## Filtering Study of Threading Dislocations in AlN Buffered MBE GaN/Sapphire Using Single and Multiple High Temperature AlN Intermediate Layers

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Conventional and high resolution transmission electron microscopy have been used to characterise GaN/AlN systems grown on sapphire (0001) by plasma assisted molecular beam epitaxy. We analyse the filtering of threading dislocations using single and multiple thin high temperature AlN interlayers between high temperature GaN layers. A reduction of threading dislocation density is obtained comparing the measured values in samples with one and with three AlN interlayers. The dislocation likelihood increases when the GaN layer between AlN interlayers is thicker.

**Introduction** Since the realization of GaN based light emitting diodes and laser diodes, the group III-nitride semiconductors have gained increasing attention due to their potential applications. The relevant properties of these materials, such as high breakdown voltage, good thermal conductivity and a band gap energy ranging from 1.8 to 6.2 eV, make the research activity on these semiconductors very promising [1, 2]. Due to the lack of a matching substrate, the III–N layers have been grown by hetero-epitaxy on various substrates. The growth of GaN on the (0001) basal plane of sapphire is the one most extensively characterized [3, 4]. Growth of GaN films on this substrate by molecular beam epitaxy (MBE) produces layers with high structural quality [5].

On the other hand, the mismatch between the lattice parameters of GaN and sapphire leads to the formation of three-dimensional islands [6]. To counteract this effect, a low temperature (LT) buffer approach has been used. Nevertheless, GaN grown using this approach still contains high threading dislocation densities. These defects affect the performance of electronic and opto-electronic devices. In order to reduce the threading defects in GaN epilayers, approaches based on the insertion of LT-GaN or LT-AlN interlayers between high-temperature GaN layers have been reported [7, 8].

In previous articles, we demonstrated the improvement of the crystalline quality by the insertion of a high temperature (HT) AlN interlayer (IL) between HT-GaN layers grown by plasma assisted MBE on sapphire. In this way a clear reduction in the density of threading dislocations was measured [9-11].

In this paper, the influence of the GaN thickness between each couple of AlN-ILs on the dislocation density reduction in interlayer systems is evaluated and explained.

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samples	spacing (nm)	number of AlN-ILs
A1	_	1
A3	100	3
B1	-	1
B3	300	3

Table 1

Number of AlN-ILs and spacing between each couple of AlN-ILs in the studied samples

**Experimental Procedure** GaN-AlN multilayers used in this study were grown on (0001)-oriented surface sapphire substrates in a Riber32 UHV chamber by PAMBE. The RF plasma source generated nitrogen from ultra pure  $N_2$  gas. Two series of samples, A and B, have been characterized. Both series were grown using an AlN buffer layer on the sapphire substrate before starting growth of the GaN epitaxial layer. Each series has two samples, with one and with three HT-AlN-ILs, respectively. AlN-ILs were grown with a thickness of 2.5 nm in all the samples. The first thin AlN-IL was deposited for both series on a nominally 100 nm thick GaN layer without any growth interruption at 690 °C. In series A, the GaN layer thickness between AlN-ILs was 100 nm (sample A3), whereas in series B, the thickness between interlayers was 300 nm of GaN (sample B3). Further growth details and AFM, XRD and mobility data on these samples were published elsewhere [12]. Samples are listed in Table 1.

The crystalline quality of the samples was studied by conventional transmission electron microscopy (CTEM) in a JEOL-1200 EX transmission electron microscope working at 120 kV. AlN-IL thickness was measured by high resolution transmission electron microscope (HRTEM) in a JEOL-2000 EX transmission electron microscope operated at 200 kV. Planar view and cross-sectional TEM specimens (PVTEM and XTEM, respectively) were thinned down to 100  $\mu$ m by mechanical grinding and dimpled down to 5  $\mu$ m. The electron transparency was achieved by ion milling at 4.5 kV with a liquid N<sub>2</sub> cold stage to reduce the damage in the samples.

**Results and Discussion** The introduction of a thin HT-AlN interlayer in a GaN layer grown by plasma assisted MBE on sapphire turned out to reduce the threading dislocation density reaching the epilayer surface. This reduction was higher when the HT-AlN thickness was below the critical value because the misfit strain changes the threading dislocation line direction and the interactions between dislocations are more likely to occur. The Fischer model applied to the AlN/GaN multilayer system explains the experimental results previously obtained [13]. The grown AlN-IL layer thickness was smaller than 5 nm to guarantee an AlN-IL thickness below the critical value previously calculated.

Three beam (transmitted, 0002, and 0002) HREM images with the electron beam near the  $\langle 11\overline{2}0 \rangle$  zone axis were produced for both series of samples to measure the AlN-IL thickness in the films. The incident electron beam was always kept in the (0001) plane. Figure 1 shows an image obtained under these HRTEM conditions in sample B3. Using (0002) GaN planes as reference, we estimate an AlN-IL thickness of  $(2.5 \pm 0.5)$  nm. A detailed study about the measurement of thin interlayer's thickness in these samples using Rutherford backscattering (RBS) and Fresnel fringes analysis has been carried out and will be published elsewhere.



Fig. 1. HREM image recorded in three beam conditions (transmitted, 0002 and  $000\overline{2}$ ) near  $\langle 11\overline{2}0 \rangle$  zone axis showing the fringe contrasts corresponding to {0002} planes in AlN/GaN system

The microstructure of the layers, the dislocation morphology, and the density have been studied using different TEM techniques on both plane view and cross section specimens. Figure 2 shows PVTEM images recorded in two beam conditions with  $g = 11\overline{2}0$  for both series of samples. Wide regions of all the samples were analysed to obtain threading dislocation density measurements. The dislocations considered were those with Burgers vectors  $\mathbf{b} = 1/3\langle 11\overline{2}0 \rangle$  and  $\mathbf{b} = 1/3\langle 11\overline{2}3 \rangle$  reaching the surface of the overgrown GaN layer. The dislocations with such Burgers vectors in the system AlN/GaN represent the majority of threading dislocations [10, 11]. The measured values of dislocation densities at the surface ( $g_D$ ) for the different samples are summarized in Table 2.

A reduction of  $\rho_D$  happens, as expected, when introducing three AlN-ILs instead of only one AlN-IL in both series of samples. However the dislocation density reduction factor is not equal for both series. It is 1.4 in sample A3 compared to sample A1. On the other hand, in series B a factor of 5.3 results comparing samples B3 and B1. This means that the dislocation density reduction factor is almost four times larger for samples of series B.

In order to explain these results, the threading dislocation behaviour across both HT-AlN multilayer systems has been studied in detail by XTEM (Fig. 3). The influence of the GaN thickness between each couple of AlN-ILs on the dislocation interactions has been characterized in samples A3 and B3. As can be seen in Fig. 3a some dislocations change their dislocation line directions when reaching the AlN interface. In sample A3 the GaN thickness between AlN-ILs (100 nm) is not large enough to have many threading dislocations interacting. On the other hand, in the interlayer system B3, with thicker GaN layers between a couple of AlN-ILs (300 nm), a larger number of threading dislocation interactions has been observed. The stress due to the mismatch between AlN and GaN changes the threading dislocation line direction. Nevertheless the interactions between dislocations are more favoured in the sample B3 due to the higher thickness of the GaN layer between AlN-ILs.

In fact, a thickness of 300 nm between AlN-ILs in sample B3 was chosen after analysing XTEM images of sample A1. As can be observed in Fig. 3b, the distance from



(a)

100 nm



(c)

(d)

(b)

Fig. 2. Bright field PVTEM images taken in two beam conditions with  $g = 11\overline{2}0$  near the  $\langle 0001 \rangle$  zone axis for samples a) A1, b) A3, c) B1 and d) B3

AlN-ILs for most of the couples of interacting threading dislocations in the GaN interlayers is over 100 and below 300 nm. In fact, interactions are observed even at distances greater than 200 nm. Therefore in sample A3 the spacing between each couple of HT-AlN-ILs is not large enough (only 100 nm) and so prevents the interactions be-

> Table 2 a-component threading dislocation densities measured from PVTEM images and the dislocation density reduction factor in the studied samples

samples	dislocation density $(10^{10} \text{ cm}^{-2})$	dislocation density reduction factor
A1 A3 P1	$5.3 \pm 0.3$ $3.7 \pm 0.4$	A1/A3 = 1.4
B1 B3	$4.3 \pm 0.4$ $0.8 \pm 0.4$	B1/B3 = 5.3





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Fig. 3. Weak Beam XTEM images recorded under two beam conditions for g = 0002 near the  $\langle 11\overline{2}0 \rangle$ zone axis for samples a) A3 and b) B3. The dislocation interaction probability (see arrows in the images) is higher in the sample B3 as can be seen in these images

tween a number of dislocations after bending produced by the stress in the second or third GaN/AlN interface.

Conclusions In this paper we have analysed the filtering of threading dislocations in GaN/sapphire systems using single and multiple thin (2.5 nm) HT-AlN-ILs. The GaN interlayer thickness between each couple of AlN-ILs influences the dislocation density. The dislocation density reduction factor between a sample with one and a sample with three HT-AlN-ILs is almost four times larger in series with 300 nm of GaN interlayer than in the other one with 100 nm GaN interlayer thickness. The dislocation line bending takes place in both series but the dislocation interaction probability increases with the thickness of the GaN interlayer between AlN-ILs.

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