CHAPTER 3

GALLIUM NITRIDE MATERIALS TECHNOLOGY

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INTRODUCTION

The Group-III nitride-based semiconductors have emerged as the leading material for the production of blue LEDs, blue laser diodes, and high power, high temperature electronics. The achievement of high brightness (HB) blue InGaN LEDs has basically led to a revolution in LED technology and opened up enormous new markets that were not accesible before. The historical evolution of GaN materials and device technology in Japan to the USA and Europe in the early 1990's is regarded as one the key developments in solid-state devices today. In Europe gallium nitride materials and devices have gained increasing attention from both academic institutions and industry. The industrial effort is being led by the Siemens/Osram joint venture (Osram Opto Semiconductors), which is in full scale production of blue and white LEDs. In addition, Philips lighting has a joint venture with Agilent technologies (LumiLedsLighting LLC) to develop GaN-based solid-state lighting. The automotive industry in Europe is leading the world in implementing GaN LED backlights in dashboards with several models of Volkswagen automobiles already in production. Europe also possesses leading efforts in both bulk crystal growth and the leading producer of epitaxial systems (Aixtron). The purpose of this chapter is to review the status of GaN materials technology with an emphasis on epitaxial growth.

MATERIALS TECHNOLOGY FOR GAN

Substrates

Several problems in the epitaxial growth of nitrides originate from the non availability of single crystalline GaN substrates or other high quality single crystalline substrates with the same lattice parameters as GaN. For this reason, so far, most of the epitaxial growth of nitrides has been performed on sapphire or SiC substrates. In both cases, problems due to the lattice mismatch between the nitride epi-layer and the substrate (16% for sapphire and 3.5 % for SiC) have to be overcome. One of the major breakthroughs in the growth of device quality group-III nitride material was the implementation of nucleation layers. Using sapphire substrates, thin AlN or GaN nucleation layers deposited at temperatures between 500 and 750 °C showed to remarkably improve the quality of the GaN film grown at temperatures above 1000 °C (Akasaki, et al. 1989; Nakamura 1991). In the case of SiC substrates, the growth is usually initiated with the deposition of a thin AlN nucleation layer at high temperatures (Weeks, et al. 1995).

By this means, GaN material of comparable quality on both types of substrates could be achieved. Since so far, most GaN growth has been performed on c-plane sapphire substrates, in the following section just the growth on c-plane sapphire will be discussed.

Additional substrate materials are currently being examined to determine if the properties of the GaN thin films can be enhanced by improved structural matching. From Figure 3.1 we can see that in addition to sapphire several other substrates offer potentially much better latticed and thermal matching. To this end, 6H-SiC, ZnO, and 3C-SiC, MgO are alternative substrate materials. ZnO has a wurtzite structure with lattice constants of (a=3.32A, c=5.213A) and thus offers an better structural match to the equilibrium wurtzite nitride. 3C-SiC and MgO are both cubic zinc-blende structures having better structural and thermal match to the nitrides than sapphire. 3C-SiC and MgO have cubic lattice constants of a=4.36A and a=4.22A, respectively. Although the nitrides are most commonly observed as the wurtzite (2H) polytype, they can also crystallizes in a metastable zinc-blende structure (a=4.52A) when using non-equilibrium based growth techniques. The identification of a suitable substrate material that is lattice matched and thermally compatible with GaN wurtzite structure (a=3.19A, c=5.185A) will alleviate many of the difficulties associated with the deposition of device quality material.

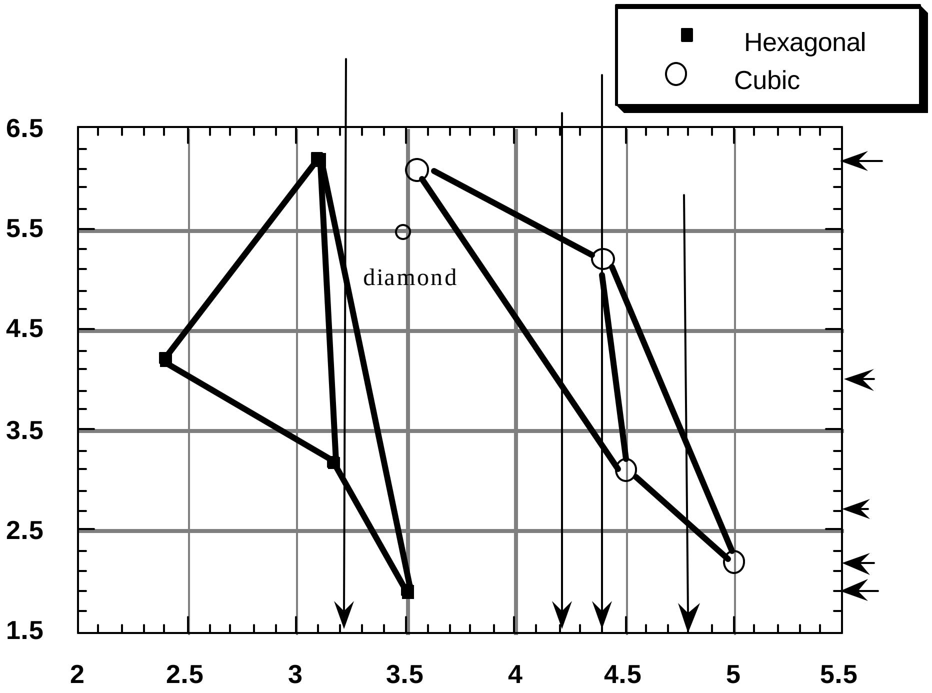

Fig. 3.1. Bandgap and Wavelength of III-V Nitrides versus Lattice Constant.

MATERIALS GROWTH

Metalorganic Chemical Vapor Deposition (MOCVD)

In the past few years, metalorganic chemical vapor deposition (MOCVD) has evolved into a leading technique for production of III-V nitride semiconductor optoelectronic devices and electronic devices. For commercial GaN device applications MOCVD has emerged as the leading candidate because of the achievement of super-bright blue LEDs (Nakamura, et al. 1994) and the large scale-manufacturing potential of the MOCVD technique. The majority of all GaN based p-n junction light emitting diodes (LEDs) typically employ impurity related transition for blue and green emission (Pankove 1972; Nakamura, et al. 1994; Akasaki, et al. 1993; Kahn, et al. 1995). Recently, direct bandgap emission in the blue-green spectral region has been obtained using high In content in single quantum well (SQW) LEDs and lasers using the two-flow MOCVD technique (Nakamura, et al. 1996). Full-color LED displays can now be made entirely with the MOCVD technique when combining the blue and green GaN LEDs with the very high brightness yellow and orange emitting LEDs which were demonstrated in the AlGaInP materials system in the early 1990's (Kou, et al. 1990; Sugawara, et al. 1991). Understanding the growth of AlInGaN/GaN based materials by metalorganic chemical vapor deposition (MOCVD) is therefore of extreme importance in improving the properties of these optoelectronic and high temperature electronic devices.

MOCVD Reaction Chemistry

The basic MOCVD reaction describing the GaN deposition process can be described by the following reaction:

$$Ga(CH3)3(v) + NH3(v) \text{-->} GaN(s) + 3CH4(v)$$

where (v)=vapor and (s)=solid

This balanced expression ignores that the specific reaction path and reactive species are largely unknown. The details of the reaction are not well known and the intermediate reactions are thought to be complex. A more likely reaction pathway leading to growth of the GaAs epitaxial layers involves the homogeneous decomposition of TMGa as reported in an earlier study on GaAs epitaxy (Nishizawa, et al. 1985; DenBaars, et al. 1986).

$$Ga(CH3)3(v) \text{--->} Ga(CH3)2(v) + CH3(v)$$

$$Ga(CH3)2(v) \text{--->} GaCH3(v) + CH3(v)$$

$$Ga(CH3)(v) \text{--->} Ga(v) + CH3(v)$$

The Group-V hydride source, is thought to decompose heterogeneously on the GaN surface or reactor walls to yield atomic nitrogen or a nitrogen containing radical at high growth temperatures. Abstraction of the first hydrogen bond is thought to be the rate limiting step in the decomposition of ammonia.

$$NH3(s/v) \text{--->} NH(3-x)(s/v) + x H(s/v)$$

Therefore one possible growth mechanism of GaN that might occur at the solid vapor interface could be expressed as follows:

$$GaCH3(s/v) + NH(s/v) \text{--->} GaN(s) + 1/2H2$$

However, the level of understanding of the growth process is inadequate at best. The most difficult topic, and certainly the least developed, is the area of the kinetics of the process and growth mechanisms occurring at the solid/vapor interface during MOCVD growth. Pyrolysis and diffusion of the group-III source through the boundary layer is the main pathway controlling the growth rate. However, parasitic side reactions such as solid adduct formation between TMAl and ammonia will decrease the growth rate by limiting the flux of group-III sources to the growing interface.

Optimization of MOCVD growth is typically done by empirical studies of external parameters such as growth temperature, V/III ratio, substrate tilt and mass flow rates. These studies have identified three regions of growth: mass transport limited, desorption and surface kinetically limited regimes. Conventional GaN MOCVD is usually performed in the mass transport limited regime that occurs over a wide temperature range (600°C–1100°C). In this temperature region growth is limited by mass transport of the column III reactant to the growing interface. Because the diffusion process is slightly temperature dependent, there exists a slight increase in the growth rate in this temperature range.

MOCVD System and Reactor Design Issues

Both atmospheric-pressure and low-pressure MOCVD reactors are employed by various research and industrial groups in the growth of GaN. Atmospheric pressure reactors are favored because a high partial pressures of ammonia or nitrogen containing precursor is achievable. MOCVD Reactor design for GaN growth must overcome problems presented by high growth temperatures, prereactions, flow and film non-uniformity. Typically in GaN growth very high temperatures are required because of the high bond-strength of the N-H bond in ammonia precursors. Compounding this fact is the thermodynamic tendency of ammonia to prereact with the group-III metalorganic compounds to form non-volatile adducts. These factors contribute to the difficulties currently facing researchers in the design and scale-up of III-V nitride deposition systems. Further research and development is needed in the scale-up and understanding of the mechanism of gallium nitride growth by MOCVD.

MOCVD Systems for Production

Currently, several types of MOCVD reactor geometries are being developed for the mass production of GaN based materials and devices. Both atmospheric pressure and low-pressure systems are being produced by the major MOCVD equipment manufacturer (Aixtron GmbH, Emcore Corp., Nippon Sanso, and Thomas Swan, Ltd. (Now Aixtron Subsidary). The two types of geometries used by Aixtron and Swan are illustrated in figure 3.2. This figure shows a closed space RDR for atmospheric pressure growth, and a two-flow horizontal flow pancake reactor. Both reactor designs are producing high quality GaN materials and it is not the intent of the author to judge one superior to the other. The benefits to each approach will be specific to the ultimate device and materials being grown.

The closed space RDR has the benefit of atmospheric pressure operation because the low free height eliminates free convection. The two-flow horizontal planetary rotationTM reactor from Aixtron can also be operated at near atmospheric pressure and can accommodate large wafer volumes (>7 wafers). The selection of any reactor has to be carefully considered against factors such as: material quality, high throughput, reproducibility, maintenance, and source usage.

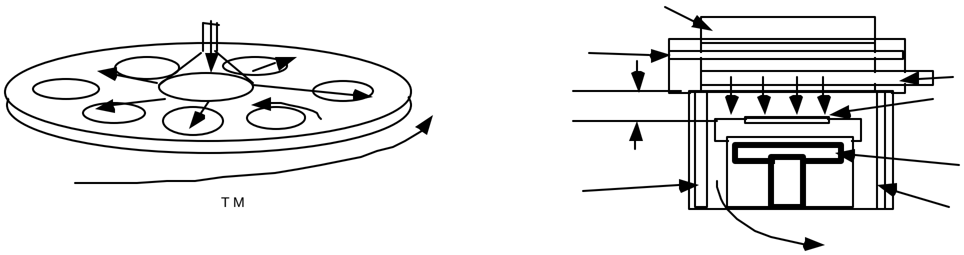

Fig. 3.2. Two types of MOCVD reactors currently produced in Europe, planetary rotating discs (Aixtron) and closed space RDR by Swan (now Aixtron)

GaN p-type and n-type doping

One of the key breakthroughs in the development of GaN technology was the achievement of p-doping using Mg and LEEBI treatment. Akasaki et al. (1989), observed that under low electron beam irradiation (LEEBI) Mg-doped GaN exhibited much lower resistivity and the PL properties drastically improved. This achievement subsequently leads to the development of p/n GaN diodes with good turn on characteristics. Nakamura et al. (1993) built upon this fundamental breakthrough to achieve even higher p-doping and uniform activation of Mg by using high temperature thermal annealing under a nitrogen ambient. The passivation requires post growth treatment for MOCVD material to activate the dopants. During the growth, interstitial hydrogen is incorporated and a H-Mg acceptor complex forms which passivates the acceptor. This H-Mg bond can be broken by a high temperature annealing step under an inert environment. This work demonstrated that hydrogen compensation of Mg in the MOCVD growth of GaN was the principal problem that plagued previous researchers. High room temperature p-doping is further complicated by the high activation energy of magnesium as the most commonly used dopant (270 meV) and the passivation of acceptors with hydrogen during CVD growth. The binding energies of dopants are dependent of the dielectric constant and effective mass of the material. The nitride system has low dielectric constant (GaN, e(0)=9.5) and large effective masses (GaN, me=0.2 mo, mhh=0.75mo) resulting in large binding energies. This is especially pronounced in p-type doping when comparing GaN to GaAs the acceptor levels are very deep because of the large hole mass. This has led to difficulty in high p-type doping. This is the result of two effects: high n-type background concentration compensating the p-dopant and the incomplete activation of the dopants at room temperature. The low p-type doping (typical values are 1017 /cm3) leads to high contact resistance's and problems with current spreading. Further work on increasing the p-doping level and developing new p-dopants will result in substantial payoff in producing LEDs and lasers with lower operating voltages and higher power efficiencies.

Doping n-type is rather straightforward in GaN with silicon being the typical n-dopant. The as-grown material is typical unintentionally n-type, which is widely believed to be due to intrinsic nitrogen vacancies. The Si donor lies just below the conduction band (Ea = 15-25 meV). Therefore well-controlled n-type doping can be easily accomplished using silicon as the donor. The typical MOCVD precursor for n-type doping are silane (SiH4) and disilane (Si2H6) which are typical diluted with hydrogen in the 200 ppm range. Doping levels between $1(10)^{17}$ to $2(10)^{19}$ cm-3 are easily achieved in the doping of GaN with silane.

Growth of AlGaN and AlGaN/GaN Heterostructures

High quality AlGaN films have been demonstrated by atmospheric pressure MOCVD as well as epitaxy performed under low pressure conditions. At growth temperatures below 1100 °C, the mole fraction of aluminum in the AlGaN epitaxial layer was found to be almost directly proportional to the mole fraction of TMAl in the gas phase. At temperatures above 1100 °C, the incorporation efficiency of gallium atoms decreased. This behavior was explained by a decreased sticking probability of Ga molecules at this high temperatures (Hirosawa, et al. 1993).

High quality AlGaN/GaN heterostructures are characterized by a very high mobility of the two dimensional electron gas at the interface (Kahn, et al. 1991). Values as high as 1500 cm2/Vs at room temperature have been achieved in the authors' laboratory (Wu, et al. 1996). The optical properties of AlGaN/GaN quantum wells (Kahn, et al. 1990) were found to be determined by both, quantum and strain related effects (Krishnankutty, et al. 1992; Krishnankutty, et al. 1992b). MOCVD AlN films showed a full width of half maximum of the (002) x-ray rocking curve peak as low as 97 arcseconds (Saxler, et al. 1994). AlN/GaN Superlattices of high structural and optical quality have also been fabricated by switched atomic layer MOCVD (Kahn, et al. 1993).

Growth of InGaN and InGaN/GaN Heterostructures

Growth of high quality InGaN is necessary to obtain good electrical and optical characteristics from LEDs. However, the growth of high quality InGaN has proven to be more difficult than GaN. InGaN growth needs to be performed at much lower temperatures than that of GaN, due to the low dissociation temperature of InN (Matsuoka, et al. 1988; Koukitu, et al. 1996). Furthermore, the decomposition of ammonia becomes less efficient with decreasing temperature due to the high kinetic barrier for breaking the nitrogen - hydrogen bonds. The growth of InGaN has to be performed at temperatures below 850 °C because of the high volatility of indium at common GaN growth temperatures of above 1000 °C. But even on InGaN layers grown at temperatures below 800 °C, In droplet formation was observed (Shimizu, et al. 1994).

Molecular Beam Epitaxy (MBE)

Several researchers in Europe have begun developing MBE for growth of the III-V nitrides. Several approaches have been investigated for supplying an atomic source of nitrogen. RF plasma and electron cyclotron resonance (ECR) microwave plasma sources are the two most successful techniques discovered to date (Lei, et al. 1991; Strite, et al. 1991; Paisley, et al. 1989). In these systems the plasma source is used to crack molecular nitrogen. The plasma sources use a cylindrical cavity geometry to efficiently couple microwave energy into the nitrogen discharge area. The plasma stream is a complex mixture of atomic, molecular, and ionic N radicals. When using ECR sources a tradeoff between growth rate and ion damage has been observed (Lei, et al. 1991) under normal ECR use the flux of low energy reactive N species is so low that only low growth rates of 500Å/hr can be achieved. At higher microwave powers high growth rates can be achieved, but ion damage leading to deep levels and semi-insulating electrical properties is observed. A major advantage of MBE for nitride growth is the low growth temperature that can be achieved due to the atomic nitrogen source. This is in contrast to MOCVD which must employ high growth temperatures (>1000C) to crack the ammonia molecules. The lower growth temperature should result in lower thermal stress upon cooling, less diffusion, and reduced alloy segregation. This is especially important in the AlGaN alloys which posses a large mismatch in the thermal expansion coefficient.

The GaN molecular beam epitaxy (MBE) effort at Siemens was reviewed in detail by Dr. Henning Reichert. Currently the MBE effort is shifting its focus away from optoelectronics and is emphasizing the high microwave power electronic devices. However, very good fundamental study of GaN and InGaN growth by MBE was recently carried out by Dr. Reichert. P-type doping as high as 1E+18cm-3 was achieved by MBE. This is important for low contact resistance to LED's and lasers. A high growth rate of 1.4microns/hr was achieved and mobilities as high as 460cm2/Vsec by MBE growth of GaN on MOCVD GaN templates. An impressive study of InGaN growth yielded good PL as long as 540nm. Recently single crystal InN was achieved at a growth temperature of 400C. Dr. Reichert observes at thermal decomposition temperature of 530C in MBE which is close to the value obtained by Ombacher et al in 1999. P-type GaN studies indicate activation energy of 150meV.

GaN Bulk Crystal Growth

In Poland, world-leading bulk GaN crystal growth is being achieved at Unipress. The Unipress research group is located at the High Pressure Research Center (HPRC) in Warsaw, where they employ GaN bulk crystal growth from a melt. As shown in figure 3.3 pressure as high as 10Kbar are required to growth GaN from a melt. One of the most impressive achievements the panel saw in Europe was the "defect free" bulk GaN wafers grown from melt under extremely high pressure. Dr. Isabel Grzegory gave an overview of the high-pressure solution growth method used for depositing the bulk single crystals.

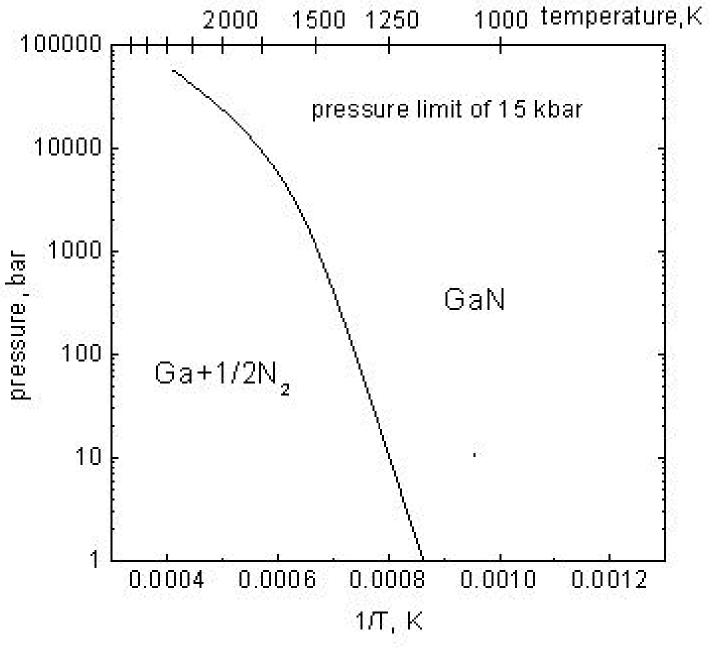

Figure 3.3. Nitrogen pressure vs. growth temperature for bulk gaN substrate growth.

The size of the substrates has steadily increased up to 1cm in 1999 from 5mm in 1997. In comparison to GaN on sapphire technology which exhibits 1e+9cm-2 defect densities, or ELO films which are in the 1E+6cm-2 to 5E+5cm-2 range the bulk GaN crystals possess 10 to 1 defect per square centimeter. Figure 3.4 shows a image of a 1cm diameter crystal grown by the Unipress group. These densities were estimated from etch pit counts and correlated to TEM measurements of higher defect densities found on standard GaN sapphire technology. The team was allowed to observe the bulk crystals under both optical microscopes and field emission SEM. Both n-type bulk crystals and semi-insulating bulk crystal have been grown. The semi-insulating substrates were highly resistive 1E+5ohm-cm and could be used for thin film deposition of high power AlGaN/GaN microwave amplifiers (Litwin-Staszewska, et al. 1999). Very high quality homoepiatxial growth of GaN by MOCVD was obtained on top of these substrates and the films exhibited narrow double-Crystal x-ray diffraction (DCXRD) linewidths as low as 21 arcsec. The group at University of Ulm in collaboration with Unipress has obtained thin films which exhibit the narrowest reported PL linewidths at low temperature (0.1meV) which is indicative of the uniform high quality film (Kornitzer, et al. 1999). MBE has also been performed on the bulk crystals. Both Ga-face and N-face polarity bulk substrates have been produced. The bulk crystals also display extremely smooth cleaved facets with rms roughness of 5 angstroms which would make excellent laser facets.

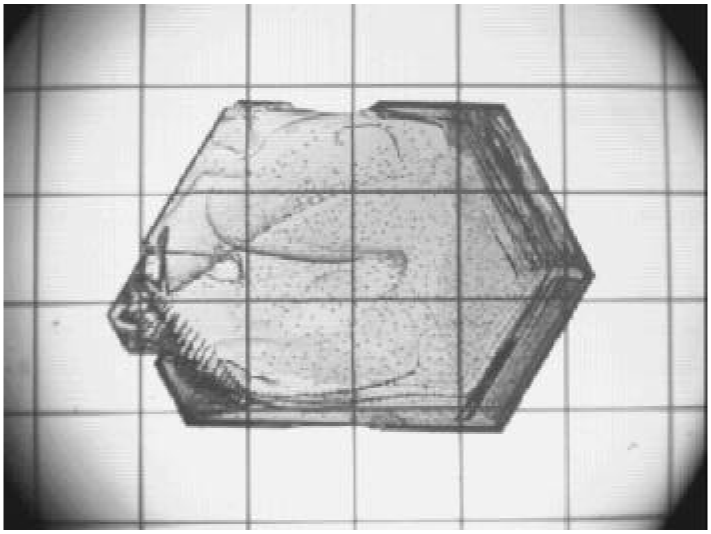

Fig.3.4. 1cm Diameter Bulk GaN Substrate

Lateral Epitaxial Overgrowth

Lateral Epitaxial Overgrowth (LEO) is an attractive method to produce GaN films with a low density of extended defects, which is beneficial both to studies of the fundamental properties of the GaInAlN materials system and to GaN-based device technology. The basic concept is to reduce defect propogation in masked regions of the substrate where the laterally overgrowing GaN is defect free. This is illustrated in the transmission electron micrograph in Figure 3.3. Recent studies in Europe, Japan and USA have confirmed that the density of threading dislocations (TDs) is reduced by 3-4 orders of magnitude in the LEO material grown on 6H-SiC (Zheleva, et al. 1997) and Al2O3 (Sakai, et al. 1997; Marchand, et al. 1998; Marchand, et al. 1998b) substrates, and the mechanisms of threading dislocations evolution during LEO have been investigated. Studies of the optical properties of LEO GaN (Chichibu, et al. 1998; Freitas, et al. 1998) and InGaN quantum wells (Rosner, et al. in press) have revealed that TDs act as non-radiative recombination centers. However, the minority carrier diffusion length (<200 nm) is smaller than the average distance between Tds such that the emission mechanisms of the carriers that do recombine radiatively appear to be unaffected by moderate TD densities (~106-109 cm-2). On the other hand, reducing the TD density has been shown to reduce the reverse leakage current by ~3 orders of magnitude in GaN p-n junctions (Kozodoy, et al. 1998), InGaN single (Mukai, et al. 1998) and multiple (Sasaoka, et al. 1998) quantum well light emitting diodes, and GaN/AlGaN heterojunction field-effect transistors (Vetury, et al., 1998) fabricated on LEO GaN. More recently, ultraviolet p-i-n photodetectors fabricated on LEO AlGaN have exhibited a similar reduction of the reverse leakage current by up to 6 orders of magnitude (Parish, et al. 1998). The use of LEO GaN has also resulted in marked improvements in the lifetime of InGaN/GaN laser diodes (Nakamura, et al. 1998).

OPTOELECTRONIC MATERIALS

LED Materials

A recent development is that Seimens has recently spun off Infineon Technologies, which will be responsible for semiconductor components. Infineon and Osram also have a joint venture (Osram Opto Semiconductors) to develop GaN blue LEDs and solid-state white lighting using GaN based LEDs. Infineon has a large GaN on SiC LED fabrication facility at Regensburg Germany. White LED products have recently been introduced by Osram under the trademarked name of TOPLED. The advances in GaN based LEDs has enabled many new markets to be opened for LEDs. Only now can one use three LEDs to tune to any color in the visible range, or even use a single blue LED in combination with phosphors to make “white LEDs”. This concept is particularly attractive because the solid state nature of semiconductor devices produce very high reliability. The average lifespan of an LED is on the order of tens of years (Craford 1977). In addition, Philips Lighting BV, has also formed a joint venture with Agilent Technologies in the US, to focus on solid state lighting. Lumileds lighting is the name of the Philips JV and is 50% owned by the parent companies.

Fig. 3.5. Automotive dashboard back-lit with blue LEDs.

Full-color and white LEDs are now appearing in numerous applications ranging from in figure 3.5, Volkswagen is using blue LEDs to back-light the dashboard in the 1999 VW Passat. Another potentially large application of GaN is in for fabricating blue laser diodes (LDs) for extremely high-density optical storage systems. Because the storage density of optical compact discs (CDs) and digital video discs (DVDs) is inversely proportional to the square of the laser wavelength, a 4-8 fold increase in capacity could be realized with short wavelength laser diodes. Future research is needed to commercialize GaN for optical storage, energy efficient lighting, communications, printing, projection TV, and even surgery. As GaN manufacturing volumes increase and costs decrease one can expect to see GaN LEDs and LDs in an ever increasing number of applications wherever economical and reliable illumination is needed.

Two types of Blue LEDs are commercially available: Laterally contacted devices with quantum well (QW) InGaN active regions on sapphire substrates from Nichia Chemical Industries, Ltd., Toyoda Gosei, Hewlett-Packard Optoelectronics, and vertically contacted GaN/AlGaN double heterostructure (DH) devices on conducting SiC substrates by Cree Research, Inc. and Siemens. The SQW LEDs are extremely efficient currently. The best external efficiency achieved by Nichia for the blue and green LEDs is 10% and 12% external quantum efficiencies, respectively (Nakamura & Fasol 1997). The white LEDs are approximately 7% and have a luminous efficacy of 15 lumens/watt, which is becoming competitive with existing incandescent sources. In addition, newly developed InGaN amber LEDs are superior to conventional AlGaInP amber LEDs in terms of temperature performance. The wavelength shift as a function of temperature is much smaller in GaN than in GaAs based LEDs.

The second type of LED structure is grown on SiC substrates and is made by Cree Research Inc. These devices feature a MOCVD grown GaN-AlGaN p-n junction grown on 6H-SiC substrates. SiC has the advantage of having a smaller lattice misfit to GaN and the possibility of providing n-doped substrates. Another advantage is the vertical conduction pathway allows for a drop in replacement part similar to the other GaP and GaAs based structures, which have the same vertical geometry. The high thermal conductivity of SiC substrates might also benefit the high temperature high output power applications of GaN LEDs.

Blue Laser Materials

Several groups in Japan, USA, and now Europe have recently reported obtaining laser diodes in the GaN materials system. Osram Opto Semiconductors was the first European group to achieve reasonable pulse blue laser performance in 1999 (HÄRLE 2000). Of all the groups in the world to date, only Nichia has obtained long-life continuous wave (CW) emission, and the laser diode was market released in January 1999. Nichia was the first company in the world to successful achieve a blue laser diode in GaN materials system. Initially the GaN laser lasted only a few seconds but reliability has improved dramatically to the current state at which Dr. Nakamura has achieved CW operation up to 10,000 hours at room temperature, elevated temperature testing at 50°C has projected the actual lifetime is in excess of 20,000 hours at 2mW. Current estimates are the reduction in the spot size which can be achieved with blue GaN lasers will yield a storage capacity of 12GB per 5.25 inch diameter discs, enough for recording a full-length motion picture in high resolution mode.

CONCLUSIONS

The Group-III nitride semiconductors have emerged as the leading material for fabricating high reliability short wavelength emitters, and emerging high power electronic devices. MOCVD and MBE are the leading growth technologies for depositing high quality GaN/InGaN heterostructure based devices. Lateral epitaxial overgrowth (LEO) and bulk crystals are some of the key technologies requiring further study to enable the next generation of GaN based devices.

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