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Growth of AlN by vectored flow epitaxy

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Abstract

The growth of AlN using ammonia and trimethylaluminium is reported using a novel technique, vectored flow epitaxy. The reactor is designed to pre-crack the ammonia, run at atmospheric pressure and keep the precursors spatially separated to avoid the gas-phase interaction that can lead to an involatile adduct. These three innovations have allowed the growth of high-quality AlN at over 2 μm/h with a V/III ratio of only 50:1 at very high group III efficiencies. The precursor separation also leads to a dust-free environment with no appreciable filter load even after the growth of 100 μm of material.

Keywords: A1. X-ray diffraction; A3. Metalorganic chemical vapour deposition; A3. Metalorganic vapour phase epitaxy; B1. Nitrides; B2. Semiconducting III–V materials

1. Introduction

AlN is an important material for UV optoelectronic devices [1]. Their growth, by metalorganic vapour phase epitaxy (MOVPE), has been limited by low growth rate and poor quality material, unless special steps are taken to overcome some fundamental problems while using the standard precursors of ammonia and trimethylaluminium (TMAl). The problems are three-fold, first the ammonia is not catalytically cracked on an AlN nitride surface, unlike GaN [2]; and this results in very poor conversion of ammonia to atomic nitrogen, the species needed on the growing surface. Second, there is a strong adduction between the lone pair on the ammonia molecule and the vacant p-orbital on the TMAl molecule [3]. This results in much of the TMAl being consumed before the growing layer. The adduct [4] can be incorporated into the growing layer making for poor structural quality unless low-growth rates are used. The adducting has been reduced by growing at very low pressures [5], but this in turn leads to the third problem that by being forced to grow at reduced pressure the group III efficiency is compromised over and above the loss of TMAl to the adduct. Other solutions to these problems include growing with alternate supply of precursors in atomic layer epitaxy (ALE) [6] where the precursors are kept temporarily separate and grown at low temporarily to reduce the adduct formation.

These problems result in the following:

In some reactors the V/III ratio needs to be very high, typically $<5000{:}1$ [7] to circumvent the poor ammonia cracking [8]. The efficiency of alkyl usage is low because of a combination of adduct formation and reduced pressure growth, (typically 200 nm/h at 200 Torr for 120 sccm TMAl) [5]. The quality of the layer is compromised because of adduct inclusion in the growing layer so the growth rate needs to be low to achieve good material quality.

We introduce here, the concept of vectored flow epitaxy (VFE). The reactor schematic, Fig. 1, shows the plan view of the various components. The arrangement is similar to reactor designs reported before [9] with the following differences. A radial injector for the group III species, a tangential injector for the group V species and two

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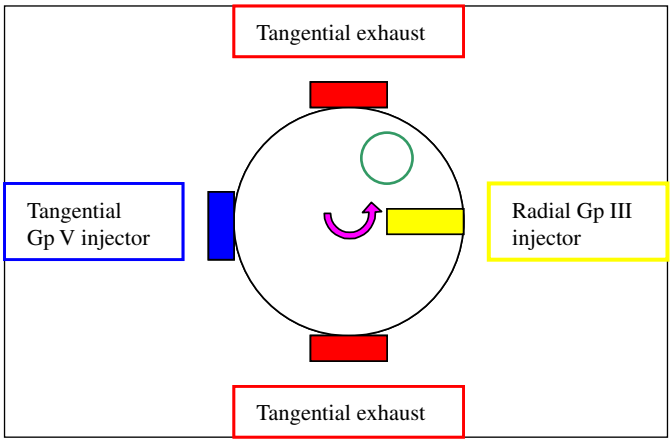

Fig. 1. Reactor schematic, only one of the seven 2″ wafer positions is shown for clarity.

tangential exhausts, orthogonal to the group V injector. The ammonia carrier gas is nitrogen while that for TMAl is predominantly hydrogen with a small amount of nitrogen introduced to control the uniformity of growth across the wafer.

In addition to keeping the reactants separate, the ammonia is pre-cracked just before injection via the tangential injector. The reactor runs at atmospheric pressure to encourage the efficient use of precursors by maximising the reactant partial pressure. VFE deals with the aforementioned problems as follows:

The ammonia is catalytically cracked [patent pending] before entering the growth chamber, the precursors are kept spatially separate whilst flowing continuously and the reactor is run at atmospheric pressure.

2. Experimental procedure

Standard precursors were used, ammonia (Air Liquide 5–9's grade), TMAl 18 °C and TMGa 4 °C both (EMF ltd, DEOX grade). Epi-ready *c*-plane sapphire substrates were heated to 1150 °C for 10 min under nitrogen–hydrogen mixture. Ammonia was then added for 5 min to the nitride surface, and then the platen temperature lowered to 950 °C. This is a relatively low temperature for the growth of AlN but it was chosen to minimise the thermal strain and dopant diffusion. Rotation speeds between 30 and 120 rpm have been investigated.

3. Results

Fig. 2 shows the uniformity of one of the first non-optimised layers, 2 μm thick on a 2″ sapphire substrate grown using 60 μmol/min TMAl and 200 sccm ammonia.

Fig. 3 shows an SEM micrograph of the same layer, the quality was confirmed by powder crystal measurements showing only 2–3 planes.

This sample was investigated by high-resolution X-ray diffractometry (HRXRD). Fig. 4 shows the θ–2θ scan of

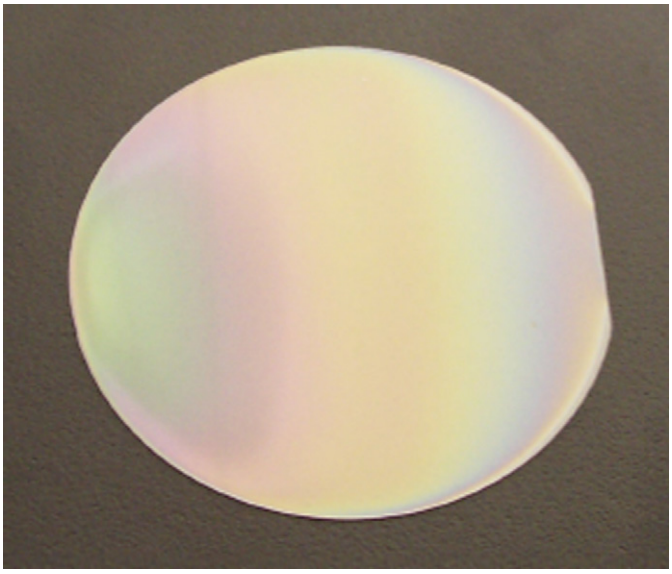

Fig. 2. White light photograph of 1 μm thick AlN layer on sapphire.

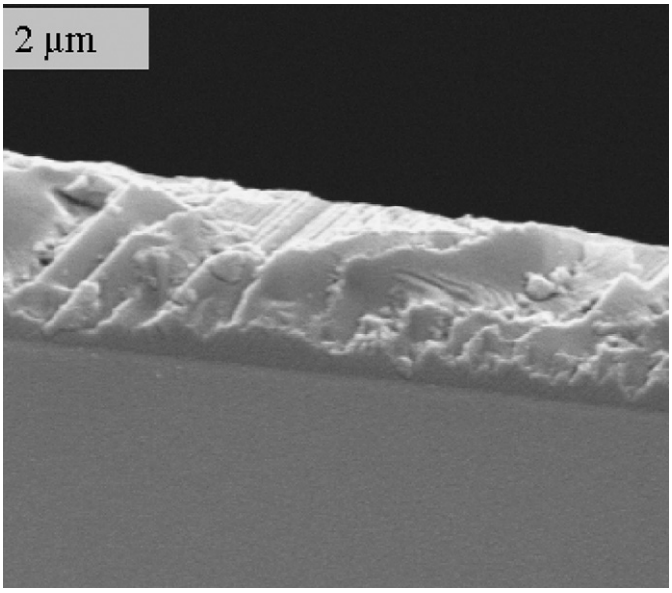

Fig. 3. SEM cross section of AlN layer on sapphire.

the AlN layer, where the predominant crystal orientation is (0 0 2). A small volume (<1%) of material is found and is perhaps (1 0 1) orientation, although usually only (0 0 *n*) is observed in MOVPE grown material.

In order to further check the quality of the AlN layer, a rocking curve was taken through the (0 0 2) peak and this is shown in Fig. 5 the FWHM of 288 arcsec indicates that tilt is not a problem in this material. The twist of the material is best estimated by an asymmetric scan and this was found to be les than 70 arcsec, again suggesting high-quality material.

Such a high-quality layer is encouraging given that no attempt has been made to ameliorate the lattice mismatch between the sapphire and the AlN by using low-temperature buffer layers.