Comparative Growth of AlN on Singular and Off-Axis 6H and 4H-SiC by MOCVD

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Abstract

A comparison study of the growth of aluminum nitride (AlN) single crystal epitaxy on 6H-SiC and 4H-SiC substrates has been performed. The material has been characterized using atomic force microscopy (AFM) and reflective high energy electron diffraction (RHEED). AlN crystals were deposited on the following 6H-SiC substrates: singular with and without an initial SiC epilayer, and 3.5^o off-axis with and without an initial SiC epilayer. AlN crystals were deposited on 8.0^o off-axis 4H-SiC with and without initial SiC epilayers. AFM shows that the deposition of AlN on 6H-SiC and 4H-SiC with an initial SiC epilayer displays high quality quasi-two dimensional growth as atomically flat or step flow epitaxy.

Introduction

AlN is a wide band-gap (Eg = 6.2ev at RT) semiconductor material that is suitable for acoustic and visible to deep ultraviolet opto-electronic semiconductor devices [1][2]. One of the biggest drawbacks for growing nitride materials is the availability of suitable substrates [3] [2]. At this time there are no commercial nitride substrates, but researchers have learned to grow heteroepitaxially on semiconductors such as silicon carbide (SiC) and sapphire (Al_2O_3) to produce quality single crystal AlN material [4][5][3].

In this paper, the surface morphology of single crystal AlN deposited on 6H-SiC and 4H-SiC substrates with and without initial SiC epilayers are compared to determine which substrates promote superior growth.

Experimental Procedure

The AlN experiments were performed in a low pressure MOCVD reactor. This reactor possesses a vertical pancake configuration and is heated resistively. The AlN

https://doi.org/10.1557/S1092578300002702 Published online by Cambridge University Press

depositions were performed between 1160°C and 1190°C at 10torr with a total H_2 carrier flow of 5L. The precursors used were trimethylaluminum (TMA) and 5% ammonia (NH_4) balanced in hydrogen (H_2).

The substrate materials for the experiment were 6H and 4H single crystal SiC. All of the substrates were supplied by Cree Research with bulk dopings on the order of $10^{18}cm^{-3}$. The epilayers were grown on the SiC substrates by vapor phase or liqiud phase epitaxy. All the epilayers were approximately 4μm with dopings ranging from the high $10^{16}cm^{-3}$ to low $10^{17}cm^{-3}$. In total, there were 7 different silicon-faced substrates. The substrates were as follows:

Table 1. SiC Substrate Material

Poly Type	**Orientation**	**Epilayer***
6H	singular	n-type
6H	3.5° off-axis	p-type
6H	3.5° off-axis	p+-type
6H	singular	none
6H	3.5° off-axis	none
4H	8° off-axis	p-type
4H	8° off-axis	none

* On-axis by LPE and off-axis by VPE

Several surface cleaning experiments were performed to determine the best method for sample preparation before growth. Three different cleaning procedures were performed for both 6H and 4H-SiC substrates without epilayers. After being cleaned, the growth surfaces were compared using RHEED.

For the first cleaning procedure, two substrates, one 6H and one 4H, were prepared by a thorough cleaning using standard solvents, trichloroethylene (TCE), acetone, and methanol. The two samples were then placed in the reactor, ramped up to 1200°C in H_2, and allowed to sit for 10 minutes. The second cleaning procedure was exactly the same as the first except the two samples were ramped up to 1200°C in vaccum. As for the last procedure, two substrates , one 6H and one 4H, were prepared

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by a thorough cleaning using standard solvents, trichloroethylene (TCE), acetone, and methanol. The samples were then cleaned in a standard RCA sequence as follows: HNO_3 heated to 70°C for 15 minutes; followed by a 1:1:5 mixture of $NH_4OH:H_20_2:H_2O$ heated to 70°C for 15 minutes; followed by a 1:1:5 mixture of $HCl:H_20_2:H_2O$ heated to 70°C for 15 minutes; 50:1 H_20:HF for 1 minute. After the acid etches, the substrates were rinsed in deionized water and blown dry with filtered nitrogen

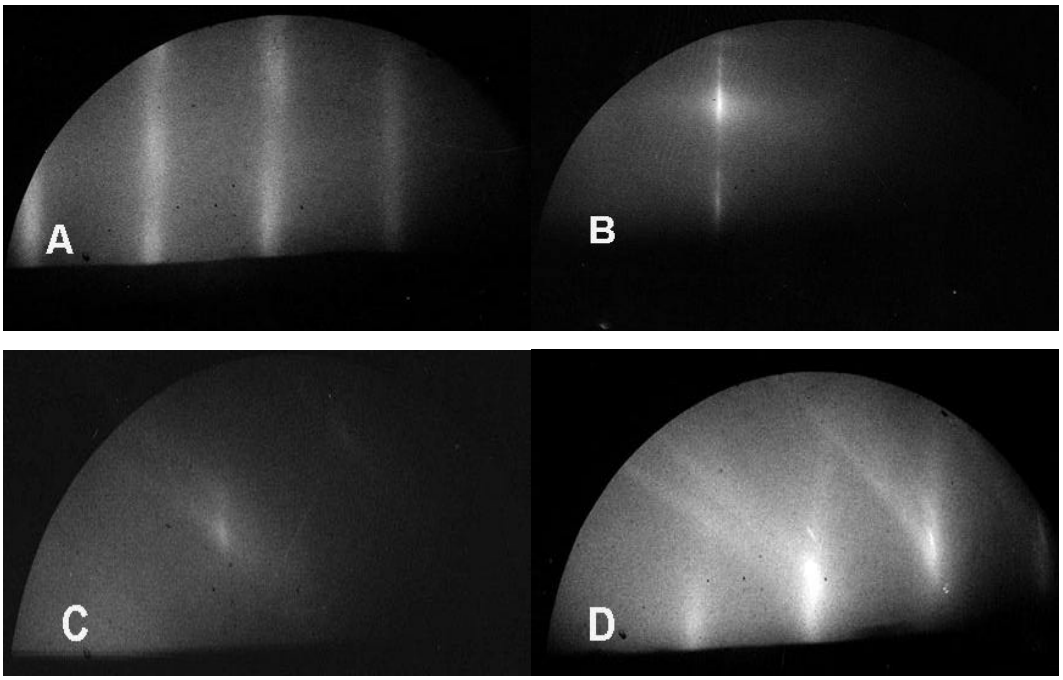

Figure 1. RHEED patterns of (A) 6H-SiC annealed at 1200°C in H_2 for 10 min (B) 6H-SiC prepared with RCA (C) 4H-SiC annealed at 1200°C in H_2 for 10 min. and (D) 4H-SiC prepared with RCA. RHEED for samples annealed in vacuum resemble the RHEED for the sample annealed in H_2.

The cleaning procedure which included the RCA clean produced the "best" RHEED (Figs. 1B and 1D). Therefore, all of the 6H-SiC and 4H-SiC substrates were prepared by a thorough cleaning using standard solvents and then cleaned using the RCA sequence. We noted that whatever the cleaning procedure the oxidation removal from the 6H-SiC was more complete than on 4H-SiC.

Directly after cleaning, AlN was deposited using various growth parameters optimized for the type of substrate in relation to crystallinity. Following each deposition,

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RHEED was performed to determine the degree of crystallinity (Figure 2). If a sample displayed poor crystallinity, the growth was re-optimized in relation to the temperature or V/III ratio and repeated. After growth, the surfaces of the AlN epitaxy were analyzed using AFM to determine quality and type of growth .

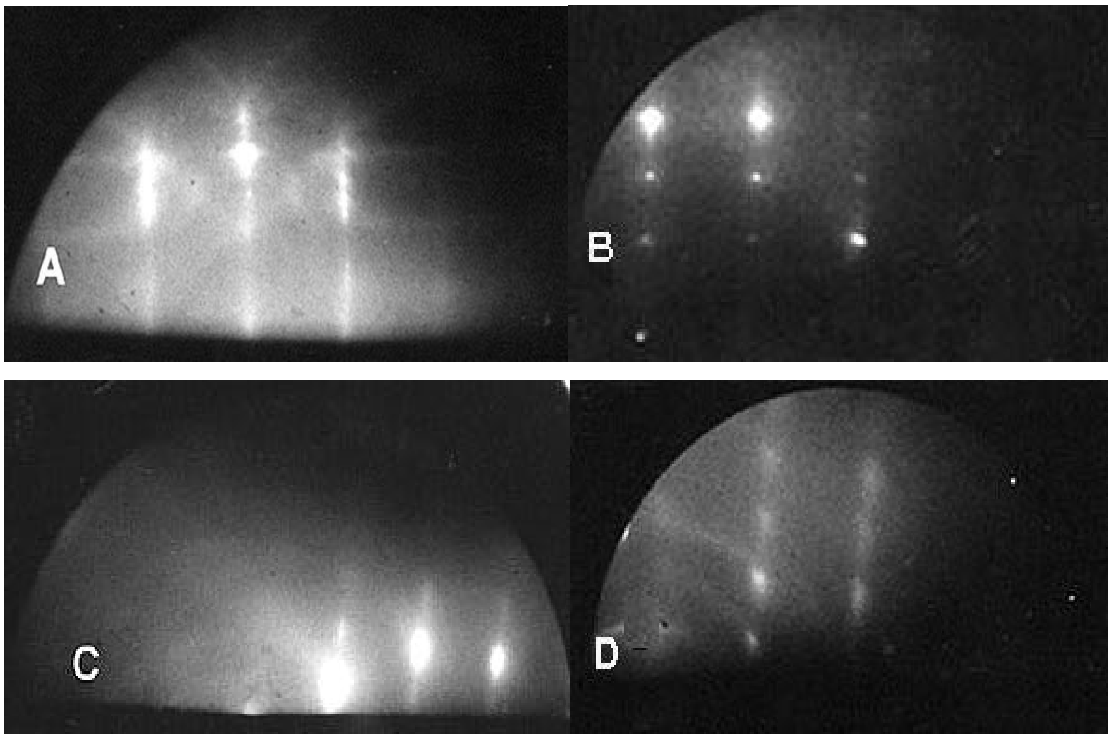

Figure 2. Typical RHEED patterns of (A) AlN/6H-SiC smooth surface (B) AlN/6H-SiC rough surface (C) AlN/4H-SiC smooth surface and (D) AlN/4H-SiC rough surface.

Results and Discussion

The morphology of AlN deposited on 6H-SiC with a intermediate SiC epitaxial is shown in Figures 3-5. In all cases a quasi 2-D step flow growth is observed. We note that these are not atomic steps, but are at least 40 angstroms in height and probably represent step bunching. The average measurement of roughness of the surfaces was 17 to 25 nm. When AlN is deposited on 6H-SiC without epitaxial growth, we have seldom observed step flow like growth. Figure 6 and 7 show morphology that is typical of growth directly on 6H-SiC substrates. This growth is randomly nucleated. This type of growth is characteristic of nitride material on a substrate without a buffer layer [6][7].

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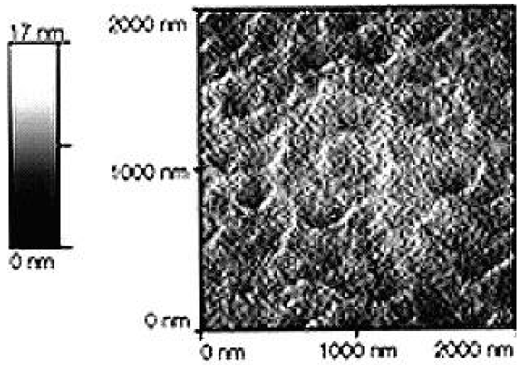

Fig. 3. AlN/n-type epi./6H-SiC singular

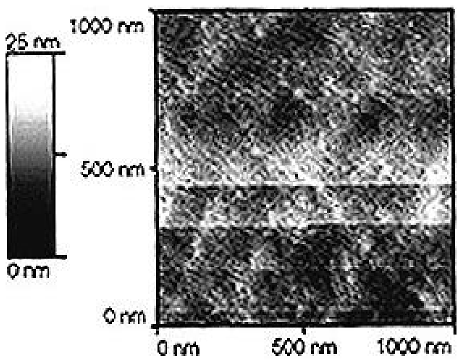

Fig. 4. AlN/p-type epi/6H-SiC off-Axis

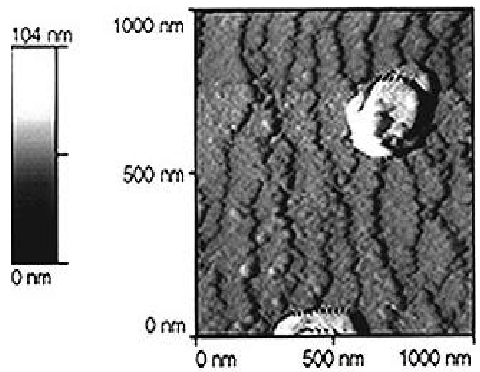

Fig. 5. AlN/p+-type epi/6H-SiC off-Axis

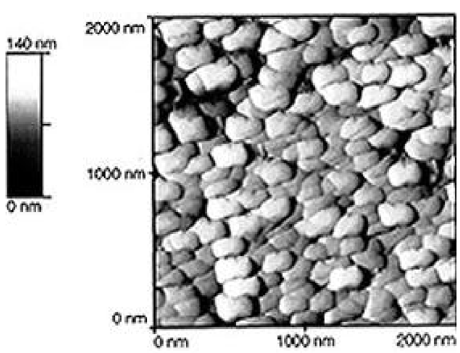

Fig. 6. AlN/6H-SiC Singular

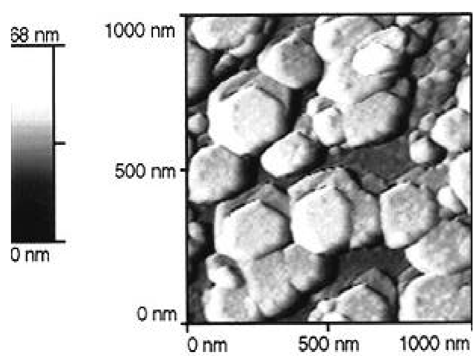

Fig. 7. AlN/6H-SiC off-Axis

When growth of AlN was performed on 4H-SiC substrates the quality of the material was not as reproducible as that on 6H-SiC. Despite the overall quality of the

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growths on SiC, we were able to obtain step flow growth when the starting contained an initial epitaxial layer. Like the 6H-SiC , the 4H-SiC with initial SiC epilayers (Figure 8) displayed either a smooth surface or a step flow growth. In relation to figure 8, the AlN on 4H-SiC sample with an initial p-type epilayer displays high quality step flow growth with a surface step to step height of approximately 14nm.

Again like the 6H-SiC, the 4H-SiC samples (Figs. 9 and 10) without an initial epilayer displayed 3-D random nucleated growth. The islands formed in relation to the nucleation are small, approximately 30nm above the coalesced surface, and independent.

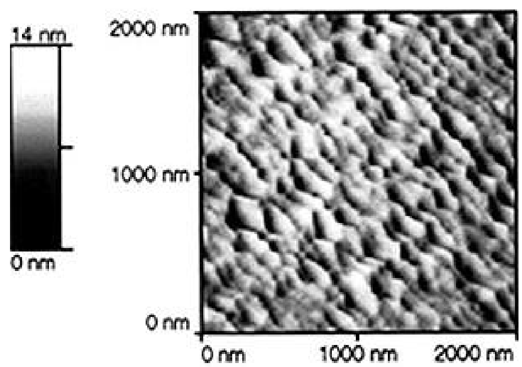

Figure 8. AlN/p-type epi/4H-SiC off-axis

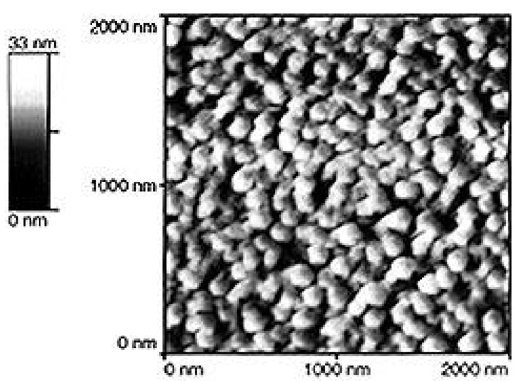

Figure 9. AlN/4H-SiC off-axis

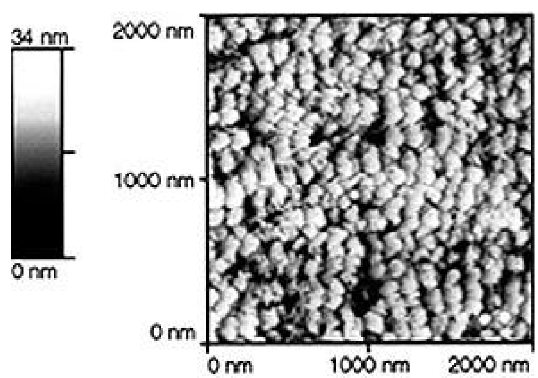

Figure 10. AlN/4H-SiC off axis

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Conclusion

AlN was deposited on off-axis and singular 6H and 4H-SiC substrates with and without initial SiC epilayers. All of the AlN deposited on the SiC with initial epitaxial layers displayed relatively flat surface or step flow growth. The AlN deposited on the singular 6H-SiC with the n-type initial SiC epilayer and on the off-axis 6H-SiC with the p-type initial SiC epilayer achieved atomically smooth morphology. The other samples, AlN deposited on off-axis 6H-SiC with an initial n-type SiC epilayer and AlN deposited on singular 4H-SiC with an initial p-type SiC epilayer, achieved high quality 2-D step flow growth.

Acknowledgements

The authors acknowledge the support of the Office of Naval Research, contract monitor Dr. Colin Wood.

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