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ANNUAL TECHNICAL REPORT

OCT. 1978 - SEPT. 1979

EVALUATION OF GALLIUM NITRIDE FOR ACTIVE MICROWAVE DEVICES

Contract Authority: NR 243-004

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Planar single crystal layers of GaN that are almost defect free and completely transparent can now be grown routinely by CVD on basal plane sapphire substrates. The native shallow donor density still remains high (10^{18}- $10^{19}/cm^3$) unaltered by growth under varying Ga:N ratios, but decreasing somewhat with increased growth rate. The results of growth under N_2 at high pressures are inconclusive at present. Doping with Ge

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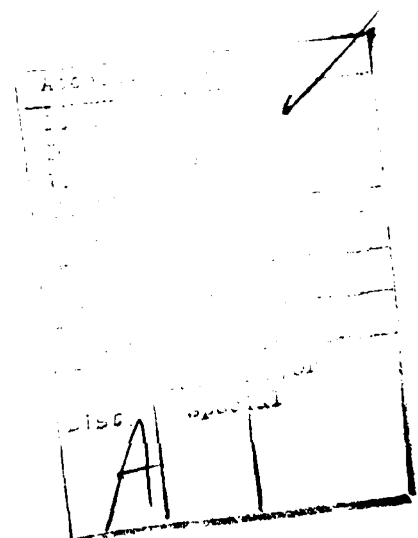
or C during growth does not compensate the donor. Low temperature photoluminescence studies of proton implanted samples and of samples grown in the absence of H_2 provide additional evidence that the shallow donor is not associated with H. Work is in progress to interpret much fine structure observed in the luminescence spectra, to complete the identification and characterization of donors and acceptors introduced by ion implantation and to observe any effects on the native donor density caused by the implantation of Ga and of N.



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INTRODUCTION

GaN is a wide bandgap (3.3 eV) direct gap semiconductor with a low electron effective mass (0.2) and a high optical phonon energy (120meV). As a result, both its saturated drift velocity and its pair production threshold should be high, making it an excellent candidate as an IMPATT device material for high power-high frequency microwave amplifiers. As such, its figure-of-merit is 20 times greater than that of Si. Under this Contract, our object over the past several years has been to show experimentally that this is indeed the case, and that GaN can be handled as a "normal" semiconductor material.

In order to measure the saturated drift velocity and pair production in GaN, single crystals are required with uncompensated electron concentrations of $10^{17}/cm^3$ or below. We have shown previously that single crystals of GaN could be grown epitaxially on sapphire substrates at 1050^oC by the chemical vapor deposition reaction between GaCl and NH_3. The true melting point of GaN in equilibrium with N_2 is very high ($\sim 2{,}000^o$ C) and the corresponding equilibrium pressure of N_2 is extremely high ($\sim$ 40,000 atm.) However, NH_3 at lower pressures and temperatures serves as an active form of N_2. At 1000^oC the three-phase $[(GaN)_{solid} - (Ga{+}N_2)_{liquid} - (N_2)_{gas}]$ equilibrium corresponds to a N_2 pressure of 100 atm. If NH_3 is used instead of N_2 the $2NH_3 \rightarrow N_2 + 3H_2$ equilibrium must be considered. At 1000^oC, the equilibrium is almost completely towards the N_2 side. 100 atm. of N_2 should be in equilibrium with only about 5 Torr of NH_3. However, below 1100^oC, the decomposition of NH_3 into N_2 and H_2 is extremely sluggish in the absence of a suitable catalyst. Thus, below 1100^oC, GaN can be grown using 5 Torr of NH_3 instead of 100 atm. of N_2 rendering crystal growth feasible without resorting to high pressure equipment.

Sapphire is the only substrate compatible both with the crystal structure of GaN and with its lattice constants, and with the GaCl CVD growth chemistry. At first, our single crystal layers of GaN grown on sapphire by this method, were characterized by electron densities which decreased with thickness, and were of order of $10^{20}cm^{-3}$. Hence, our initial goals were to grow very thick layers. Orientation experiments indicated that the most rapid growth occurred on the R-plane sapphire orientation. Thus, we set out to grow thick GaN single crystals on R-plane sapphire. GaN layers as thick as 5mm were produced on substrates of area 1 x 2cm. However, these crystals exhibited microcracks, voids, Ga precipitation and the carrier density remained high, in the $10^{20}cm^{-3}$ range. The microcracks were due to differential thermal contraction between GaN and sapphire on cool-down from the growth temperature. An analysis of the resulting stresses showed that epitaxial layers of thickness greater than 200 μm would always crack. We showed that layers greater than 200 μm could be grown free of cracks by first growing a layer less than 200 μm thick, grinding away the sapphire substrate, then continuing the growth. The voids were shown to be due to the formation of equilibrium GaN surfaces which tended to form overhangs during growth. These led to the development of cave-like structures which impaired the flow of the reactant vapor species required for growth. Hence, stagnant gas pockets developed in the caves with the resultant formation of voids. Slow growth rates (∿ 1 μm/hr.) avoided this problem. The precipitation of Ga on the growth planes, with Ga particles <100 Å in size could not be controlled by attempting to stabilize the reactant flow rates. However, the problem was eliminated by using slow growth rates (again ∿ 1 μm/hr.). Thus, good single crystal layers, up to 5 mm thick could be grown.

These layers, however, were still heavily doped, n-type, to $\sim 10^{20}/cm^{-3}$.

We then showed that the donor responsible was Si, a shallow donor derived from the fused silica walls of the growth apparatus and as a residual impurity in our starting materials. By lining the furnace walls with alumina tubes and improving the purity of our starting materials, we were able to eliminate the Si contamination. The electron density then dropped to 2-7 x 10^{18}/cm^{-3}, independent of crystal thickness.

Because this level of electron density leads to a degenerate carrier distribution, electrical measurements (Hall Effect and resistivity) vs. temperature were useless in deducing the properties of the donor or donors responsible. However, low temperature (4.2°K) near-gap photoluminescence, which revealed bound exciton and donor-acceptor peaks, could be used to deduce the properties of many of the impurities in the crystals. Last year, by correlating the peak positions and intensities with doping, either during crystal growth, or by ion implantation and subsequent annealing to remove lattice damage (1100°C in an NH_3 atmosphere), we effectively scanned much of the Periodic Table to determine which elements were shallow donors and which were shallow acceptors in GaN and we deduced their ionization energies. We found that even the shallowest acceptors (Zn, Cd, Ge, Mg) were relatively deep (> 150 meV), so that we deduced that low resistivity p-type GaN would be impossible to achieve at room temperature. The dominant deduction, however, was that the shallow donor (10^{18}-10^{19}/cm^3) responsible for the degenerate n-type behavior of all of our undoped samples was not due to any chemical impurity; it must be due to a shallow (30 meV) native defect, with the proviso that hydrogen was only eliminated as a possibility on the basis of circumstantial evidence.

Therefore, our program for this past year was based on the following assumptions:

1. GaN cannot be prepared low resistivity p-type. Therefore, microwave devices must be based on Schottky barrier structures.
2. The dominant shallow donor is a native defect and its density must be greatly reduced. (However, hydrogen was never eliminated definitively.)
3. The decrease of donor density with crystal thickness was due to an impurity, Si. With its elimination, very thick crystal layers are no longer necessary.

Hence our experimental program this past year consisted of (1) growing good crystallographic layers of GaN, not necessarily thick, (2) investigating the effects of hydrogen, (3) attempting to influence the presence of the native shallow donor by growing under extreme conditions of N_2 and Ga, and (4) completing the determination of shallow donors and acceptors in GaN via low temperature photoluminescence studies of crystals doped during growth or by subsequent ion implantation.

We note at this point that we were only partially funded for what we expected to do this past year, and that we were only made aware that our funding was to be reduced from what we had anticipated approximately midway through the year. Thus, we began the contract year with an ambitious program, only to completely run out of support approximately midway through the contract year. Hence, the results reported here are based only on 6 months worth of effort. Because of this change of support in midyear, some of the personnel involved left the program at that point and will not be involved in any continuation of this program.

CRYSTAL GROWTH

Prior to this year, our CVD (GaCl + NH_3) growth used R-plane sapphire substrates. This substrate orientation, on which $(11\bar{2}0)$ prism plane GaN grew, allowed us to grow rapidly, producing the very thick GaN crystals we desired. However, the growth surfaces were non-planar. $(\bar{1}01n)$ pyramidal facets (with $n \geq 2$) developed on the growth surface. The re-entrant edges produced by the intersection of these facets facilitated rapid growth; but these also led to the development of the hanging terraces, resulting in caverns and leading eventually to the formation of voids. With the elimination of Si contamination, thick crystals were no longer necessary; the carrier density no longer depended upon crystal thickness. This year, therefore, we have concentrated on crystal growth on basal plane sapphire which leads to planar growth of basal plane (0001) GaN.

Basal plane sapphire substrates are more expensive than R-plane substrates (normally used for silicon-on-sapphire growth) and the surface energy for nucleation of GaN on basal plane sapphire is lower than for nucleation on R-plane substrates, leading to greater sensitivity to the influence of contamination. However, the resultant GaN crystals exhibit mirror-like surfaces, with no evidence of faceting and the voids and Ga precipitation, characteristic of R-plane growth can be completely eliminated. Thus the crystals are completely transparent.

The previous R-plane growth was always done under an excess of NH_3; the arrival rate of Ga always determined the growth rate. Here, on basal plane substrates, we started from the beginning, varying the flow rates of both the GaCl and the NH_3 for two reasons. First, we wished to establish the conditions for best nucleation (much more crucial on basal plane substrates)

and growth. Second, as discussed below, we wished to vary the surface concentration of N to determine if we could influence the incorporation of the dominant native donor.

The total flow rate (NH_3 + HCl + H_2) was varied between 930 and 1850 cc/min. The net pressure of injected NH_3 ranged from 0.23 to 0.40 atm and that of HCl from 2.3×10^{-3} to 4.4×10^{-3} atm.

At the lower NH_3 pressures, nucleation consists of a small number of hexagonally shaped islands, which grow both laterally and in height, until they merge, without producing grain boundaries, to form a stepped, non-planar surface. At the higher NH_3 pressures (>0.35 atm), nucleation consists of a much greater density of islands. These merge before the isolated islands have grown very thick. Thus the resulting surface is much more planar. Growth then proceeds by a step-wise, planar (two-dimensional nucleation) means and the resultant crystals grown, at 35-60 μm/hr, are planar, with a mirror-like surface.

Crystals grown at the lower NH_3 pressures exhibit some internal light scattering, probably due to Ga precipitation, but the planar structures grown at high NH_3 pressures are completely transparent. The only defects that are visible are some hexagonal etch pits on the surface. These may be due to preferential etching by the HCl or facet formation during growth, both mechanisms due to the protrusion of dislocations through the surface. At higher growth rates the density of such etch pits is reduced but their size (4-170 μm) is increased.

The morphology of these crystals and their structural defects (cracks, voids, precipitates) are greatly improved over our previous GaN layers grown on R-plane sapphire substrates. However, from Hall Effect and resistivity and from luminescence measurements, we note that the native shallow donor density

remains in the range 3-10 x $10^{18}/cm^3$. Thus, the native donor cannot be due to, or associated with, any of these simple structural defects.

Our previous growth on R-plane substrates was always done under NH_3-rich conditions: the growth rate always varied linearly with the arrival rate of GaCl, i.e. with the HCl flow rate. From this, we deduced that the growing surface was always saturated with an adsorped NH_3 layer (or a chemisorbed N-containing species, such as NH_2, NH or even N). If the native donor incorporation was being determined by equilibration with an external phase, in this case a constant concentration of N species on the growing surface, then the donor density would remain constant. Thus, we tried to grow GaN under conditions where we were not saturated with NH_3, that is, where the growth rate depended on NH_3 as well as on Ga.

At NH_3 pressures below 0.15 atm island nucleation does not occur on sapphire, only whiskers form. Above 0.15 atm, island nucleation occurs and subsequent growth leads to coherent crystals. At low NH_3 pressures, the growth rate depends on both the NH_3 and the Ga arrival rates, but the resultant electron concentration was found to depend on the net growth rate, not on the NH_3/Ga ratio. The electron density decreased from 8 x 10^{19} cm^{-3} to 1 x 10^{19} cm^{-3} as the growth rate increased from 14 to 60 μm/hr. Hence the defect probably is a native defect, but it is not in equilibrium with the surface ratio of Ga to N.

These experiments on both R-plane and basal plane sapphire substrates, as well as experiments in which a second substrate was placed behind the first substrate in the growth chamber, clearly indicate that the growth mechanism is kinetically controlled, with mass transport being the dominant controlling agent.

We noted above that hydrogen was eliminated as the source of the dominant

shallow donor on the basis of circumstantial evidence only. Our crystals are grown with H_2 as the carrier gas. H_2 is part of the equilibrium reaction by which NH_3 reacts with GaCl to form GaN. Previously we had tried using N_2 instead of H_2 as the carrier gas, with no significant change in the resultant electron density in the GaN grown; but the optimum growth parameters were changed. This time we replaced the H_2 with He. He and H_2 are well matched with respect to viscosity and thermal conductivity. Crystals grown on R-plane sapphire using He instead of H_2 were more planar, the density of nuclei increased and their mergence occurred at lower thicknesses, i.e. the crystals were of better structural quality. However, the carrier density remained high (5×10^{19} cm^{-3}) providing more circumstantial evidence that hydrogen is not associated with these electrically active defects.

HIGH PRESSURE-HIGH TEMPERATURE N_2 PROCESSING

Assuming that the dominant shallow donor is a native defect, we have attempted to anneal GaN and to grow crystals under varying pressures of N_2 rather than NH_3 and at temperatures closer to the true equilibrium melting point.

GaN crystals, previously grown by our standard CVD growth technique were heated to 1200°C under N_2 pressures up to 9,000 atm. in our high pressure-high temperature apparatus. No change in carrier density was observed, but the crystals were observed to exhibit some surface decomposition, presumably due to Ga transport as Ga_2O by residual O_2 or H_2O contamination in the system. Accordingly, any out-diffusion or in-diffusion of the residual donor could have been matched or exceeded by the surface decomposition.

Rather than attempting to eliminate the O_2 or H_2O, we next tried to grow GaN on R-plane sapphire from Ga solutions containing only dissolved N_2. In these experiments, sapphire substrates were submerged under Ga in a crucible in which a thermal gradient of 50 - 100°/cm was established. Epitaxial crystals were grown between 950 and 1180°C and at N_2 pressures between 100 and 9,000 atm. The grown crystals were polycrystalline, but their resistivities were very high. The high resistivity was due either to the reduction of the native donor, to insulating grain boundaries, or to the incorporation of compensating acceptor impurities. At this point, our funding ran out and the experiments were discontinued without determining the cause of the high resistance. However, the samples remain available for photoluminescence investigation.

Ge DOPING

From low temperature photoluminescence studies on Ge ion-implanted and subsequently annealed GaN samples, we showed last year that Ge is incorporated both as a shallow donor (0.067 eV) and as a relatively shallow acceptor (0.175 eV). Presumably, the donor is due to Ge on a Ga site, the acceptor to Ge on a N site. From simple thermodynamics, the donor-acceptor ratio should depend upon the donor and acceptor impurities already present in the crystal. Thus, in the presence of the ubiquitous native shallow donor, Ge should be incorporated primarily as an acceptor, thereby compensating the native donor. Hence, we attempted to introduce Ge during normal CVD growth.

1% GeH_4 in H_2 was injected into the growth apparatus via a tube which released it beyond the Ga boats just before the growth zone. Because the GeH_4 cracked at elevated temperatures leaving a deposit of solid Ge in the injection tube, HCl was added to the GeH_4-H_2 flow to transport the Ge as the halide at the elevated temperatures. A second method of introduction was to add some Ge to the Ga boat. In this case, the HCl picks up both Ga and Ge from the boat and transports them to the growth chamber.

Surface nucleation of GaN on sapphire is sensitive to surface contamination, especially on the low free energy surface of basal plane sapphire. Hence, in the latter case, the Ge was introduced only after planar GaN had already begun to grow.

The results of low temperature photoluminescence spectra displaying the Ge donor and the Ge acceptor separately, and of Hall Effect and resistivity measurements, indicated that the Ge was being incorporated primarily as a donor, irrespective of the presence of $\sim 10^{19}/cm^3$ of the native donor. Ge yielded electron concentrations greater than $10^{20}/cm^3$, but the Ge acceptor never compensated the native donor. Hence this line of investigation was abandoned.

C DOPING

From the low temperature photoluminescence studies we performed last year on ion-implanted samples, we deduced from a strong low energy emission peak observed at 3.427 eV that C was neither a shallow donor nor a shallow acceptor. We assumed that the level we observed, a deep level, was due to C on a N site, since the tetrahedral covalent radius of C and its electronegativity were much closer to N than to Ga. Thus C should be a deep acceptor. Since C should be very easy to introduce (as CH_4) during normal CVD growth of GaN, we attempted such experiments in order to compensate the native shallow donor.

Again, as for the case of Ge, the C severely affected the nucleation and the growth morphology of the GaN. Under CH_4 pressures of 6×10^{-4} to 1×10^{-2} atm., (the higher pressures leading to precipitation of C containing particles), the electron concentration went up (to $1 \times 10^{20}/cm^3$), not down, following the C peak as monitored in photoluminescence. Hence our simple deductions were wrong again. C must be a deep donor, not an acceptor. Again, these experiments were abandoned.

ION IMPLANTATION

Last year we described the results of a study of GaN samples ion implanted with much of the Periodic Table in an attempt to decide if the dominant shallow donor was due to a chemical impurity and to discover which elements behaved as shallow donors and shallow acceptors in GaN. This year we tried to complete the investigation. Si, Ge, Sn, S, Se, Te and the native defect are shallow donors (30-80 meV) and Ge, Zn, Cd, Be and Mg are relatively shallow acceptors (150-200 meV). (A number of samples, however, have not yet been analyzed - by photoluminescence.)

In addition, we added two new programs. First hydrogen had been eliminated as the source of the "native" shallow donor on the basis of circumstantial evidence only. Thus we had some proton bombardments done. The results of the photoluminescence studies are described below. Second, since we have already deduced (more or less) that the donor is a native defect, its presence should depend on the presence or absence of excess Ge or N. Hence we had a series of GaN samples separately bombarded with Ga and with N ions. Again the results of these experiments will be described below.

LOW TEMPERATURE PHOTOLUMINESCENCE

Last year we described in detail the results and conclusions from an analysis of low temperature photoluminescence of GaN samples ion implanted or doped during growth with many of the expected shallow donors and acceptors in GaN. The ion implanted samples were annealed at $1100^{o}C$ in an NH_3 atmosphere for one hour to remove implantation damage. The shallow donors and acceptors were identified from their bound exciton emission, deeper levels were described via their participation in donor-acceptor recombination and very deep levels were characterized only by the energies of their emission peaks.

This year we completed a survey of all the elements expected to be shallow donors or acceptors. The luminescence data were all recorded, but the interpretation was halted when our funding ran out in mid-year. Hence we cannot report on these results at this time.

In addition, the luminescence of proton implanted samples was investigated. Here, after the standard annealing procedure, the luminescence spectra revealed no new peaks, but all the peaks in the unimplanted samples, including the dominant native donor peak, were down in intensity. The obvious conclusion is that hydrogen produces a recombination center (either non-radiative or at very low energies) that competes very successfully with all the radiative transitions. Thus, proton implantation does not preferentially enhance the native shallow donor peak. Therefore, the native shallow donor peak cannot be associated with hydrogen. However, the annealing to remove implantation damage must be done in an NH_3 ambient to avoid surface decomposition. The hydrogen containing NH_3 could restore hydrogen equilibrium with the GaN, thereby weakening the above conclusions. Thus, we can deduce that hydrogen is unlikely to be associated with the native donor, although further annealing experiments (does the native donor peak recover at the same rate as the less

intense impurity peaks - mostly Si, with annealing) are in order.

Next, we have a series of samples implanted separately with various fluences of Ga and with N ions. These have not yet been annealed nor characterized by photoluminescence. We expect that annealing-luminescence experiments may show whether the native donor is indeed influenced by the presence of excess Ga or N. Again, these experiments were halted in mid-year when our funding was dissipated.

Finally we note first, that there are almost invariably a series of narrow (non-instrumental) emission lines superimposed on almost all the low temperature luminescence near the band gap, and second, that the shapes of the bound exciton peaks are highly non-Gaussian. We have no understanding of the former phenomenon, but we believe that the latter effects are associated with the high electron density in these crystals, i.e., that the sharp tops of the bound exciton peaks result from exciton recombination in isolated, carrier-free regions of the crystal, whereas the remainder of the broad bands is due to recombination in the presence of a degenerate (screening) electron gas. The peak shapes should then provide us with evidence for the shape of the electron distribution.

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