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Final Report

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“Non-equilibrium Approach to Doping of Wide Bandgap materials by Molecular Beam Epitaxy”

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1. INTRODUCTION

It is well known that it has been difficult to obtain good bipolar doping in wide bandgap semiconductors (see e.g. Neumark 1997a, 1997b). We have recently developed a new doping technique, involving use of a standard dopant, together with a “co-dopant” used to facilitate the introduction of the dopant, and have vastly alleviated this problem (Lin et al., 2000; Guo et al., 2001). Specifically, we have applied this to ZnSe, using N as dopant (for p-type doping) and Te as co-dopant; these were introduced in sub-monolayer amounts in δ -layers (specifically three such layers for good doping). We note that ZnSe, and related alloys, have been extensively studied. For these materials, p-doping has been a problem (Neumark et al., 1994 and Neumark, 1997a) and our technique has given about an order-of-magnitude improvement over bulk doping (Lin et al., 2001) or delta-doping without a “co-dopant”. Moreover, measurements of the activation energy of luminescence quenching gave energies as low as 40 meV, i.e. close to the activation energy of N in ZnTe (Kuskovsky et al. 2002a), rather than that of 110 meV of N in ZnSe. Moreover, SIMS results show that Te and N tend to stay within the delta-layers, and thus, it appears self-evident that association between N and Te must be involved. Furthermore, TEM studies have shown that there is a periodic structure associated with the Te layers, but that a complete superlattice (SL) is not found, confirming the presence of nano-clusters (Kuskovsky et al., 2003a). This work is summarized in the following sections.

1.1. Growth And Doping

The method we selected to explore the enhancement of p-type doping in ZnSe by non-equilibrium concepts was delta doping (δ -doping, whereby the dopant is incorporated in planes, during growth interruptions, separated by undoped spacer regions). Prior work on δ -doping of ZnSe by Nitrogen (e.g. Zhu et al., 1995) had shown that only modest enhancements in doping could be achieved by this technique (Zhu et al., 1995). However, Jung et al. (1997) reported that in short period superlattices (SL) of ZnSe (9 monolayers) and ZnTe (1 monolayer) a dramatic enhancement of p-type doping over that possible in ZnSe can be achieved. Nevertheless, the work of Jung et al. (1997) has the disadvantage that the relatively large ZnTe content of their structure produces a large lattice mismatch with the desired substrate (GaAs). This is undesirable for practical applications of the SL material in place of ZnSe in device structures. We therefore decided to try this (N + Te) approach with both deposited simultaneously in the dopant plane, but with sub-monolayer amounts, rather than the full monolayer of Jung et al. (1997).

Our initial efforts were aimed at determining the growth sequence that would produce the desired structure and provide good dopant activation. After some initial experimentation, we showed that the MBE shutter sequence given by Lin et al. (2000) yielded an improvement over using N alone. We explored the manipulation of the N acceptor environment further by carrying out a modified delta doping sequence, in which three consecutive layers (sub-monolayers) of N and Te were deposited within one δ -region (so-called "triple delta doping"). The triple δ -doped regions were separated from each other by undoped spacers, as in the previous case. The purpose for this modification was to surround the N acceptors within a ZnTe-like environment (Lin et al., 2000 and Sect. 1.2).

The average Te content of our samples was estimated from X-ray diffraction (XRD), which yielded values of less than 2% ZnTe (e.g., Lin et al., 2000) and, in some cases, as low as 0.5% (e.g., Guo et al., 2001) as well as from secondary-ion mass spectroscopy (SIMS) yielding Te concentration of $\sim 1\%$ (Kuskovsky et al., 2003). Since the Te level was very low, this was accomplished with only minimal variation in the lattice-constant and thus no strain related defects (Kuskovsky et al., 2003). We also performed preliminary investigation of the effects of (N + Te) delta-doping in ZnCdSe and ZnMgSe layers (Lin et al., 2000) and ZnBeSe (Guo et al., 2001). Similar enhancements were observed in these materials, although the compensation problems were more severe, and the ultimate doping levels achieved were lower.

1.2. Electrical Properties

The net acceptor concentration ($N_A - N_D$) was measured via electrochemical capacitance-voltage (ECV) profiling. The results (Lin et al., 2000) are summarized in Table 1.1. Several points are worth noting. First and foremost, the net acceptor concentration for the δ^3 -case with both N and Te is in the mid $10^{18}/\text{cm}^3$ range, which is adequate for reasonably good devices; moreover, this is an order of magnitude higher than uniform N-doping and, for that matter, than δ -doping with N alone. We note that this conductivity is obtained with excellent lattice matching (as already mentioned in Sect. 1.1). Such matching is important not only for low defect densities but also for quantum well and confinement structures. For comparison, N-doped ZnSe/ZnTe superlattice structures, which give good contact resistivities ($2\text{--}8 \times 10^{-3} \text{ ohm cm}^2$) and which have been suggested for improved contacts to ZnSe-based devices (Fan et al., 1992), have the problem of lattice mismatch between ZnSe and ZnTe.

Table 1.1. Comparison of net acceptor concentrations in p-type ZnSe obtained using different doping techniques

Doping	Number of undoped monolayers (ML)	$N_A - N_D$ (cm ⁻³)
N bulk		3.0×10^{17}
N δ -doping	5	6.0×10^{17}
(N+Te) δ -doping	4	1.5×10^{18}
(N+Te) δ^3 -doping	7	6.0×10^{18}
(N+Te) δ^3 -doping	12	5.0×10^{18}

1.3. Photoluminescence Characterization

Photoluminescence (PL), together with its dependence on excitation intensity, temperature, pressure, etc., is a very powerful technique for characterizing the microscopic nature of centers in materials. We have used PL to characterize the above δ -doped samples (Kuskovsky et al., 2001b). In addition, we have also used it to characterize samples of ZnSe δ -doped with Te only (i.e. without N) as control samples (Kuskovsky et al., 2001a, Gu et al., 2003).

We first discuss some of the results of the PL of Te only (no N) doped samples, since this provides a “baseline” for understanding the properties of co-doped (Te+N) samples as well as additional points of interest when considered on their own. We show the PL spectra at various excitation intensities for a δ^3 -doped sample in Fig. 1 (see also Kuskovsky et al., 2001a). The

Figure 1. PL from a δ^3 -doped ZnSe:Te sample as a function of excitation intensity; upper inset: integrated PL intensity vs excitation intensity, showing a single slope, indication one type of transition; lower inset: peak position for the “green band”, showing Type II behavior for the excitonic emission

Figure 2. Temperature dependent properties of the nano-structure related PL.

spectra show two bands at $\sim 2.65\text{eV}$ ("blue band") and 2.49eV ("green band"). These had been, at the time, attributed by Kuskovsky et al. (2001a) to isoelectronic excitons bound at centers consisting of several Te atoms. We note that one also observed a decrease of the full-width-at-half-maximum (FWHM) of the green band with temperature (Fig. 2), which was attributed to the presence of the Te_n centers with different ionization energies. However, the precise origin of such centers has not been understood.

Very recent studies by our group (Gu et al., 2003 and Fig. 2) have proven unequivocally that, instead, the green band originates from "centers" that exhibit quantum confinement effects and thus should be classified as nano-particles (QDs, nano-island, nano-clusters, etc.).

Indeed, we have shown by additional analysis of cw PL (see the lower inset in Fig. 1) together with the temperature dependent life-time measurements that the green band PL exhibit properties which are fingerprints of quantum disk like centers (Fig. 2). First, we note that the lifetime of the green band PL increased by a factor of 5 compared with bulk ZnSeTe (for bulk data see e.g., Akimova et al., 1986). Such an increase is a "fingerprint" for low dimensional systems (Citrin, 1992; Akiyama et al., 1994; Sugawara, 1995). Evidence for low dimensionality is also given by the fact that the PL lifetime increases with T (Citrin, 1993; Gotoh et al., 1996; Okuno et al., 1999) as shown in Fig. 2-(a). Furthermore, as already mentioned, the FWHM decreases with T (Fig. 2.2-(b)) within the same temperature region where the peak position exhibits a red shift relative to the bandgap (Fig. 2-(c)); this means that one is dealing with an ensemble of such low dimensional structures (Brusaferri et al., 1996; Sanguinetti et al., 1999; Karczewski et al., 1999). It is more important that the careful analysis of the green band peak position at various excitation intensities showed that it increases linearly with the cubic root of excitation intensity (the lower inset in Fig. 1). Such a behavior has been predicted and indeed observed for Type II quantum structures (see e.g., Ledentsov et al., 1995 and Glaser, et al., 1996). We note that the ZnTe/ZnSe system is indeed characterized by Type II band offset.

We next discuss the "doped" system, i.e. the one with both Te and N. The PL spectra are usually shifted to the red as compared to previously discussed system. Specifically, the PL as a function of excitation intensity is shown in Fig.3-(a) for a triple δ -doped sample, with Fig. 3-(b) showing the results for a single δ -doped sample for comparison. A very striking result for the triple-doped

Figure 3. (a) The low temperature PL as a function of excitation intensity for a δ^3 -ZnSe(Te,N) sample; (b) the same for a δ -ZnSe(Te,N) sample.

case is the very large peak shift, towards the blue, with increasing excitation intensity. Moreover, this shift is larger for higher Te content (Table 1.2) and, of particular high interest, the shifts for the single δ -doped sample are far smaller. Such shifts with intensity are a hallmark of donor-acceptor pair (DAP) PL. However, the present shifts for the triple case are far larger than obtained by DAP theory considering only random recombination by pairs at different distances (see e.g. Neumark, et al., 1985 for a description of this theory). Large shifts can also be obtained for preferential donor-acceptor pairing (see e.g. Neumark, et al. 1994), but if this were the cause of the present large shifts, one would expect comparable results for the single δ -doped case. However, the ZnSe/ZnTe heterostructure is of Type II (e.g., Link et al, 1994; Malonga et al., 1995; Wei and Zunger, 1996), which would provide spatial separation between the electrons (in ZnSe) and the holes (in ZnTe), and will lead to, additionally to DAP, a blue shifts with increasing excitation intensity (for a discussion of spatial separation effect see, e.g. Ledentsov et al. (1995) and Glaser et al. (1996)). It is also of high interest that the triple delta-doped structure is thick enough to give heterostructure behavior, whereas such behavior is not obvious for the single delta-doped case, where results are consistent with "standard" DAP theory.

Table 1.2. Sample Parameters and Photoluminescence Properties

Te Concentration	N_A-N_D (cm ⁻³)	PL Quenching Activation Energy (meV)	Peak Shift* (meV)
<3%	6.0×10^{18}	38	60
1.3%	4.0×10^{18}	72	42
<1.0%	3.0×10^{18}	87	35

*The excitation intensity was changed by two orders of magnitude.

Furthermore, the PL in the δ^3 -doped sample quenches with an activation energy between 38 and 87 meV (depending on the sample) (Table 2.2. and Kuskovsky et al., 2003a). This corresponds to the activation energy of the N acceptor in ZnTe (Duddles et al., 1994, Fan et al., 1994, Jung et. al., 1997), rather than the N activation energy in ZnSe of 111 meV (Dean et al., 1983). Thus enough N must be located in ZnTe rich regions to lead to values close to the ZnTe activation energy.

1.4. Micro-Structural Characterization

A. TEM

We have also performed preliminary studies on the microstructure of δ^3 -ZnSe:(Te,N) using transmission electron microscopy (TEM) in collaboration with Dr. Zhu's group at Brookhaven National Laboratory (Kuskovsky et al., 2003a). Techniques such as bright/dark field imaging, high resolution electron microscopy (HREM), selective area electron diffraction (SAED), and Energy Dispersive X-ray Spectroscopy (EDS, see above) were used. The initial work was on cross-section TEM samples (with enhanced Te concentrations in order to increase the contrast), which were viewed along the [100] direction. Results are shown in Figs. 4-(a) and (b) with the corresponding SAED pattern shown in Fig. 2.3-(c).

The direct TEM and HREM observations (Figs. 4-(a) and (b)) show the presence of a modulated structure with an optical period of 1.5nm – 1.7nm along the growth direction ([001]), while this is absent in the ZnSe buffer layer as well as the substrate. Such a period is in excellent agreement with the nominal thickness (~1.4-1.7nm) of the spacer layers in the investigated sample. Moreover, and it is significant, that the TEM images consistently show a discontinuity of the modulated structure (Fig. 4-(a)) in a basal plane and thus this modulated structure is not a perfect

superlattice. Thus one can conclude that nano-clusters are formed in (001); we note that the optical size of such clusters is less than 15nm. It is also very important to point out that, on the whole, the periodic atomic structure of a δ^3 -ZnSe:(Te,N) system is largely preserved and does not create misfit dislocations, as seen from the HREM image (Fig. 4-(b)).

Further information as well as confirmation of the above observations is given by SAED. Indeed, in the SAED, additional satellite spots (Fig. 4-(c) and the inset) are present along with the primary

Figure 4. Microstructure of a δ^3 -ZnSe:(Te,N) sample with 10-12ML thick spacers and 20s (Te+N) deposition time: (a) a TEM image taken at medium magnification along the [100] zone axis, showing modulated structure in the film; (b) a high resolution image shown at grazing angle to enhance modulation contrast, revealing the absence of misfit dislocations; (c) a composite SAED pattern corresponding to Fig.4-(a), revealing elongated satellite reflections (enlarged in the inset) associated with this modulation.

ZnSe reflections. These spots exhibit elongated shapes within the (001) plane. The presence of such spots confirms the existence of a modulated structure, while the observed elongation means that no full SL is formed. In fact, if a perfect SL existed, then such satellites would *form a superlattice reflections in the diffraction pattern, with "perfect dots" instead*. Using the ZnSe {002} and {020} reflections as a reference, we have determined that the period of the modulated structure is 1.6 ± 0.1 nm in excellent agreement with direct observations.

Figure 5. SIMS Profile of a δ^3 -ZnSe:(Te,N) sample with 50s (Te+N) deposition (see Kuskovsky et al., 2003 for details). The correlation between N and Te is clearly seen. The observed period in Te and N concentrations corresponds to nominal thickness of the spacer layers in this sample.

B. SIMS

An important question is the location of N in reference to Te. In Sect. 1.3 we have shown that the PL quenching activation energies suggest that considerable N must be within Te rich nano-clusters. We further performed preliminary SIMS studies that have shown (Fig. 5 and Kuskovksy et al., 2003a) that Te and N atoms tending to be restricted to delta-layers, supporting our conclusions about cluster formation with N preferentially embedded in these.

1.5. Island Formation

Since the Te is deposited randomly on the ZnSe surface, ZnTe island formation is presumed to take place by some form of self-assembly process, possibly driven by the lattice-mismatch strain. Self-assembly of a strained layer into nano-scale clusters or islands by the Stranski-Krastanov mechanism (Madhukar, 1996) (for example, in the formation of self-assembled quantum dots) has been observed in a number of systems (e.g., InAs/GaAs (Ledentsov, 1996) and CdSe/ZnSe (Xin et al., 1996)). In those cases, several monolayers of highly strained material are deposited on the "substrate". This leads to the build-up of a significant strain energy, which in turn drives the surface migration processes required to form clusters whose shape minimizes the strain energy. In our case, however, while we anticipate that a similar process may be occurring here, some other mechanisms (e.g. surface enhanced diffusion) must be involved since we are depositing only sub-monolayer amounts. The large delay times built into our delta-doping growth sequence (which were designed to allow desorption of excess species from the surface) may be providing the necessary times for surface migration to take place. This point should be investigated further.

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