Nitride MOVPE tops the bill

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The predominance of activity in the nitrides field and the emergence of *in situ* monitoring into mainstream metal organic vapour phase epitaxy were among the themes of the 8th European Workshop on MOVPE. The location of Prague close to the geographical centre of the united Europe did much to encourage participation from all corners of the continent, with over 200 delegates from 21 countries attending the workshop, held from 8–11 June 1999.

Workshop falling only two weeks after the US Workshop on OMVPE I was able to see the comparisons and contrasts between the two meetings. Clearly, nitrides played a prominent part in both workshops but I was impressed by the diversity of activity at the European Workshop and some of these new ideas may provide the basis for the next generation of emergent MOVPE technology.

GaN MOVPE

The current hot topic in GaN MOVPE is lateral epitaxial overgrowth (LEO) which was described by Hugues Marchand (University of California, Santa Barbara, USA). The temperature. V/III ratio and mask width for <1-100> stripe alignment on GaN/Al₂O₃ influences the shape of the sidewalls. {10-11} facets produce jagged edges, while {11-20} facets produce straight, vertical edges (Figure 1). There is some speculation about the cause of these different edge morphologies but Marchand et al. proposed that it depended on V/III ratio due to incomplete decomposition of the NH₂.A comparison was made with p-i-n detectors made on the LEO region, coalescence region and onto a GaN/Al₂O₃ substrate. The reverse bias leakage current was eight orders of magnitude lower in the LEO region compared with the conventional substrate.

Indicating the rapid progress that has been made towards production of GaN devices were the presentations from manufacturers of results from multiwafer production reactors. For example, C.A. Tran of EM-CORE Corp (Somerset, NJ, USA) presented results on a 6 x 2" reactor producing high brightness blue LEDs. Impressive results were also presented from layers grown on AIX-TRON and Thomas Swan reactors. Ingrid Moerman (University of Gent, Belgium) showed that ELOG growth resulted in an increase in LED brightness, in contrast with earlier reports.

It was encouraging to see that during this rush to demonstrate blue emission there were some thoughtful contributions on some of the scientific aspects of the growth of this intriguing material system. The Mg doping of GaN was investigated by Bertram Kuhn (University of Stuttgart, Germany) as a function of growth parameters such as temperature and V/III ratio. Alois Krost (University of Magdeburg, Germany), using X-ray reciprocal space mapping, studied the problem of InGaN alloy composition control (Figure 2). This showed that for higher In concentration samples, a partial relaxation of the strain occurred through phase separation, with the tendency to form an $In_{0.5}Ga_{0.5}N$ phase.

VCSELS

Lutz Korte of Infineon Technologies (Munich, Germany) reviewed the topic of vertical cavity surface-emitting lasers (VCSELs). MOVPE has attributes such as fast growth rates and precise control over composition



Figure 1. The LEO process with stripes aligned in the <1-100> directions. Depending on V/III ratio, growth temperature and mask width the sidewalls consist of (a) {10-11} facets, which result in a jagged morphology; (b) {11-20} facets, which are vertical and smooth; (c) {11-22} facets, which usually break up into small {10-11} facets. (Courtesy of H.Marchand et al.)



Figure 2. Reciprocal space map of 100 nm InGaN on 1 µm GaN/a-sapphire substrate around the GaN (0002) Bragg reflection. (Courtesy of A.Krost et al.)

and laver thickness which are attractive for VCSEL growth, but, outside of the 780-1000 nm wavelength range, there is still the need for further technical developments. Problems are encountered with suitable materials for the distributed Bragg reflector (DBR) further into the infrared (IR) and poor quality epitaxy for the shorter wavelength nitride based allovs (Table 1). The specification on the growth parameters is so severe, even for the more developed 850 nm VCSELs, that run to run drift can cause the centre wavelength to change by up to 10 nm. Using in situ control to avoid this drift has reduced the run to run variation to within 2 nm.

The related poster session covered a number of contributions on VCSELs, including a 1.55 µm VCSEL with a strained InGaAsP/InP quantum well by Patrick Abraham (UC Santa Barbara). This session was shared with a number of II-VI contributions, mostly on ZnSe based lay-

ers. A different approach to the ptype doping problem was taken by Holger Kalisch (RWTH Aachen, Germany) who used Sb from trisdimethylaminoantimony. Substrates remain a major issue for these materials and a novel approach was taken bv Matthias Strassburg University, (Technical Berlin. Germany) who grew lattice matched ZnMgCdSe onto InP substrates.

The session on low-dimensional structures, theory and precurcovered some of the sors fundamentals of MOVPE growth and gave us a glimpse of new prospects. An invited talk by Jean Michel Gérard (France Telecom, Bagneux) described some novel prospects for self-assembled quantum boxes (QB). The possible advantages for self-assembled QB devices over conventional quantum wells includes good radiative yield on dislocated substrates and 1.3 µm emission from InAs on GaAs.

The issue of temperature dependence of the optical properties of InP QB arrays was considered in a poster by Jorge Porsche (University of Stuttgart). His results demonstrated a 40-fold improvement in the room temperature photoluminescence intensity by adding Al to the GaInP barrier layer.

Fundamentals

Considering the fundamental aspects of MOVPE, Hilde Hardtdegen (Institut für Schichtund Ionentechnik, Jülich, Germany) examined the modelling of heat transfer and mass transport during AlGaAs growth in a nitrogen or a hydrogen ambient using an AIX 200 reactor. Her poster showed that the different heat transfer in the two gases leads to fully developed flow in hydrogen but more block-like flow in nitrogen, giving better uniformity in the nitrogen ambient. This is encouraging for some industrial applications where a hydrogen ambient may be considered too hazardous.

Precursor purity was considered by Rajesh Odedra (Epichem, Bromborough, UK). A variety of different analytical techniques - ¹H nuclear magnetic resonance spectroscopy (NMR), gas chromatography-atomic emission detection and cryogenic mass spectrometry - were used to examine the moisture content of 1,1 dimethylhydrazine. This precursor is attractive as an alternative to ammonia for growing InGaAsN. Chemical bonding of the water molecules to the precursor makes moisture removal difficult but good progress has been made. While NMR was not sensitive enough to quantify the amount of water present, the study verified the usefulness of the technique as a rapid screening tool.

Devices

MOVPE production of pseudomorphic HEMT material was the subject of an upbeat presentation by Peiter Frijlink (Philips Microwave, Limeil

Wavelength	Active material	Mirror material	Substrate	Performance, quality	Main challenges
Visible blue 300-450 nm	GaInN	GaAlN/AlGaInN, dielectric	Sapphire, SiC	Poor	Epitaxy
Visible green 450-500 nm	GaInN	GaN/AlGaN, dielectric (ZnSSe)	Sapphire, SiC	Poor	Epitaxy
Visible red 630-670 nm	AlGaInP	AlGaGa/AlAs	GaAs	Good, into production	High-temperature performance
Infrared 780-1000 nm	AlGaInAs	AlGaAs/AlAs	GaAs	Excellent, into production	No major problems
Long wave 1300-1550 nm	GaInPAs, GaInNAs, GaSb	GaInPAs/InP, AlAs/GaAs (wafer fused), AlGaSb, dielectric	InP, GaAs	Initial research results	High-temperature performance

Table 1. Materials for different VCSEL emission wavelengths

Brevannes, France). He described production using a planetary reactor, with a capacity of 4000 wafers per year. Performance can now compete with molecular beam epitaxy (MBE) in the 0.9 to 60 GHz range, with excellent uniformity and yield.

A number of posters were on the topic of the application of multiwafer reactors and device uniformity. Harry Protzmann of AIXTRON (Aachen, Germany) reported on 7x2 inch InGaN/GaN MQW wafers with better than 2 nm variation in the photoluminescence emission wavelength between wafers. Frank Dimroth of the Fraunhofer Institute for Solar Energy Systems (Freiburg, Germany) grew tandem solar cells on a 5x4 inch reactor, achieving efficiencies up to 24.2% (AM1.5g) for GaAs on GaAs and 23.3% (AMO) with GaInP on GaAs.

Some of the more novel device applications discussed included MOCVD electrodes grown onto GaAs and InP X- and γ -ray detectors (Frantisek Dubecky, Slovak Academy of Sciences, Bratislava), and growth of InP for micro optoelectro-mechanical systems (Dietmar Keiper, Royal Institute of Technology, Kista, Sweden).

Charles Joyner of Lucent Technologies (Holmdel, NJ, USA) described passive and active waveguide structures in InP. Selective area, low pressure growth was used to grow the waveguide structures, which were designed to change the mode confinement in the propagation direction by selective area growth induced changes in composition.

In situ monitoring

In situ monitoring is emerging more into the mainstream of MOVPE applications, with the benefits ranging from studying growth surfaces to routine monitoring of layer growth rate. An invited talk by Thomas Zettler (Technical University, Berlin) covered both ends of this range, describing the use of optical reflectance techniques to monitor the growth of III-V devices. Reflectance anisotropy spectroscopy (RAS) was used to study the uppermost atomic layers and was combined with interferometry for measurement of layer thickness. Examples of the applications included predicting the performance of AlGaAs laser Bragg-reflectors and p-doping of AlInP.

An elegant system for transfer of MOVPE-grown InP samples into a ultrahigh vacuum (UHV) system for surface examination was the subject of a presentation by Thomas Hannappel (Hahn-Meitner-Institut, Berlin, Germany). This enabled the optical technique of RAS to be combined with UHV surface examination using LEED and Auger analysis together with a scanning tunnelling microscope (STM). The InP layers, grown from tertiarybutylphosphine (TBP), did not show any significant surface degradation or change of bulk properties down to a V/III ratio of 2.5. This is good news for those who are considering switching to this phosphorus precursor!

The area that has seen the most dramatic rise in the use of in situ monitoring has been in the growth of GaN, which nucleates in 3-D islands and subsequently coalesces to form a smooth GaN layer. M. Luenenbuerger of AIXTRON used normal incidence reflectance (interferometry) to monitor the growth of GaN/InGaN quantum wells (QW) grown in a planetary reactor. The transition in growth mode during GaN buffer growth was monitored and thickness determined for the QW structure, which agreed with ex situ characterization.

Another *in situ* monitoring technique is surface photoabsorption (SPA), used by Oleg Khrykin of the Institute for Physics of Microstructures (Nizhny Novgorod, Russia) to examine the strain relaxation mechanism during InGaAs growth on GaAs. A step in the SPA signal was seen to be correlated with a transition from 2-D to 3-D growth, which was associated with heteroepitaxial strain relaxation.

Moving the focus away from the nitrides and arsenides, a number of contributions on the antimonides created some lively discussion. A contentious issue has been the addition of bismuth to InSb to reduce the bandgap for long wave infrared detectors. Magnus Wagener (University of Port Elizabeth, South Africa) showed that Bi tends to form Bi-Ga inclusions at the GaAs/InSbBi layer interface, releasing As to form InAsSb layers. It appears that the solubility of Bi in InSb is extremely low.

Mismatched substrates

Patriarche from France Gilles Telecom presented transmission electron microscopy studies of heterostructures. Cross-sectional images of strained InP/InGaAsP MQW structures show that the InP layers tend to fill the undulations formed in the alloy layer. These undulations were more pronounced, tending towards 3-D growth for layers under tension. An increasingly popular alternative for strained heterostructures is wafer fusion where the laver is grown onto a lattice matched substrate and subsequently bonded to the mismatched substrate by a combination of pressing and annealing. Cross sectional images of an InP/GaAs substrate show networks of dislocations confined to the interface. This opens the possibility for some unlikely layer combinations to satisfy the needs of new devices.

A similar talk by Ingrid Moerman (University of Gent) showed that the twist angle between the compliant substrate and bonded layer (host substrate) is critical for the morphology of layers overgrown on the compliant substrate. The reduction in mismatch using compliant substrates can result in substantial improvements in the quality of the heteroepitaxial material.

The final session of the conference gave us a glimpse of where MOVPE technology might be going with the explosion in the demand for higher bandwidth telecommunications. Rodney Moss of Hewlett-Packard (Ipswich, UK) gave an invited talk on MOVPE for production of opto-electronic devices. The move to wavelength division multiplexing (WDM) is placing new demands on the precision and need for high uniformity in the MOVPE growth, as well as the need for new materials such as GaInAsN for improved 1300/1550 nm lasers. Short product lifetimes mean that the manufacturer needs a toolbox of materials that can satisfy a range of applications. In addition, Moss said that traditional development times will have to be shortened with a new methodology that will include good control of the growth process, increased automation and *in situ* monitoring as a quality control.

The next European Workshop on MOVPE will be held in June 2001 in Wrexham, North Wales, UK.

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