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Quarterly Progress Report

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Visible Light Emitting Materials and Injection Devices

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(I) Molecular Beam Epitaxy Growth of II-VI and III-Nitrides (Robert Park)

(a) Widegap II-VI

We performed real-time, *in-situ* CL intensity measurements this quarter during the MBE growth of $\text{Cd}_x\text{Zn}_{1-x}\text{Se}/\text{ZnSe}$ MQW structures in which we varied the barrier and/or well widths for a fixed $\text{Cd}_x\text{Zn}_{1-x}\text{Se}$ composition ($x=0.2$). In each case we quantified the integrated intensity of the CL emission observed during the growth. For the MQW structures grown thus far, the most intense integrated CL emission was detected from a structure comprised of 200 Å thick ZnSe barriers and 50 Å thick $\text{Cd}_{0.2}\text{Zn}_{0.8}\text{Se}$ quantum wells. The 200 Å ZnSe/50 Å CdZnSe MQW structure also exhibited strong room temperature exciton emission when *ex-situ* photoluminescence (PL) was performed on the sample. The PL intensity was measured by Dr. Simmons' group. Consequently, it appears that real-time, *in-situ* CL monitoring could have interesting applications with regard to the design and production of MQW structures and we plan to continue working in this area.

(b) Column III-Nitride

We have continued to develop our understanding of the growth by rf plasma-MBE of GaN on c-plane sapphire, and have recently grown very high quality films as indicated by 7K photoluminescence measurements. Figs. I.1 and I.2 are 7K PL spectra recorded from a 0.6 μm thick GaN/sapphire film grown recently. Fig. I.1 shows the full spectrum while Fig. I.2 details the excitonic regime. The PL is dominated by free-exciton emission at 3.472 eV while broad deep level emission around 2.2 eV appears to be absent in the spectrum. For the 0.6 μm thick film, the FWHM of the free-exciton peak is around 15 meV. Table I-1 presents a summary of the PL data from the literature for GaN grown by various techniques to various thicknesses. As can be seen from the table, our FWHM value for the free-exciton peak is still on the high side, but our film is only 0.6 μm thick. We expect a reduction in the exciton linewidth when thicker films are grown. The clean-nature of the PL spectrum with a dominant free-exciton emission, however, is very encouraging.

(II-A) Ohmic Contact Formation (Paul Holloway)

(a) ZnSe Contacts

Studies of the degradation of ZnTe/ZnSe (pseudo-graded or multiquantum well) contacts to p-ZnSe have continued. To perform the contact degradation, samples from 3M Company were mounted onto a copper block (heat sink) which is inserted into an ADP cold stage (cooled by helium compressor). Electrical connections were made to the samples through a front side Pt/Au ohmic contact (to the ZnTe/ZnSe structure) and a backside Ti/Au ohmic contact (to the GaAs substrate). These are the contact metallizations currently used at 3M for diode lasers. The samples were held at a constant current density of 10 A/cm², the cold stage temperature was set at 300°K, and the sample voltage and temperature were monitored throughout the experiment.

Preliminary results indicate current densities of 10 A/cm² are obtained for a ~ 0.1 cm² sample with ~ 1 A of current by applying between 3 to 4 volts. A rapid localized failure of the contacts occurred when fine point electrical probes (tip radius < 1 mm) were used to make contact to the front side Pt/Au contact. This failure resulted in a small reaction

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PL spectrum (7 K) of GaN

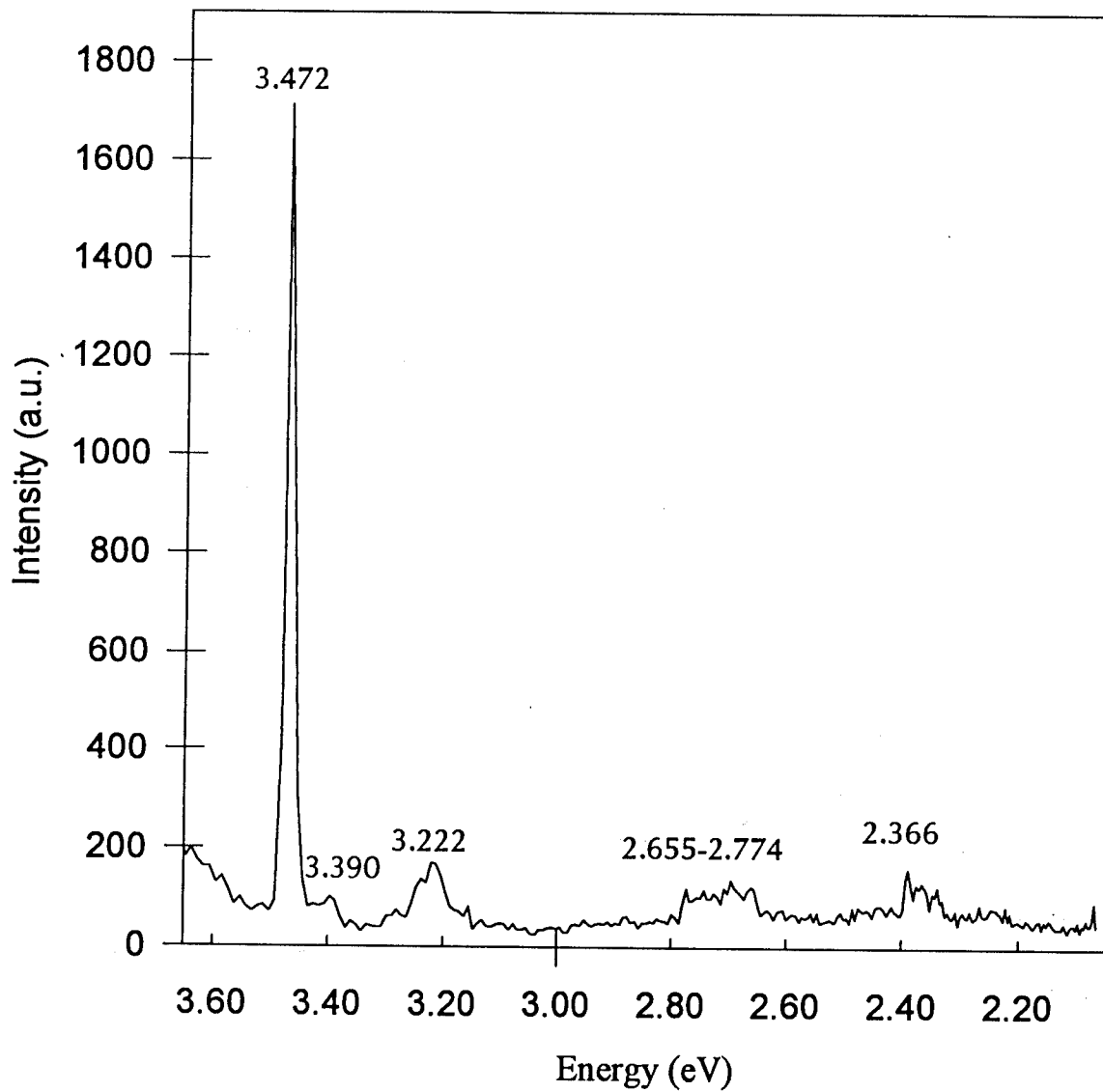


Fig. I.1.

PL spectrum (7 K) of GaN

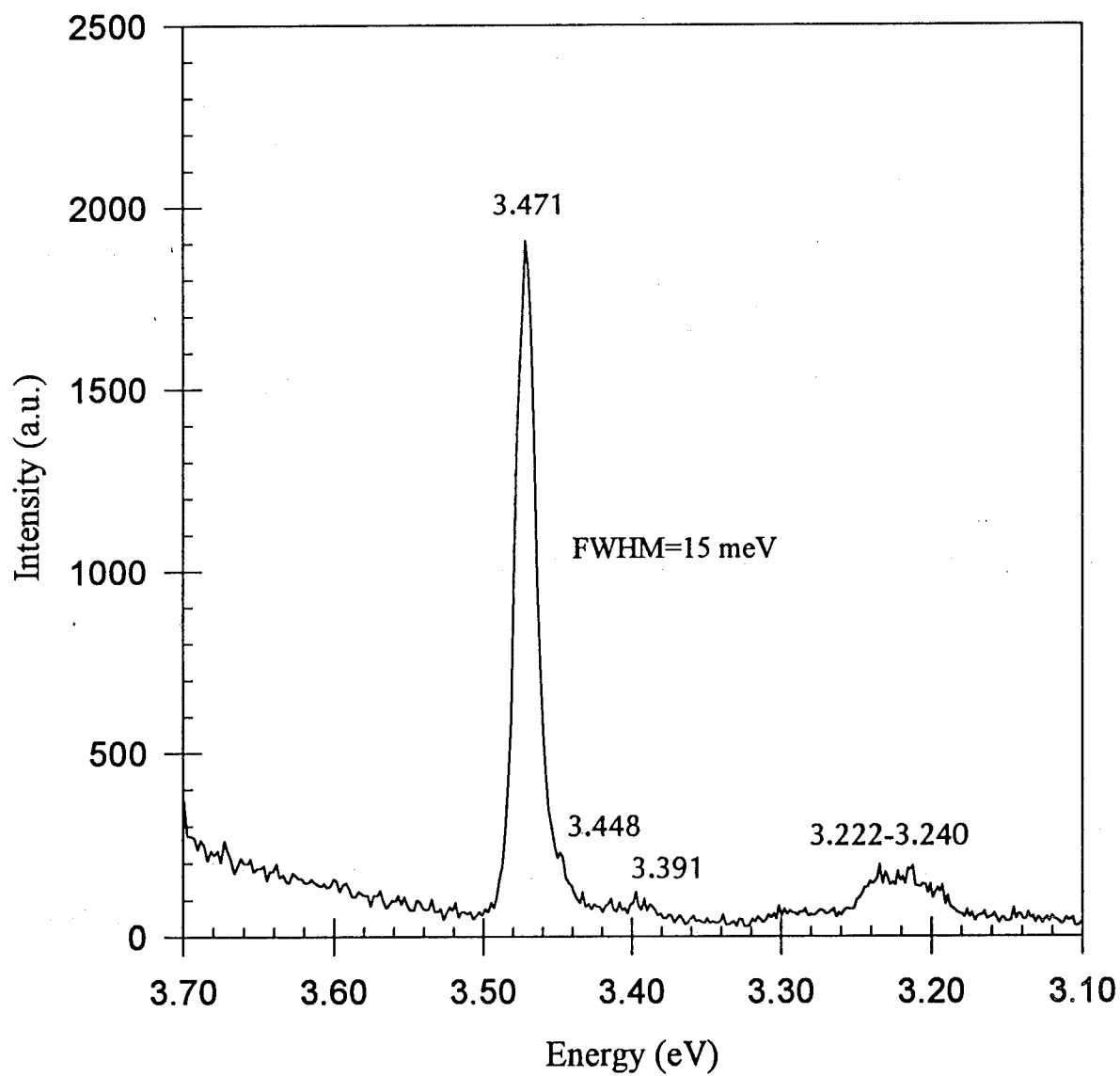


Fig. 1.2.

PL Literature Data

Technique	Substrate	Film Thickness	Exciton Peak Energy (measurement temp.)	FWHM of exciton peak
ECR-MBE (U. of Illinois)	SiC	1 μm	3.474 eV (5K)	5 meV
ECR-MBE (U. of Illinois)	Sapphire	2.5-3.5 μm	3.47 eV (6K)	5 meV
MOCVD (Nagoya Univ.)	Si (111)	2 μm	3.46 eV (4.2K)	17 meV
	Sapphire	2 μm (?)	3.49 eV (4.2K)	3.1 meV
MOCVD (Naval Res. Lab.)	Sapphire	5.6 μm	3.472 eV (6K)	3.2 meV
ECR-MBE (U. of Illinois)	SiC	1 μm	3.47 eV (6K)	8-10 meV
Sublimation Sandwich Technique (TU München)	SiC	60 μm	3.47 eV (2K)	4 meV
ECR-MBE (Boston Univ.)	Sapphire	1-2 μm	3.5(?) eV (77K)	N/A
RF-MBE (U. of Florida)	Sapphire	0.6 μm	3.472 eV (7K)	15 meV

Table I-1.

zone (pit in the Pd/Au layer) surrounding the point of contact. To prevent this failure from occurring, larger electrical leads ($\sim 4\text{mm} \times 4\text{mm}$) were bonded to the front and backside of the samples using silver epoxy. Samples mounted with this configuration have exhibited a slow degradation over more than 6 hours at 10 A/cm^2 . The I-V characteristics have shown a change from ohmic to rectifying behavior. During testing, the contact structures will be analyzed to determine the cause of this change.

In addition, the results (reported in last quarter's report) for Au and Ag contacts evaporated onto p-ZnSe (N doped with carrier concentration of $3 \times 10^{17} \text{ cm}^{-3}$) were extended. The Au and Ag contacts were heat treated at 150 to 400°C in oxygen and forming gas and it was shown that the reverse bias breakdown voltage of the contacts was reduced by heat treatment in oxygen as opposed to forming gas.

(b) ZnTe Contacts

Studies of the formation of ohmic contacts to p-ZnTe have continued with emphasis on Au electrical contacts. Gold films (1100 \AA) were analyzed using current-voltage (I-V) measurements to determine thermal stability upon post deposition annealing. Auger electron spectroscopy (AES), optical and scanning electron microscopy (SEM), and secondary ion mass spectrometry (SIMS) were used to study composition changes at the interfaces. Non-linear I-V curves were obtained for the as deposited contacts, while the curves became linear and the resistance was lower upon annealing at temperatures greater than 150°C for 15 minutes. Resistance increased 5-10% with annealing time up to 90 minutes, and the resistance increased by a factor of 25 at a temperature of 350°C for 15 minutes. AES depth profiles were collected from samples annealed for 90 minutes at both 200°C and 250°C . No compound formation was detected while the interfacial width increased with increasing temperature. SEM micrographs showed a coalescence and surface diffusion of Au up to $100 \text{ }\mu\text{m}$ across the surface at high temperatures (250°C) leading to an extended zone around the dot contacts with a higher Au concentration. No reaction zone was observed at 200°C . SIMS data were collected, but did not aid in determining diffusion profiles or identifying the reaction which led to the surface spreading.

(c) GaN Contacts

Studies of the formation of ohmic contacts to p-type GaN have continued. The emphasis has been on investigating the Au and Ni/Au contact systems. Au layers approximately 1000 \AA thick were deposited by D. C. planar magnetron sputtering onto p-type GaN thin films. The GaN was deposited by low pressure MOCVD at APA Optics and had a measured sheet resistivity of 2-8 Ω/square . The as deposited current-voltage characteristic for these contacts are rectifying. The samples were heat treated to 200° , 400° , and 600°C and the estimated barrier height of the Schottky contacts was lowered for the two highest heat treatments, but ohmic behavior was not observed. By characterizing the contact morphology with scanning electron microscopy (SEM), it was observed that the Au film coalesced into islands after heat treating at 600°C for 15 minutes. Auger analysis of the interface is pending.

Au/Ni ($1000 \text{ \AA}/500 \text{ \AA}$) were deposited by electron beam evaporation for the Ni layer followed by sputter deposition of the layer. These contacts were also rectifying in the as deposited condition. Heat treatments of 200°C and 600°C resulted in a reduction of the

barrier height, but again ohmic behavior was not demonstrated. SEM revealed that coalescence did not occur at 600°C for the Au/Ni contacts.

(II-B) Raman Studies of ZnSe and ZnS_xSe_{1-x} (Paul Holloway)

A study of the effects of sulfur content on the Raman shift of the p-ZnSe Raman signal was begun. Raman spectra were measured for three samples: ZnSe/GaAs, ZnSe/ZnSe, and ZnS_xSe_{1-x} where x=0.055. Plots of the ZnSe peak position (Fig.II.1) and FWHM (Fig. II.2) are shown for preliminary curve fits of the data. The shift of the peak from ZnSe heteroepitaxed to GaAs and ZnSe homoepitaxed to ZnSe shows a reduction of stress in the lattice. The addition of S to ZnSSe is reported in the literature to shift the Raman peak to lower wave numbers, whereas the data in Fig. II.1 show it shifted to higher values in the 3M epilayers. This could result from large strains in the layer; further work is in progress.

(III) Microstructural Analysis of II-VI and III-V Materials (Kevin Jones)

a) Electrical Degradation Study of II-VI LED Structures

Investigation of the microstructural changes in degraded LED's was performed using electroluminescence (EL) microscopy and transmission electron microscopy (TEM). The single Cd_{0.2}Zn_{0.8}Se quantum well structure of the LED is similar to that described in the previous report. A structure with an as-grown defect density of <math><10^6/\text{cm}^2</math> was chosen for the degradation study. The conditions used for degradation were as follows: current density=100A/cm², repetition rate=10KHz, duty cycle=40%. The LED's were degraded for times ranging from 15 mins. to 1 hr. The short and long times were chosen for TEM analysis.

Cross-sectional TEM analysis of the degraded structures showed dislocations that were mainly confined to the quantum well region. Dislocations also seemed to emanate from the quantum well area and spread into the surrounding layers. Plan-view TEM analysis of the degraded structures showed two sets of dislocation lines perpendicular to each other oriented along the $\langle 100 \rangle$ directions. EL microscopy of the degraded structures showed two sets of dark line defects parallel to the $\langle 100 \rangle$ directions. The spacing between these defects decreased with increasing degradation time with the separation distance dropping from 600nm after 15 minutes to 330nm after 1 hour. The evidence so far seems to suggest that the defects observed in plan-view TEM are a close-up view of those observed by EL microscopy and are responsible for the degradation of the LED's. It also appears that the strained quantum well area is the source for the generation of degradation-induced defects. In order to further understand the mechanism for dislocation generation during degradation, samples that are in the early stages of degradation will be studied in the near future.

b) Characterization of nitride based compound semiconductors

Nitride based compound semiconductors grown on (0001) ZnO, (0001) sapphire and (001) GaAs substrates were characterized using cross-sectional TEM. ZnO (0001) substrates were provided by Litton Industries for growth of lattice-matched InGaN and InAlN layers.

Fig. II.1. Raman Peak Position for ZnSe heteroepitaxed to GaAs, homoepitaxed to ZnSe, or $\text{ZnS}_{0.055}\text{Se}_{0.945}$ heteroepitaxed to GaAs.

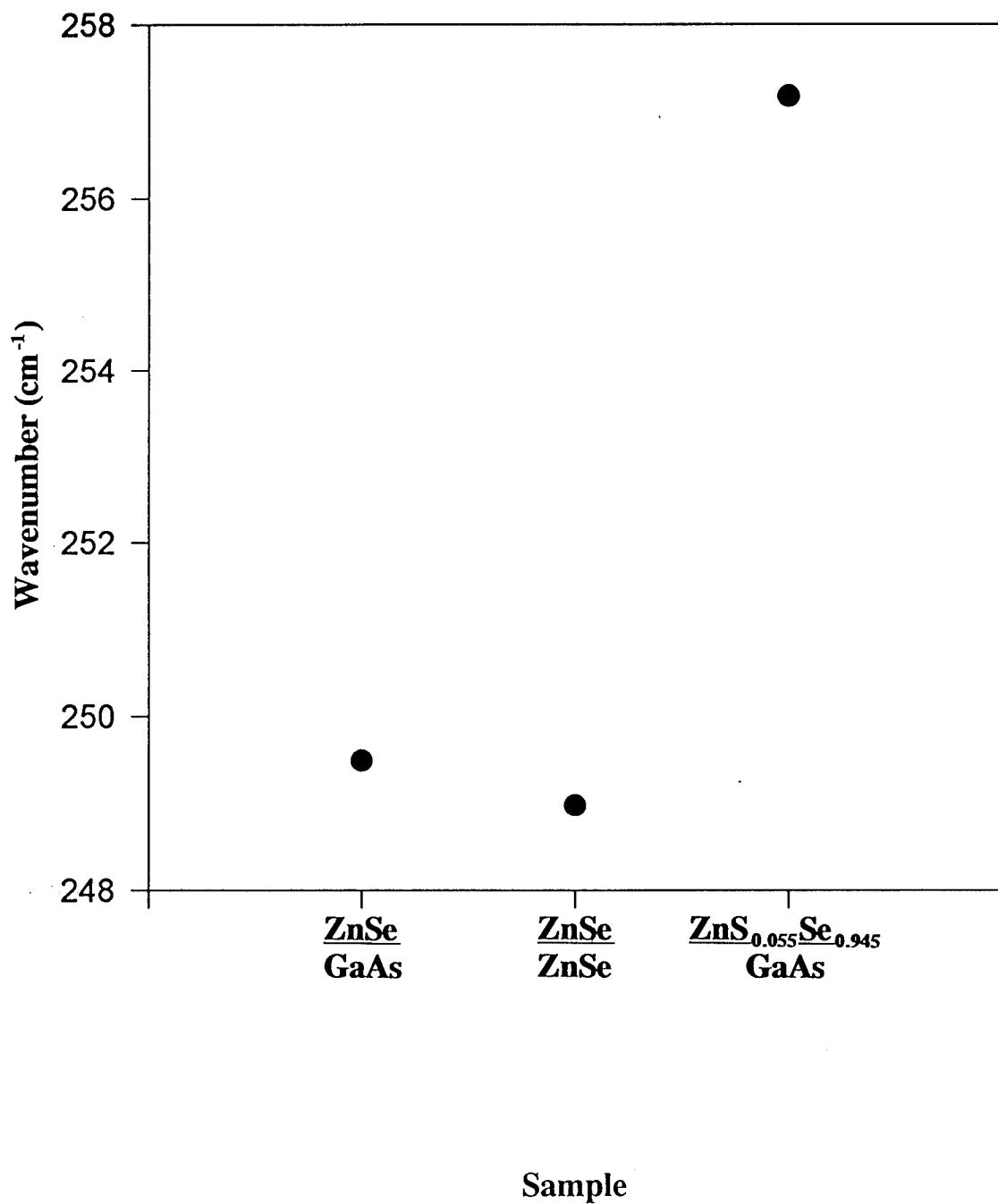
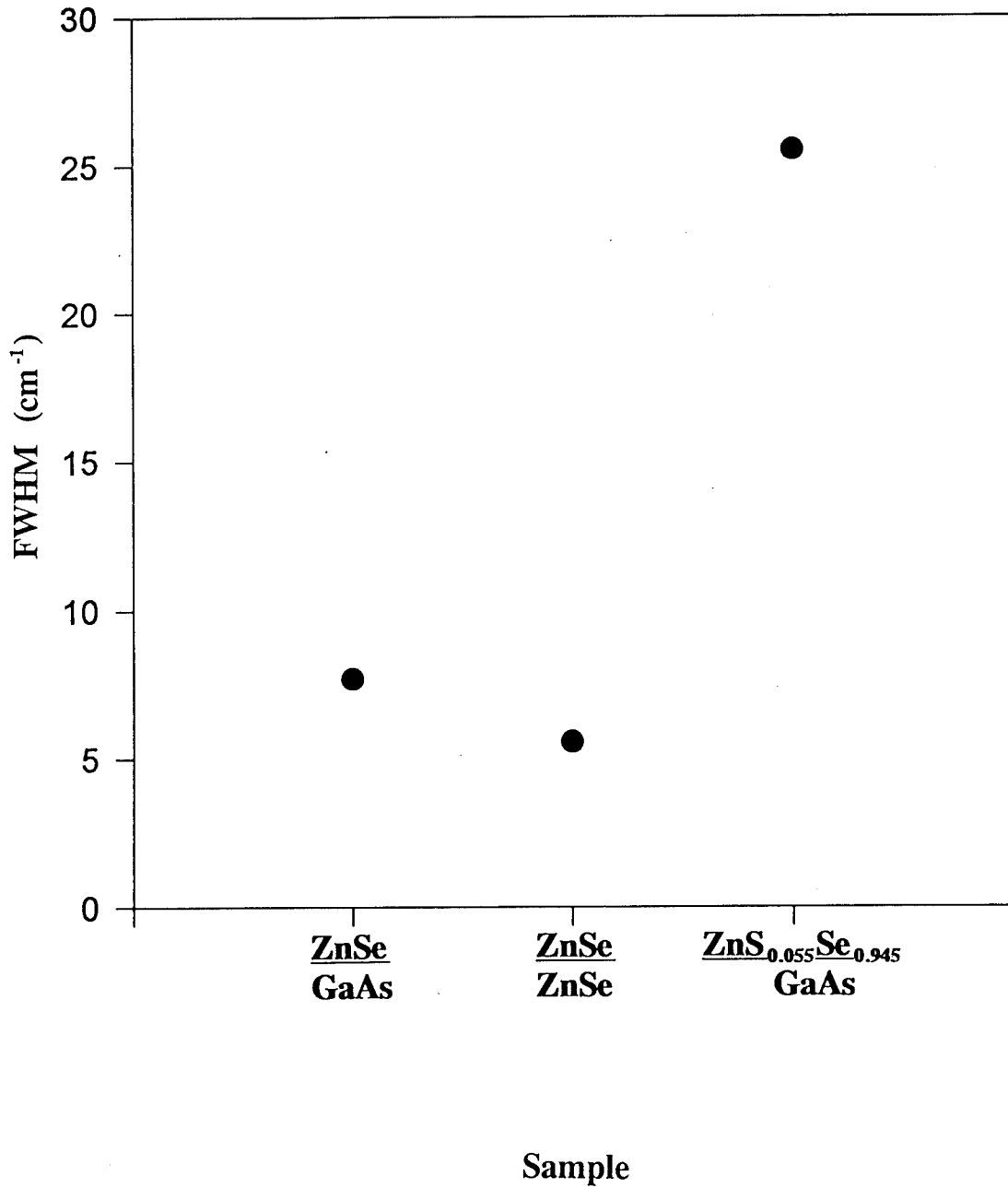


Fig. II.2. Raman Peak FWHM for ZnSe heteroepitaxed to GaAs, homoepitaxed to ZnSe, or ZnS_{0.055}Se_{0.945} heteroepitaxed to GaAs.



Prior to growth, high resolution x-ray diffraction (HRXRD) analysis was performed on these substrates to determine their crystalline quality. The FWHM for the (0002) peak was found to be in excess of 130 arc-seconds (compared to 15 arc-secs. for (0001) sapphire substrates). This clearly indicates that the substrates are defective and not optimum for growing high quality, lattice-matched layers. None-the-less, growth of a lattice-matched $\text{In}_x\text{Al}_{1-x}\text{N}$ ($x=0.35$) layer was attempted on these ZnO substrates using metalorganic molecular beam epitaxy (MOMBE) followed by analysis using TEM. The analysis showed the layer to be polycrystalline in nature with a columnar structure. The substrate showed evidence of decomposition near the interface region, possibly due to the high growth temperature (600°C). Over the next quarter, we will attempt to acquire better ZnO substrates for growth and to optimize the growth conditions for lattice-matched InGaN and InAlN growth.

GaN layers were grown by MOMBE on (0001) sapphire substrates subsequent to the growth of a 20nm thick AlN buffer layer. The GaN layer was found to be polycrystalline in nature. The defects in the underlying AlN buffer did not seem to propagate into the GaN epilayer. This indicates that by better optimization of the buffer (thickness and growth temperature) one can improve the GaN layer quality.

InN layers were grown on (001) GaAs substrates at 525°C and subsequently annealed at 550, 650, 750 and 850°C to study interdiffusion of the various species. The samples were analyzed using Auger electron spectroscopy (AES). The results showed that annealing at 650 and 750°C caused In diffusion into the GaAs substrate and Ga outdiffusion into the InN layer. Annealing at 850°C completely depleted the surface of In and only a Ga signal was detected. Cross-sectional TEM specimens are being prepared to study the microstructures after the various interdiffusions.

(IV) Optical and Electrical Characterization of ZnSe (J.H. Simmons)

To study the effects of structural defects on the behavior of carriers in QW films in collaboration with R. Park, we have begun to measure carrier behavior in ZnCdSe/ZnSe quantum well stacks with different well width and barrier thickness. This study will identify the kinds of photoexcited carriers formed in the wells and the dynamics of their behavior with respect to trajectories in and between the wells. We expect to observe exciton and biexciton formation, measure their lifetimes and determine the degree of interwell tunnelling. The results will be reported in the next quarterly.

(V) MOCVD Growth of GaN Thin Films (Tim Anderson)

The effect of MOCVD deposition length on the thickness and the structural quality of GaN films has been studied as part of this on-going research. Under identical growth condition, a series of GaN films was grown at 850 °C on Al_2O_3 (0001) substrates. A low temperature GaN buffer layer was deposited prior to each high temperature deposition. Film of different thicknesses were characterized by a Dektak thickness profilometer and by High Resolution X-Ray Diffraction (HRXRD).

All of the GaN films were transparent. However, shades of various colors were observed when the surface was viewed obliquely, consistent with a variation in the

thickness. Like insulating SiO_2 and Si_3O_4 layers, the color of GaN film follows a characteristic cyclic pattern that depends on its thickness and index of refraction. The change in thickness with growth time is shown in Fig. V.1. As expected, the thickness of the film increased with longer growth times. Above 1300 Å, the growth rate is constant with time. This growth region corresponds to the epitaxial bulk layer grown during the high temperature deposition. However, a non-linear growth region is also evident below 1300 Å. The film formed during low temperature buffer deposition and few minutes of growth may have contributed to this non-linear region.

X-Ray peaks corresponding to (0002), (0004), and (0006) reflections of wurtzite GaN were observed from these films. These peaks were very narrow and sharp, indicating that the films were epitaxial with their C-axis oriented along the substrate's C-axis. The lattice constant of the GaN films along the C-axis was calculated from HRXRD omega rocking curve using the (0002) peak. This value was very close to the C values obtained from the second and third order reflection peaks. The change in the C values as a function of growth time is also depicted in Fig. V.1. There appears to be three distinct regions: (a) an initial strain relieving region up to 3000 Å, (b) an intermediate strain enhancement region between 3000 and 5000 Å, and (c) a final strain relieving region above 5000 Å. The lattice constant, C of an unstrained wurtzite GaN film is 5.185 Å. More work is underway to explain these observations.

(VI) Development of Diode Lasers (Peter Zory)

(a) Diode Pumping

Single quantum well, CdZnSe unprocessed material capable of lasing cw at room temperature was received from Michael Haase of 3M. Part of the material is being processed in a form suitable for use in TEM degradation studies (collaboration with Dr. Jones and student, J.S. Kim). The other part is being processed into surface-emitting LEDs for thermal performance studies and comparison to similar devices made at 3M.

An unprocessed InGaN double-heterostructure LED wafer has been received from Shuji Nakamura of Nichia Chemical. Luminescence studies and comparisons with other laser-type structures in different material systems are underway. The goal is to understand the nature of the p-type conduction mechanism in GaN-based LEDs and its relationship to the problem of designing GaN-based diode lasers.

(b) Photopumping

The 337 nm nitrogen laser is being used in the quality evaluation of GaN-based films grown by MOCVD in Dr. Anderson's laboratory. The quality of his GaN is improving with more experience in growth of GaN on sapphire.

(c) Electron Beam Pumping

Cleaved facet, ZnSe thin film devices fabricated from material grown at UF and used previously in optical pumping experiments at UF have been sent to McDonnell-Douglas for use in electron beam pumping experiments. The McDonald-Douglas work is supported by an ARPA contract entitled "Electron Beam Semiconductor Laser Projector Technology" and involves interactions with various institutes in the former Soviet Union.

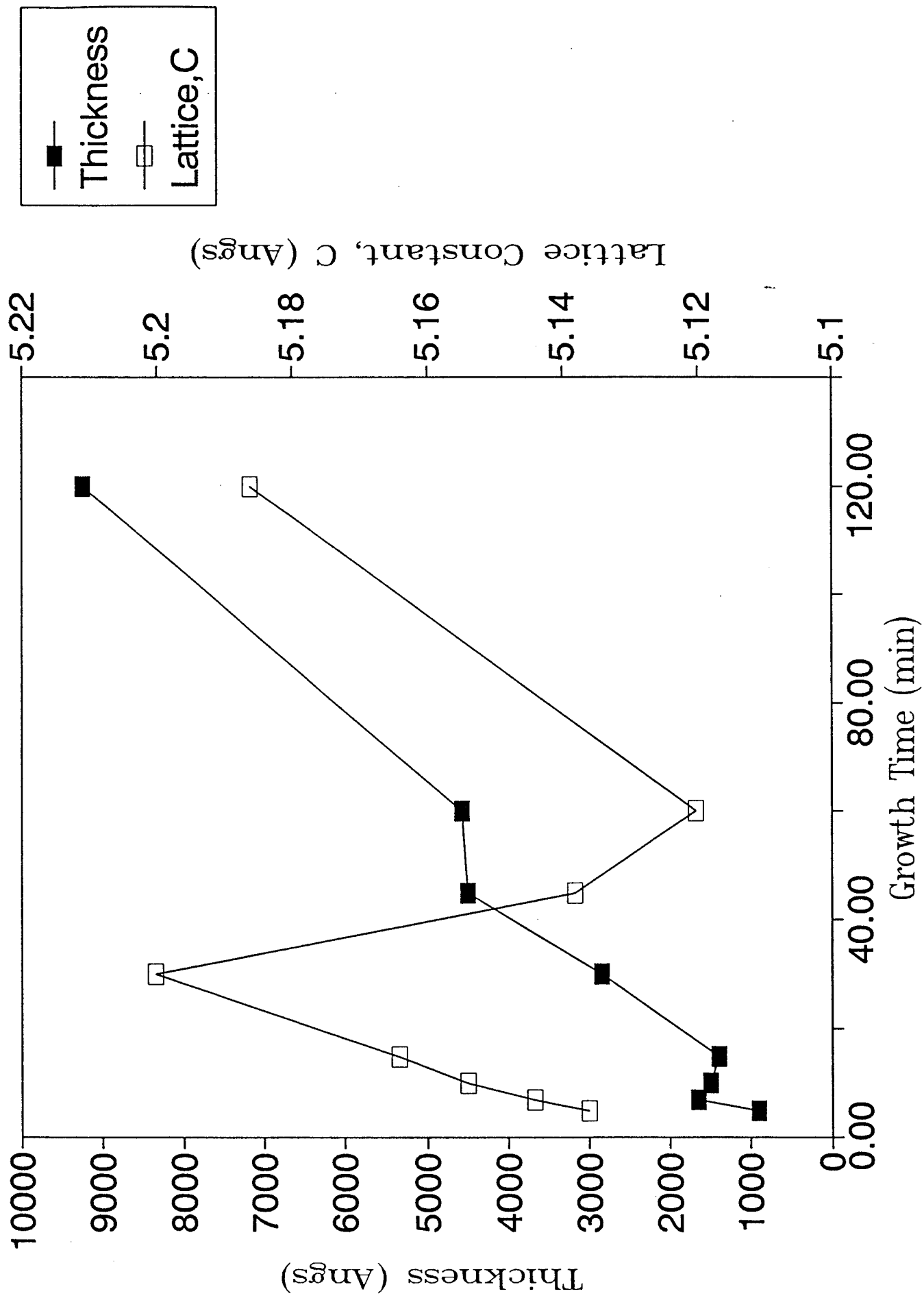


Fig. V.1.

(VII) Theoretical Calculations of Dopants of ZnSe (Gertrude Neumark)

As described in the previous progress report, we have obtained a very satisfactory fit between screening theory [G.F. Neumark, Phys. Rev. B5, 402 (1972)] and a change with temperature in the activation energy of ZnSe:N, which had been reported in the literature [Bowers et al., J. Electr. Mat. 23, 251 (1994)]. This means there is no need to invoke an interstitial location for N, which had been the explanation suggested by Bowers et al. This work had been written-up in Mr. Kushovskiy's MS thesis. In addition, the thesis also includes a novel approximation to the screened hydrogenic Schoedinger Equation which shows that for relatively small Bohr radii (as in ZnSe:N), the actual value of this radius is not required for evaluation of the screened energy (as had been necessary in prior work).

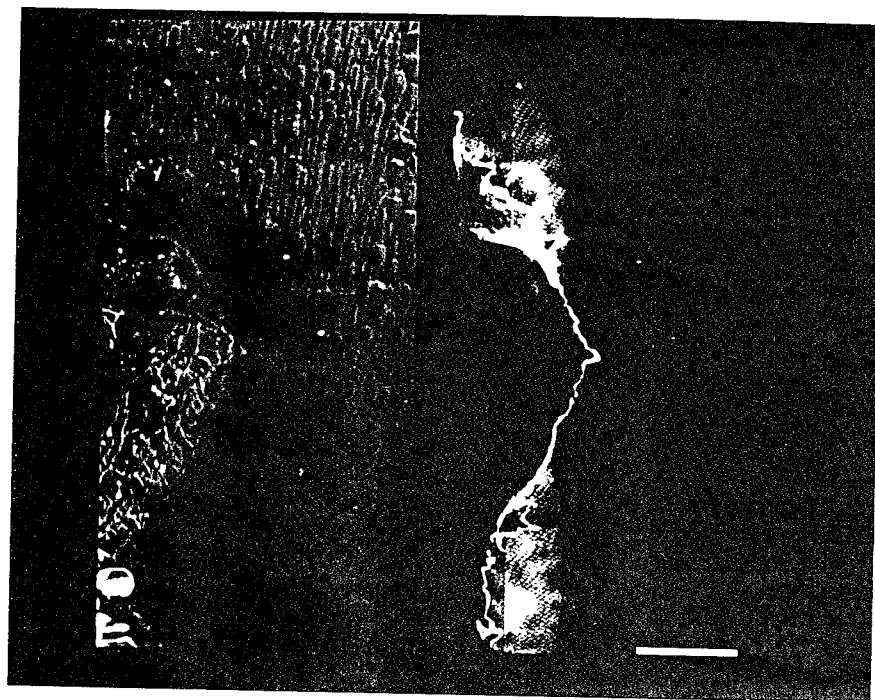
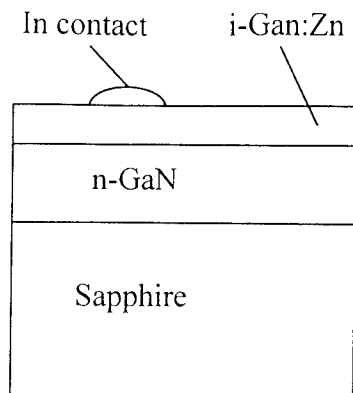
The application of the screening theory to the above-mentioned data, obtained via luminescence, was relatively easy, since free carriers are expected to dominate the screening in this instance. However, this should be extended to analysis of electrical data of carrier concentration versus temperature in order to obtain proper values of the compensation ratio. This analysis requires, in addition, proper consideration of screening by charged ions. We are presently investigating this point.

(VIII) MOCVD Growth of GaN (Jacques Pankove)

The emphasis in crystal growth this quarter has been on utilizing and characterizing the buffer layer technique to further improve the crystal quality. In addition, we have verified the consistency and repeatability of crystal growth since the two growth systems were separated last quarter. We have again improved upon our best material to date with a sample grown on a GaN buffer layer. This sample has an electron mobility of $220 \text{ cm}^2/\text{Vs}$ and an electron concentration of $4.5 \times 10^{17} \text{ cm}^{-3}$. We expect that further optimization of the buffer growth conditions will produce the decrease in carrier concentration necessary for p-type doping, and we are consequently planning to begin doping studies soon.

In addition to the emphasis on growth, we have also expanded our characterization capabilities by adding an electron beam induced current (EBIC) stage to our JEOL-35C scanning electron microscope. This feature will allow us to more completely characterize p-n junctions to be used in LEDs. Shown in Figure VIII.1 is a split SEM image of the indium contact of a GaN m-i-n structure LED. The image on the left is the secondary electron image showing half of the indium contact and the surface of the Zn-doped i-layer. The image on the right is the EBIC image of the same area. The bright line following the edge of the In contact marks a region in which electron-hole pairs excited by the SEM's electron beam are separated by the built in electric field of the junction and are collected and measured as the EBIC signal. We expect this addition to be a valuable tool in achieving high quality p-n junctions and LEDs.

We have also expanded on our work on the temperature dependence of photoconductivity in both undoped and Mg-doped GaN films. Figure VIII.2 shows a plot of E_0 (as defined by the expression $PC = A \exp(E/E_0)$) versus temperature for a Mg-doped GaN sample. We hope to understand this behavior through further analysis.



GaN M-I-N diode

Side View

SEM -- Top view

Fig. VIII.1. EBIC Image of GaN m-i-n LED.

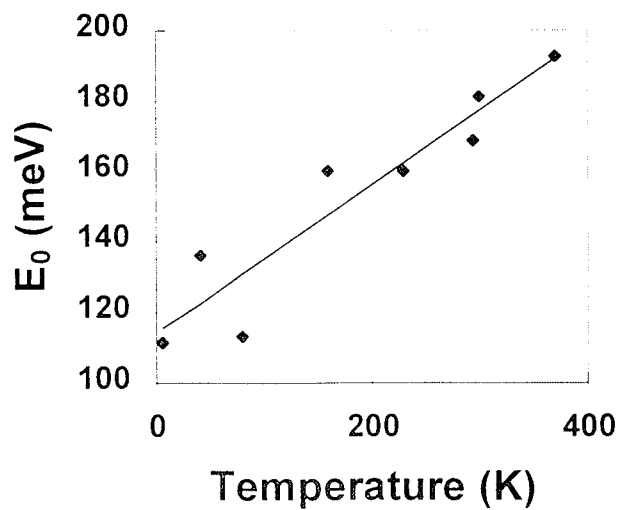


Fig. VIII.2. E_0 vs. temperature for Mg-doped GaN sample.

(IX) Gain Modeling in II-VI Strained-Layer QW Structures (Reinhart Engelmann)

The modeling of a wide band gap heterostructure has been performed on the two dimensional device simulation program MEDICI. The advantage of such a simulation program is the fact that it supports a variety of physical models such as the Poole-Frenkel effect which is important in explain hole freeze-out in p-type ZnSSe and ZnMgSSe thin films. Besides QW structures, a novel Si bipolar transistor with a ZnS emitter has been simulated.

a) Two-Dimensional Device Simulation

As the structure of high performance lasers has become increasingly more sophisticated, it has become evident that the multidimensional nature of light and carrier interactions need to be taken into account in laser device modeling. Recently, several in-house two-dimensional diode laser simulators have been reported that are not commercially available.

For electrical modeling, the problem is similar to the one in transport device models which includes solving the Poisson and continuity equations. Because there are several elaborate commercial transport device simulators available (such as MEDICI from Technology Modeling Associate, Inc.), integrating our optical model into these well develop electrical models will speed up our development time. Further more, the important effects in II-VI semiconductor transport modeling such as the Poole-Frenkel effect, has already been incorporated into the simulator [1]. As pointed out by Mensz [2], the Poole-Frenkel effect may play an important role in determining conductivity of p-type ZnSSe and ZnMgSSe thin films.

b) Device Simulation of ZnS/Si HBT

As a test case for the MEDICI simulator which we have available, we simulated a simple heterojunction bipolar transistor (HBT) relevant to our II-VI technology. We proposed and analyzed a novel Si-HBT with a ZnS emitter. The lattice constant of ZnS is 5.410 Å, very close to that of Si which is 5.43 Å (mismatch only about 0.3%). Exact lattice matching can be realized by incorporation of small amounts of Cd or Se to form the ternaries CdZnS or ZnSSe. Recently, through the rapid development in ZnSSe-based blue-green diode laser and LED technology, epitaxial growth and doping of ZnSe and ZnS based alloys have been extensively studied with the break-through in p-type doping (by N) and good quality n-type doping (by Cl) allowing the study of a variety of p-n junction devices.

An ohmic contact to n-type ZnSSe is readily available with In or Au. Thus, a semiconductor device based on the ZnS/Si heterostructure system becomes a realistic possibility. The band gap of ZnS, which is 3.66 eV, is much larger than that of Si, which is only 1.12 eV. This large band gap difference (more than 2.5 eV) leads to very interesting device physics. Depending on the band offset, which is currently not well characterized, several useful device applications are possible. If the conduction band off-set remains relatively small, estimated by the affinity rule to be near ~ 0.1 eV, a Si based npn type heterostructure bipolar transistor (HBT) with ZnS (or alloy) as an emitter can be fabricated. The simulation of such a device on MEDICI shows that a current gain beta of 100-1000,

with an associated f_t of 50-175 GHz, can be obtained with an uniformly doped un-optimized structure. If, on the other hand, the conduction band offset is relative large, estimated from the Harrison-Tersorff model to be near ~ 0.4 eV, a hot electron HBT with high-speed operation can be designed. If, the conduction band offset is so large that it is greater than the band gap of Si, as reported by Maierhofer et al. [3], then a so-called Auger transistor with a current gain alpha larger than one could possibly result.

c) Future Plan

We plan to continue the work with the two-dimensional device simulation and apply it to blue/green light emitting structures.

References:

- L. Pelaz et al., IEEE Trans. on Electron Devices, Vol. 41, No. 4, 587, 1994.
- P.M. Mensz, Appl. Phys. Lett. 65 (21), 2627, 1994.
- Ch. Maierhofer et al., J. Vac. Sci. Techn. B9 (4), 2238, 1991.

Presentations this quarter

- J. Fijol, L. Calhoun, R. Park, P. Holloway, "Comparison of Sputtered and Evaporated Au and Ag Contacts to p-ZnSe", 23rd Annual Symp. of the Florida Chpt. of the Amer. Vac. Soc., Clearwater Beach, FL, February 6-8, 1995.
- J.T. Trexler and P. Holloway, "Ohmic Electrical Contacts to p-ZnTe", 23rd Annual Symp., Florida Chpt. of the Amer. Vacuum Soc., Clearwater Beach, FL, February 6-8, 1995.
- S.J. Miller and P. Holloway, "Electrical Contacts to GaN Thin Films," 23rd Annual Symp., Florida Chpt. of the Amer. Vacuum Soc., Clearwater Beach, FL, February 6-8, 1995.
- Y. Cai and R. Engelmann, "Device Physics of ZnS/Si Heterojunction Transistor," Annual Meeting of the Oregon Academy of Science, Reed College Portland, OR, Feb. 25, 1995.

Publications during quarter

- "Preferential donor-acceptor pairing in heavily N-doped ZnSe?," G.F. Neumark, L. Radomsky, I. Kuskovskiy, Proc. SPIE, Vol. 2346, p. 157 (1994).
- "Screening Phenomena and their Application to Acceptor Activation Energy Calculations in ZnSe:N," - Thesis; Master's degree awarded to Mr. I. Kuskovskiy.
- "Design of Novel Blue/Green Diode Laser Based on MgZnSeTe Alloy," Y. Cai and R. Engelmann, Solid-State Electronics, Vol. 38, No. 3, pp. 734-736, 1995.

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