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# Growth of Solid Solutions of Aluminum Nitride and Silicon Carbide by Metalorganic Chemical Vapor Deposition

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#### Abstract

We report the growth of solid solutions of  $(AIN)_X(SiC)_{1-X}$  over the entire composition range from x=0.1 to x=0.9. We believe this is the first report of solid solution of  $(AIN)_X(SiC)_{1-X}$  by metal organic vapor deposition. Growth was performed in a low pressure vertical reactor using the silane-propane-ammoniatrimethylaluminium-hydrogen gas system on both silicon and silicon carbide substrates. Growth temperatures were between 1200-1250°C and growth pressures varied between 10 and 76 Torr. The composition of the solid solutions were strongly dependent on system pressure.

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### 1.0 Introduction

One of the material innovations of the late 70's was the ability to produce high quality heterojunctions in the III/V system. This development was responsible for the realization of several novel device structures, among them HEMTs (high electron mobility transistors) and HBTs (heterojunction bipolar transistors). It is now possible to have a similar development in the SiC-AlN wide bandgap system. In 1978 [1], it was shown that continuous solid solutions existed between AlN and SiC. Since then, the properties of  $(AlN)_X(SiC)_{1-x}$  such as lattice parameter, linear expansion coefficient and micro hardness have been studied [2]. Single crystal epitaxial layers of the  $(AlN)_X(SiC)_{1-x}$  solid solutions suitable for semiconductor applications have been grown [3-9]. It is important to note that for epitaxial grown  $(AlN)_X(SiC)_{1-x}$  a transition point from indirect gap structure to direct gap structure occurs at an AlN concentration about 70 mol% [8]. In addition, solid solutions with both p and n type conductivity have been reported [7,9]. We report on growth of the entire range of solid solutions by MOCVD.

## 2.0 Experimental procedure

In this study, solid solutions of  $(AIN)_X(SiC)_{1-x}$ , pure SiC and AlN samples where grown in a commercial low pressure vertical reactor. The details of SiC growth using this reactor have been reported [10]. In addition the details of the growth of AlN using TMA and NH3 have also been discussed [11]. The majority of the epitaxial films in this study where grown on Si (100) substrates, although successful growths were also accomplished on SiC (0001) substrates. Growths where performed at substrate temperatures of 1200-1250°C. The reactant gases used were silane (5.07% dilution in H2), propane (4.52% dilution in H2), ammonia (at either 5%, 20% or 100% concentration in H2), and

trimethylaluminum (the TMA bubbler temperature was 24.5°C) carried by H2. In one set of experiments the flow rates of SiH4 was varied between 7 and 50 SCCM for a constant Si/C ratio approximately 0.33 while keeping the TMA and NH3 fixed at 50 and 50 SCCM (5% dilution). In another set of experiments the flow rates of TMA and NH3 were varied between 25 and 100 SCCM for a constant TMA/[TMA+NH<sub>3</sub>] ratio while holding the SiH<sub>4</sub> and C<sub>3</sub>H<sub>8</sub> constant at 10 and 16 SCCM, respectively. In other growth runs the system pressure was varied between 10 and 76 Torr while maintaining the SiH4 at 10 SCCM, C3H8 at 16 SCCM, TMA at 50 SCCM and NH3 at 50 SCCM (5% dilution) for one series and the SiH4 at 10 SCCM, C3H8 at 16 SCCM, TMA at 100 SCCM and NH3 at 100 SCCM (5% dilution) for a second series. For all growths, the H<sub>2</sub> flow through the center of the reactor was maintained at 2 L and the shroud or by pass flow was between 6 to 10 L. Measurement of the sample thickness was done by angle lapping using a rotating abrasive wheel. Concentration measurements of solid solutions where made using Auger electron spectroscopy. Many of the samples where checked for crystallinity using electron channeling techniques.

# 3.0 Results and discussion

Solid solutions of SiC and AlN were grown with concentrations from 20% AlN to 90% AlN as measured by Auger spectroscopy. Figs. 1a and 1b show channeling patterns from two samples which have measured solid solution percentages of 5% and 56% respectively. It is clear from the channeling picture that both films are single crystal. The electron channeling patterns also show the cubic symmetry obtained on silicon substrates, while hexagonally symmetric patterns were obtained on silicon carbide substrates.

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Figs. 2a and 2b show two Auger profiles of of Al, N, Si, and C in solid solution. In fig. 2a, we can see that the concentrations of the elements in the solid solution are uniform from near the surface to the depth sputtered. In fig. 2b and other samples investigated by Auger we have observed that there can be a large build up of either C or Al at the surface. In these cases the concentration of the solid solution was taken when the Auger profile showed constant values of the elements verses distance.

In fig. 3, we show the dependence of AlN percentage on the ratio of TMA/ [TMA+SiH4] for solid solutions grown at a pressure of 10 Torr. From the initial data, the incorporation of AlN in the solid solution at 10 Torr is low until a minimum gas ratio of 0.32 is reached after that the incorporation of AlN in the growing film proceeds approximately linearly. It should be noted that samples which initially did not fit the trend indicated in fig. 3 showed a surface layer which was either rich in carbon or aluminum. After sputtering the aforementioned surface layer, the value of concentration obtained for the solid solutions agreed with the general trend. It is known that the growth of AlN similar to other III/V is controlled by the group III gas flow, while the growth of SiC is controlled by the silane flow. It is therefore not surprising that there is almost a linear relationship between AlN percentage in the solid and ratio of TMA/[TMA+SiH4] in the gas phase.

In fig. 4 data on the dependence of the AlN percentage on pressure for two different TMA/[TMA+SiH4] ratios is presented. In this graph it is observed that the percentage of AlN decreases nonlinearly with increasing pressure. This result can be explained by taking in to account the data for the growth of AlN as a function of pressure. It was found that the AlN growth rate was a strong

function of pressure increasing sharply at pressures lower than 40 Torr [11]. On the other hand, data taken on SiC exhibited the opposite dependence on pressure showing a decrease in growth rate for pressures less than 76 Torr. Since the distribution coefficient of AlN in the solid is related to the ratio of the growth fluxes of the two binaries, it is reasonable to expect a higher percentage of AlN in the solid solution at lower pressures.

### 4.0 Conclusion

We report the growth of solid solutions of  $(AIN)_X(SiC)_{1-X}$  by MOCVD. We believe that this is the first reported growth of  $(AIN)_X(SiC)_{1-X}$  by this technique. Solid solutions were grown over the entire composition range ( as measured by Auger). It appears that the concentration of AlN in the solid at 10 Torr is linearly related to the ratio of TMA/[TMA+SiH4] in the gas flow. Initial data on the dependence of growth rate on gas composition and system pressure is presented. The pressure dependence of the solid solution growth rate is similar to the pressure dependence of growth rate of AlN.

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## References

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1. I.B. Cutler, P.D. Viller, W. Rafaniello, H.K. Park, D.P. Thompson, K.H. Jack, Nature 275 (1978) 434

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- 2. W. Rafaniello, K. Cho, A.V. Virkar, J. of Mat. Sci. 16 (1981) 3479
- 3. R. Ruh and A. Zangvil, J. Am. Ceram. Soc. 65 (1982) 260
- 4. W. Rafaniello, M. R. Plichta, A.V. Virkar, J. Am. Ceram. Soc. 66 (1983) 272
- 5. A. Zangvil and R. Ruh, J. Am. Ceram. Soc. 71 (1988) 884
- 6. R. F. Rutz, US Patent, No. 3482837, May 10, (1983)
- 7. S.A. Nurmagomedov, A.N. Pikhtin, V.N. Rasbegaev, C.K. Safaraliev, Yu. M. Tairov and V.F. Tzvetkov, Soviet Tech Phys. Lett. 12 (1986) 431
- S. A. Nurmagomedov, A.N. Pikhtin, V.N. Rasbegaev, C.K. Safaraliev, Yu.
  M. Tairov and V.F. Tzvetkov, Fiz. Tekh. Poluprovdn. 23 (1989) 162 [Soviet Phys. Semicoond. 23 (1989) 100]
- V.A. Dmitriev, L.B. Elfimov, I. Yu. Lin'kov, Ya V. Morozenko, I.P. Nikitina, V.E. Cheinokov, A.E. Cherenkov, and M.A. Chernov, Sov. Tech. Phys. Lett. 17 (1991) 214
- 10. K.G. Irvine, I. Jenkins, W. Givens, M.G. Spencer, and M. Aluko, Mat. Sci.and Eng. B 11 (1992) 93
- 11. V. Dmitriev, K. G. Irvine, and M.G. Spencer, Presented at 1st Workshop on Wide Bandgap Materials, St. Louis, April 1992

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### **Figure Captions**

- Fig. 1. Electron channeling patterns for  $(AlN)_X(SiC)_{1-X}$  solid solution. Concentration of AlN in solid solution (x as measured by Auger) is (a) 5% and (b) 56%
- Fig. 2. Auger profiles of  $(AlN)_X(SiC)_{1-X}$  solid solution. Concentration of AlN in the solid as measured by Auger (x) is (a) 58.4% and (b) 86%
- Fig. 3. Variation of the concentration of AlN in the solid solution (x as measured by Auger) as a function of the ratio of the moles of TMA/[TMA+SiH4] injected into the reactor. The  $(AIN)_{X}(SiC)_{1-X}$  solid solutions are grown at 10 Torr with an H<sub>2</sub> flow of 2 L through the center of the reactor.
- Fig. 4. Variation of the concentration of AlN in the solid solution (x as measured by Auger) with pressure for two values of TMA/[TMA+SiH4] mole fraction ratios.

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AUGER DEPTH PROFILE



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