gas in a hot tube reactor at about 800 °C.

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We describe an atmospheric-pressure MOVPE reactor with a capacity of 2 inch by 10 or 3 inch by 8. In this reactor, the parasitic reaction of particulate generation is suppressed by adopting a high-flow-speed design. As a result of suppressing of the parasitic reaction, we have also grown GaN at a growth rate of more than 28 µm/hr at atmospheric pressure. It is also important to grow a heterostructure of Al-containing alloys continuously to enable device applications, while maintaining the proper growth conditions for other layers such as GaN:Mg. An $Al_{0.09}Ga_{0.91}N$ layer has been grown at a growth rate of 1 µm/h at atmospheric pressure. In the production environment, it is also essential to operate a reactor under a stable condition. To this end, we have adopted ex-situ dry cleaning of all reactor components with deposits on them after every growth run. Dry cleaning was conducted using chemical etching of the deposit by chlorine gas in a hot tube reactor at about 800 °C.

1 Introduction Metalorganic vapour phase epitaxy (MOVPE) of GaN based heterostructures has been established as the most suitable technique for the fabrication of light emitting optoelectronics devices such as light-emitting diodes (LEDs) and semiconductor lasers (LDs) in the violet to green spectral range with historical first successful proof of the method [1, 2]. Since then, MOVPE of GaN has developed into a production-scale technology involving the use of large, commercial multi-wafer deposition reactors. The particular challenge in controlling the growth process lies in the occurrence of parasitic reactions between the metalorganic source materials and NH_3 leading to the formation of low volatility adduct species and subsequent gas-phase nucleation. We have been studying vapor-phase reaction of organometallics and NH_3 experimentally and by quantum chemical analysis [3, 4] To the best of our knowledge, there are no reports the effects of growth pressure on both AlGaN and Mg-doped GaN in the pressure range from 300 Torr to 760 Torr in the same reactor; at these pressures, significant parasitic reactions between organometals and NH_3 begin to occur [5]. It is believed that group-III reactants are removed from the gas flow by this mechanism, which thus affects the GaN growth efficiency. The situation is further aggravated by the preference to carry out device growth at atmospheric pressure. This is motivated by the observation of lower threading dislocation density [6], better magnesium activation [7] and lower background impurity concentration [8] at atmospheric pressure. On the other hand, it is known that the growth efficiency of GaN and AlGaN may be reduced at atmospheric pressure because of reaction kinetics, gas-phase nucleation of reaction by-products and subsequent depletion of reactants. In this paper, the results of growing GaN and AlGaN at atmospheric pressure using a commercial production-scale multiwafer reactor is reported. It is also known that the reactor must be operated under a stable condition for MOVPE of GaN, particulary in a production environment. To this end, after every growth run, ex-situ dry-cleaning [9] was conducted to clean all components with deposits inside the MOVPE reactor.

2 Experimental The GaN and AlGaN layers were grown using a commercial production-scale multiwafer reactor (Taiyo Nippon Sanso corp., SR23K). The reactor is capable 10×2 inch or 8×3 inch wafers at one time. We employed tri-gas flow injection with flow speed of more than 1m/sec at the wafer center. The gas injector was located at the center of a lotatable center plate, on which wafers were placed. Each wafer was also mechanically rotatable. The details of tri-gas injector were described in our previous paper [10] GaN and AlGaN layers were grown at atmospheric pressure throughout this study. All components inside the reactor were routinely cleaned afetr every growth run using an ex-situ dry-etching system (Taiyo Nippon Sanso Corp., DEX-100) to improve the yields of the epitaxy process. Ammonia (NH_3), trimethylgallium (TMG) and trimethylaluminum (TMA) were used as precursors. Hydrogen and nitrogen were used as carrier gases. To enhance the structural quality of the GaN buffer, we used c-plane sapphire with an off-orientaion of 0.15° towards the m-plane.

The crystallization and the aluminum composition of AlGaN film were evaluated by X-ray diffractometry (XRD), and the impurity concentration was measured with a SIMS measurement system.

3 Results and discussion Figure 1 shows the growth rate for GaN as a function of TMG concentration. In this reactor, the parasitic reaction of particulate generation is suppressed by adopting a high-flow speed design. As a result of suppressing the parasitic reaction, the growth rate was also increased proportionally up to 28 μm/h. The surface morhology of the grown sample was, even at the highest growth rate, smooth without pits or facets. If the parasitic reaction of particulate generation is not well suppressed, a problem in thickness uniformity arises in the case of atmospheric-pressure growth. The thickness uniformity in a 2 inch wafer was in the range from 0.5% to 0.7%.

Figure 2 shows the full width at half maximum (FWHM) of the X-ray rocking curve (XRD-RC) for the directions of both (0002) and (10-12) with the variation of the growth rate. The FWHM values correspond to the amount of screw dislocation and the amount of both edge and mixed dislocations, respectively. All samples were grown by conventional two step growth method. Each high temperature grown GaN was directory grown on a low temperature buffer layer. The FWHM value of the (10-12) direction suggests that the lataral over growth process in similar for the sample up to the growth rate of 12 μm/h, and the grain structure is more columner in the case of the highest growth rate of 28 μm/h.

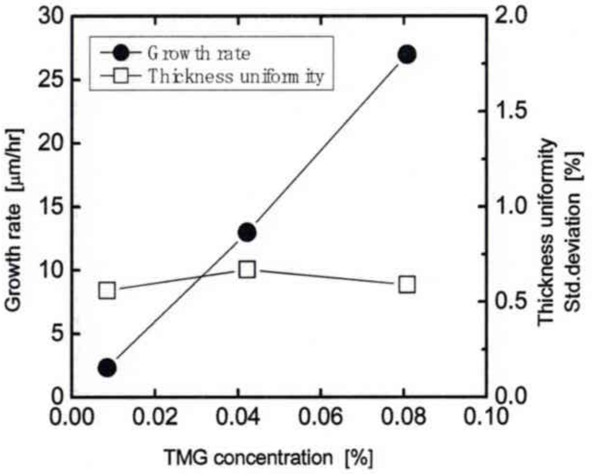

Figure 1 Growth rate of GaN as a function of TMG concentration. The solid lines are guids for the eye.

An additional information, keeping clean the reactor components in every run routinely using the ex-situ dry etching system was effective to improve the yields with a special focus on the stability of the system in a production environment. Also an influence of impurity contamination into a GaN layer was investigated by using SIMS measurement. The concentration level of [Si], [C] and [O] were almost same as GaN samples without ex-situ dry cleaning system.

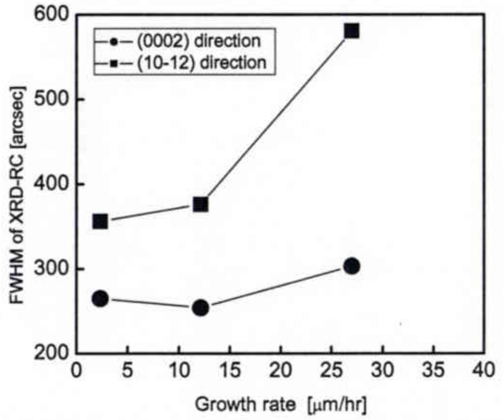

Figure 2 Full width at half maximum (FWHM) of X-ray rocking curve (XRD-RC) for the (0002) and (10-12) directions with variation of the growth rate. The solid lines are guides for the eye.

Figure 3 shows the growth rate of AlGaN as a function of the group-III precursor concentration. The growth parameters, such as growth temperature and carrier gas and NH_3 flow rates were kept constant except for group-III precursors. The partial pressure of TMA was changed in the range from 5% to 18%. The growth rate of AlGaN was increased proportionally to the partial pressure of group-III precursors. No parasitic reaction of particulate generation was observed under these growth conditions. Figure 4 shows the uniformity of thickness and the AlN content for AlGaN on a 2-inch wafer. The AlN content of 8.65% was obtained when the TMA partial pressure was 10%. A good thickness uniformity was obtained, which indicates that the decrease in the growth efficiency as a result of the parasitic reaction did not occur in this growth rate range, even at atmospheric pressure.

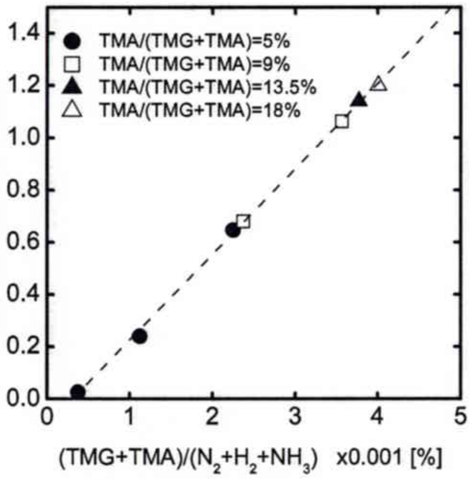

Figure 3 Growth rate of AlGaN as a function of group-III precursor concentration.

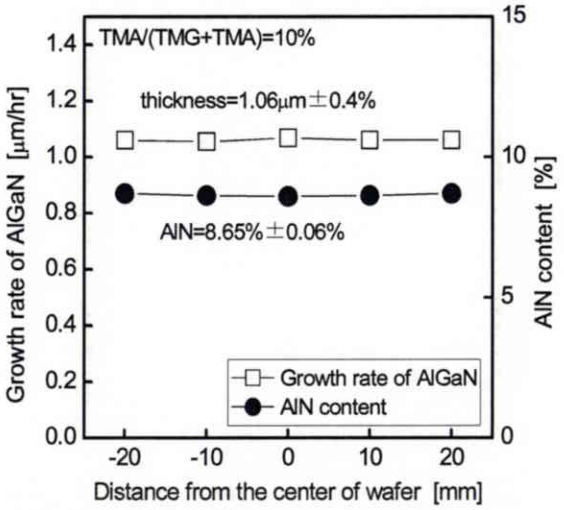

Figure 4 Uniformity of thickness and AlN content for AlGaN on a 2-inch wafer.

We routinely cleaned the reactor components after every run using the ex-situ dry-etching system so as to improve the yields, with particular focus on the stability of the system in a production environment. The wavelength variation of the LED structure at 400nm was tested with and without cleaning. The run-to-run wavelength shift was within 1nm regardless of the cleaning. This result suggests that the effect of wall deposition during a transient process from no deposition to partial deposition is negligible for the present system.

4 Summary We developed a circular gas flow reactor with a tri-gas injector and the capability to grow 10×2 inch or 8×3 inch wafers at one time. Undoped GaN was grown at growth rate up to 28μm/h. Also, we grow uniform $Al_{0.09}Ga_{0.91}N$ at a growth rate of 1μm/h. We did not observe any evidence of the parasitic reaction between TMA and NH_3 provided that Al content and the growth rate were lower than a certain value.

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