

Photon Assisted Growth of Nitrogen-Doped CdTe and the Effects of Hydrogen Incorporation During Growth

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Nitrogen doping in CdTe epilayers grown by photo-assisted molecular beam epitaxy was demonstrated using an rf plasma source. The effect of the presence of atomic hydrogen during growth of undoped and nitrogen-doped CdTe was investigated. The layers were characterized using photoluminescence spectroscopy (PL), Hall effect, secondary ion mass spectroscopy (SIMS), Fourier transform infrared spectroscopy, and atomic force microscopy. PL confirmed the incorporation of nitrogen as acceptors. While p-type carrier concentrations greater than 10^{18} cm^{-3} were easily obtained, SIMS measurements indicated that nitrogen was concentrated near the undoped-doped and epilayer-substrate interfaces which complicates interpretation of activation efficiency. Hydrogen incorporation was found to be enhanced by the presence of nitrogen. Infrared absorption measurements strongly suggested the formation of N-H complexes. Hall measurements indicated that complexes are formed which are donor-like in nature. The presence of atomic hydrogen during growth radically changed the low temperature photoluminescence in both undoped and nitrogen-doped layers. Exciton-related luminescence was quenched at low temperature. Nitrogen-related donor-acceptor pair luminescence was also absent from the N-doped hydrogenated layers, consistent with complex formation. Copper (a cation-site acceptor) donor-acceptor pair luminescence appeared to be enhanced by hydrogenation.

Key words: CdTe, hydrogen incorporation, molecular beam epitaxy, nitrogen doping

INTRODUCTION

There are a limited number of reports of p-type CdTe grown by molecular beam epitaxy (MBE).¹⁻¹¹ In addition, technological difficulties remain for most of the dopants which successfully produce high p-type carrier concentrations. For example, Cu, Li, and Ag have sufficiently high diffusion coefficients in CdTe at typical growth temperatures to preclude practical device structures. The use of illumination during growth, known as photon-assisted MBE, has allowed p-type doping with Sb^{2,3} and As⁴⁻⁷ with the highest carrier concentrations being $2.8 \times 10^{18} \text{ cm}^{-3}$ and $6 \times 10^{18} \text{ cm}^{-3}$, respectively.^{3,4} However, difficulties in reproducibility are encountered with both Sb and As.¹²

Doping using an rf plasma N source has been extensively studied for the Group II-selenides.¹³ An issue of concern is the apparently intrinsic self-compensation of acceptors in the Group II-selenides occurring with nitrogen doping, limiting p-type carrier concentrations to about $2 \times 10^{17} \text{ cm}^{-3}$. Similar to ZnSe, recent reports^{9,10} of nitrogen doping in CdTe using plasma sources have reported maximum p-type carrier concentration levels of only about $2 \times 10^{17} \text{ cm}^{-3}$. The study by Baron et al.¹⁰ indicated that even the addition of a small amount of Zn (forming a CdZnTe alloy) dramatically increased the maximum p-type carrier concentration, with a p-type carrier concentration of 10^{20} cm^{-3} for ZnTe. This led to speculation about a self-compensation mechanism associated with the presence of Cd. Also, both groups used a Cd overpressure to obtain p-type conductivity.

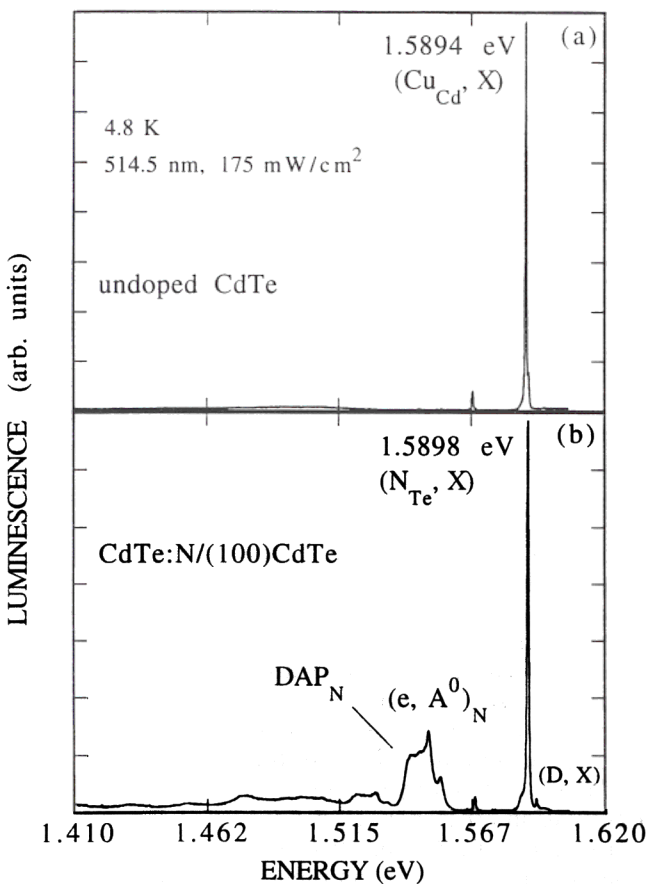


Fig. 1. Photoluminescence spectra of (a) undoped CdTe and (b) nitrogen-doped CdTe grown by MBE on (100) CdTe substrates.

Photon-assisted MBE has been shown to produce a reflection high energy electron diffraction (RHEED) surface reconstruction consistent with a Cd-rich growth surface while using a single source of CdTe.^{14,15} It has been speculated that illumination increases the number of vacant Te sites at the growing surface and directly leads to an enhanced incorporation of substitutional acceptors on this site.³ Thus, we decided to investigate photon-assisted MBE for nitrogen doping during CdTe growth using a single source of CdTe.

The introduction of atomic hydrogen into semiconductors can significantly affect the optical and electrical properties of the materials.¹⁶ The ubiquitous nature of hydrogen during the growth and processing of semiconductors requires an understanding of its effects on dopant compensation and point defect formation. Reports on the effects of hydrogenation remain scarce for II-VI compounds. Hydrogenation studies of bulk CdZnTe through exposure to a hydrogen plasma indicate that hydrogen quenches donor-acceptor pair (DAP) and electron-acceptor (e,A) photoluminescence tentatively assigned to Cd vacancies.^{17,18} Similar quenching of DAP luminescence was observed for ZnSe¹⁹ for an unspecified donor and acceptor. Annealing of nitrogen-implanted CdTe in a hydrogen atmosphere reduced DAP luminescence attributed to nitrogen.²⁰ Hydrogen forms complexes with N in ZnSe grown by metalorganic

chemical vapor deposition (MOCVD) and metalorganic MBE (MOMBE).^{21–23} Infrared absorption studies performed using a Fourier transform infrared spectrometer (FTIR) have also indicated the formation of complexes between H and As in CdTe and ZnTe, and between H and P in ZnTe grown by MOCVD.²⁴ In anticipation of investigating the use of H in heteroepitaxial growth of CdTe, we undertook a study of the influence of hydrogen on the properties of undoped and nitrogen-doped CdTe homoepitaxial layers grown by MBE.

GENERAL EXPERIMENTAL DETAILS

The CdTe epilayers were grown by MBE at West Virginia University on (100) and (211)B-oriented CdTe substrates using a single source of CdTe, both supplied by II-VI, Inc. (Saxonburg, PA). A standard set of growth conditions was chosen based on the prior experience of one of us (THM). These conditions were a laser illumination of 75 mW/cm² (Argon 514 nm), a substrate temperature 230°C, a growth rate 0.3 μm/h, and a doped layer thickness of 2.0 μm on an undoped 0.3 μm buffer layer. The standard operating conditions for the cryogenically cooled nitrogen source (Oxford Applied Research CARS-25) were an rf power of 275 W and a beam-equivalent-pressure (BEP) of 3 × 10⁻⁶ Torr at the substrate. The nitrogen gas was semiconductor-grade (ULSI 0.999999% purity) bought from Matheson Gas Products. Hydrogenation was accomplished using 1 × 10⁻⁶ Torr BEP of hydrogen passed through the thermal source operating at 9.5 amperes. Literature supplied with the source indicated a dissociation efficiency of about 5% for this operating condition. A sequence of 11 CdTe:N samples were grown on (100) CdTe substrates from the same boule with a systematic variation in parameters from the standard set. A total of four layers, which included one (211)B substrate, were grown under a hydrogen flux using the standard conditions.

Extensive PL studies at liquid-helium temperature (4.8K) were performed on the samples. The photoluminescence (PL) signals were excited using either the 514.5 nm output from an Innova 400–10 argon-ion laser from Coherent, Inc. or the tunable infrared output from a Ti:sapphire laser pumped by the argon ion laser. Power densities employed were in the range from 0.175–3.1 W/cm². The luminescence was recorded with an Instruments SA, Inc. HR-640 spectrometer and either a photomultiplier tube (PMT) with GaAs cathode or a germanium detector cooled to 77K. All data reported in this paper have been corrected for system response using a calibrated white-light source.

X-ray diffraction rocking curve measurements indicated no measurable difference in crystal quality between the epilayers and the original substrates. The actual x-ray rocking curve full widths at half maximum were limited in all cases by the presence of subgrain boundaries in the substrates. The CdTe layers were also characterized by low temperature FTIR absorption measurements, secondary ion mass

spectroscopy (SIMS), and room-temperature Hall effect measurements using a standard Van der Pauw configuration. Electroless gold (from a $\text{AuCl}/\text{H}_2\text{O}$ solution) was used to obtain ohmic contacts to the p-type layers.

RESULTS AND DISCUSSION: NITROGEN DOPING

Several undoped CdTe epilayers were also grown during the period of this investigation. A PL spectrum characteristic of these samples is shown in Fig. 1a. The spectrum is clean, with only an acceptor bound exciton ($A^{\circ}X$) transition indicative of Cu_{Cd} as well as phonon replicas. A spectrum obtained from a bare substrate was essentially identical, indicating that the Cu originated in the substrate. The presence of fast-diffusing impurities is an ongoing issue of concern in commercial CdTe substrates.^{25,26} Of particular interest is that there is no evidence for the presence of donor impurities in this sample as indicated by the absence of donor bound exciton ($D^{\circ}X$) or donor-acceptor pair luminescence.

Hall measurements indicated a p-type carrier concentration of about $5 \times 10^{16} \text{ cm}^{-3}$ and a mobility of about $90 \text{ cm}^2/\text{V}\cdot\text{s}$ in the undoped layers. SIMS analysis indicated the presence of Cu at this level, apparently diffusing from the substrate. No other impurities were detectable by SIMS above background levels in these layers away from the epilayer-substrate interface indicating the residual p-type conductivity is apparently due to the presence of Cu. Similar results were observed for all undoped layer growths.

Layers grown under a nitrogen flux on substrates from the same boule exhibited additional PL features, as shown in Fig. 1b. The ($A^{\circ}X$) transitions at 1.5898 eV and (e^-, A) transitions at 1.5503 eV are indicative of nitrogen incorporation on the Te site, and our measured transition energies are in agreement with previous studies.⁸⁻¹⁰ There is also a ($D^{\circ}X$) transition at 1.5932 eV and significant DAP luminescence at 1.543 eV, indicating the presence of donors. Since donor-related PL was only observed for nitrogen-doped layers, the donor is somehow related to nitrogen incorporation. The relative intensity and narrowness of the donor-related PL indicates a small concentration of donors. The energy of the DAP luminescence is consistent with a shallow donor with an ionization energy between 13–15 meV.

In order to identify the donor species present in the nitrogen-doped layers, a series of selective-excitation PL measurements were performed. By tuning into the ($D^{\circ}X$) transition, a resonant enhancement of the two-electron transition (TET) is produced. The energy difference between the ($D^{\circ}X$) and the corresponding TET line gives the $1s \rightarrow 2s$ splitting which is sensitive to the particular donor species. Figure 2 shows the ($D^{\circ}X$) and TET region for a CdTe:N layer. The principal donor-bound exciton line is labeled D_1° and the excited states are labeled D_1^2 and D_3^1 , following the notation given in Ref. 27. The TET transitions are labeled D_2° and D_3° . The $1s \rightarrow 2s$ splitting between

D_1° and D_2° was 10.24 meV.

Two-electron transition energies and ionization energies for several donor species in CdTe have been reported.²⁷ Based on these reported values, we can identify the donor in our layers as substitutional fluorine (F_{Te}). The ionization energy of fluorine was 13.7 meV and the $1s \rightarrow 2s$ splitting was reported to be 10.28 meV. Our identification of F_{Te} as the shallow donor in the CdTe:N layers is inconsistent with the donor being related to defect complex formation with nitrogen, as has been suggested.^{9,10} Subsequent SIMS measurements indicated the presence of F at levels of about $1 \times 10^{15} \text{ cm}^{-3}$ in nitrogen doped layers. The source of F during growth is unclear, but it appears to only occur for nitrogen doping.

Results from Hall effect measurements on the nitrogen-doped layers appeared promising for attainment of high doping levels using nitrogen, with clear trends emerging from varying growth conditions. For example, as the substrate temperature was stepped from 200 to 230 to 260°C, the room-temperature p-type carrier concentration increased from 1 to 4 to $15 \times 10^{17} \text{ cm}^{-3}$ with mobilities of 53, 65, and $92 \text{ cm}^2/(\text{V}\cdot\text{s})^{-1}$, respectively. This trend is consistent with increased nitrogen incorporation with a Cd overpressure as increasing substrate temperature has been shown to lead to a more metal-rich growth surface.¹⁵ Note that the p-type carrier concentration for the layer grown at 260°C is about an order of magnitude larger than previously reported^{9,10} for nitrogen-doped CdTe.

The effect of varying the illumination intensity during growth at 230°C was also investigated. Of immediate interest is that we obtained a p-type car-

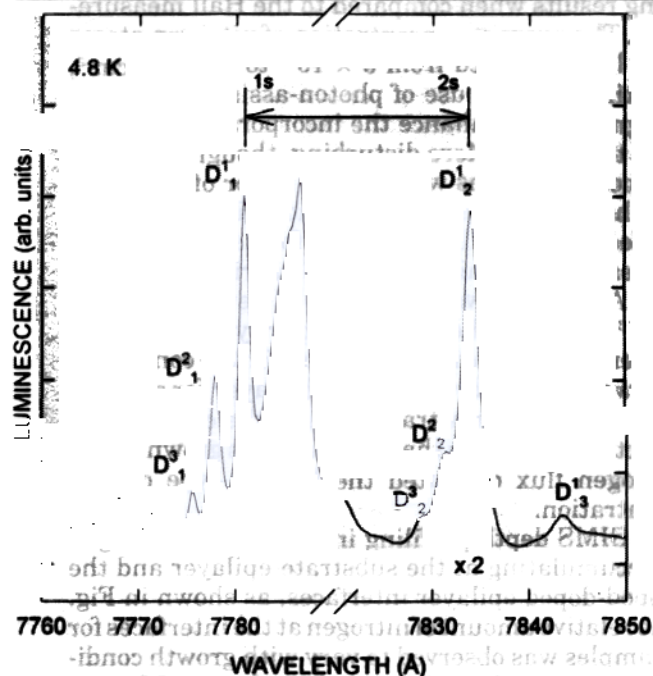


Fig. 2. Selective excitation photoluminescence of donor-related transitions in nitrogen-doped CdTe producing an enhanced two-electron transition.

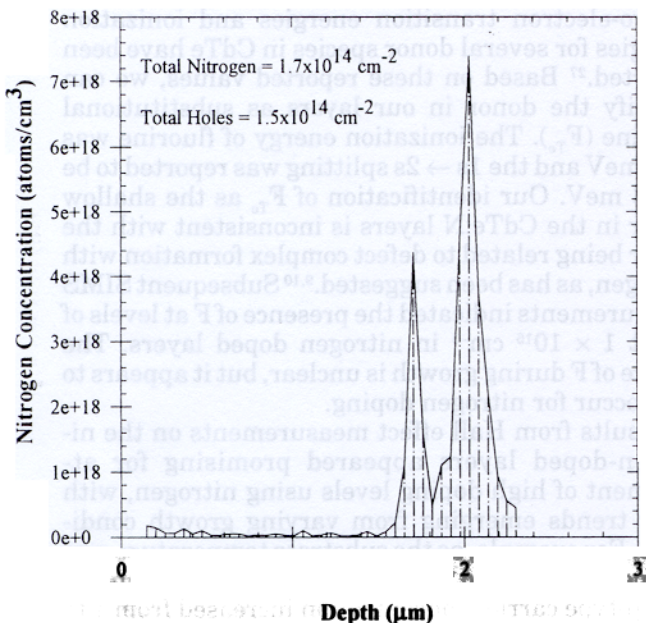


Fig. 3. SIMS analysis of a CdTe:N layer illustrating the high nitrogen concentrations observed at sample interfaces.

rier concentration of $1.3 \times 10^{18} \text{ cm}^{-3}$ for growth without illumination and without a Cd overpressure. Indeed, illumination appeared to lower the p-type carrier concentration. The lowest p-type carrier concentration observed for the nitrogen-doped layers, $9 \times 10^{16} \text{ cm}^{-3}$, was obtained for a sample illuminated at 200 mW/cm^2 . This is lower by a factor of 14 from the non-illuminated case.

SIMS analysis of the CdTe:N layers indicated surprising results when compared to the Hall measurements. The average concentration of nitrogen atoms in the layers ranged from 5×10^{16} to $2 \times 10^{17} \text{ cm}^{-3}$, indicating that the use of photon-assisted MBE did not significantly enhance the incorporation of nitrogen in the layers. More disturbing, though, was that these concentrations were over a factor of ten lower than the Hall carrier concentrations in most cases. Detailed SIMS analysis was performed on the two samples with the highest p-type carrier concentrations. Again, Cu was observed at levels of about $5 \times 10^{16} \text{ cm}^{-3}$. No other p-type impurity was found at sufficient concentrations to account for the conductivity. It was verified by removing the layer for selected samples that the substrate was not the cause of the conduction. Also, note that only samples grown under a nitrogen flux exhibited the large p-type carrier concentration.

The SIMS depth profiling indicated that nitrogen was accumulating at the substrate-epilayer and the undoped-doped epilayer interfaces, as shown in Fig. 3. The relative amount of nitrogen at the interfaces for the samples was observed to vary with growth conditions. For example, a decrease by a factor of four in nitrogen flux led to a similar decrease in nitrogen incorporated at the interfaces. The observed nitrogen atom concentrations at the interfaces were orders of magnitude larger than for any other impurity, includ-

ing Cu. The total number of nitrogen atoms at the interfaces was sufficient to account for the total number of holes indicated by the Hall measurements. The layer shown in Fig. 3 had a total of 1.7×10^{14} nitrogen atoms/cm² compared to a total of 1.5×10^{14} holes/cm². For comparison, the layer had a total of 1.6×10^{13} copper atoms/cm². Thus, we speculate that our observed high p-type carrier concentrations are due to nitrogen "δ-doping" at the interfaces which implies a high activation of N in heavily doped regions.

Our results share similarities to the previous study by Baron et al.¹⁰ While Baron et al. only observed high activation for effective carrier concentrations up to $2 \times 10^{17} \text{ cm}^{-3}$, the actual doping structures used in the study imply high activation of heavily doped regions. The source used by Baron et al. could only be run at a single high nitrogen flux value and variation in doping was accomplished by performing what they termed "pulse-doping". That is, they created heavily doped regions with atomic nitrogen concentrations of about $9 \times 10^{18} \text{ atoms-cm}^{-3}$. Thus, the layers with high levels of activation actually consisted of thin, heavily doped regions separated by large spacers of undoped material. This is similar to what we observe with our heavy doping only occurring at interfaces.

While neither this study nor the study by Baron et al.¹⁰ conclusively show that high activation can be obtained in heavily nitrogen-doped CdTe, the results are certainly suggestive. It is equally unclear that an intrinsic compensation mechanism exists for nitrogen doping in CdTe. The results are sufficiently encouraging that additional investigations should be pursued in the nitrogen doping of CdTe. In particular, δ-doping techniques should be investigated. Our study also indicates that, unlike the case for Sb and As, photon-assisted MBE does not significantly enhance the incorporation of nitrogen in CdTe.

RESULTS AND DISCUSSION: HYDROGENATION

The presence of atomic hydrogen during growth has many interesting effects on the optical and electrical properties of CdTe. An initial report of many of these effects has been published elsewhere.²⁸ In this workshop paper, we summarize this initial study and present additional results.

The surface morphology of the (100)-oriented CdTe:H layers was different than undoped CdTe or the CdTe:N layers. In particular, the layers grown without hydrogen were fairly smooth while the hydrogenated layers contained numerous features reminiscent of the "pyramidal hillocks" indicative of twin-related defects.²⁹ An atomic force microscope (AFM) micrograph of one such feature is shown in Fig. 4a. Personal experience of one of us (THM) has shown that such morphological features could be suppressed by use of either illumination or a Cd overpressure during growth of CdTe. While such layers had an improved morphology, subsequent defect etching revealed that the defects were still present at comparable densities. The presence of the hydrogen flux

appears to alter the growth kinetics to promote the growth of these features undoubtedly present in the other layers.

In contrast, the surface of the (211)B layers grown under a hydrogen flux were smooth with an absence of large morphological features. A typical AFM micrograph is shown in Fig. 4b. There is a large period background "waviness" we believe to be related to etching effects in substrate preparation. This feature has an rms. variation of about 5 nm. On a finer scale, the rms. surface roughness was less than 1 nm.

Hall measurements made on undoped CdTe layers grown under a H flux were indistinguishable from that obtained for undoped layers. Instrumental background levels for hydrogen were too large to allow quantitative determination of hydrogen concentrations in these samples using SIMS. However, a dramatic difference was observed in the PL spectra.

The 4.8K PL spectrum obtained from a CdTe:N layer grown under a H flux (CdTe:N,H) is shown in Fig. 5a. Similar spectra were obtained from CdTe:H layers grown without the presence of nitrogen. The bound-exciton, (e,A^0), and DAP emissions related to the shallow nitrogen acceptor are greatly reduced in

intensity, which is similar to effects reported by Boudoukha et al.²⁰ for nitrogen-implanted CdTe annealed in a hydrogen atmosphere. In contrast, there is a significant enhancement of the DAP emission related to Cu_{Cd} and a shallow donor, seen in the range of 1.3 to 1.45 eV with the zero-phonon band occurring at about 1.45 eV. The other peaks observed on this broad band are phonon replicas with energy spacing of about 20 to 21 meV. SIMS analysis did not indicate copper segregation in the epilayers, as is sometimes observed.^{25,26} The enhanced Cu-related DAP may indicate that hydrogenation results in an increase in the background concentration of shallow donors. If so, the concentration was too low to be observed in Hall measurements above the p-type background due to copper.

Selective excitation PL studies were performed on the CdTe:H and CdTe:N,H layers. The output from a Ti:sapphire laser was used to tune the exciting radiation above and below the CdTe bandgap. Using above-bandgap excitation, the dominant emission was copper-related DAP as discussed above. The curve shown in Fig. 5b was obtained using excitation at 1.5867 eV

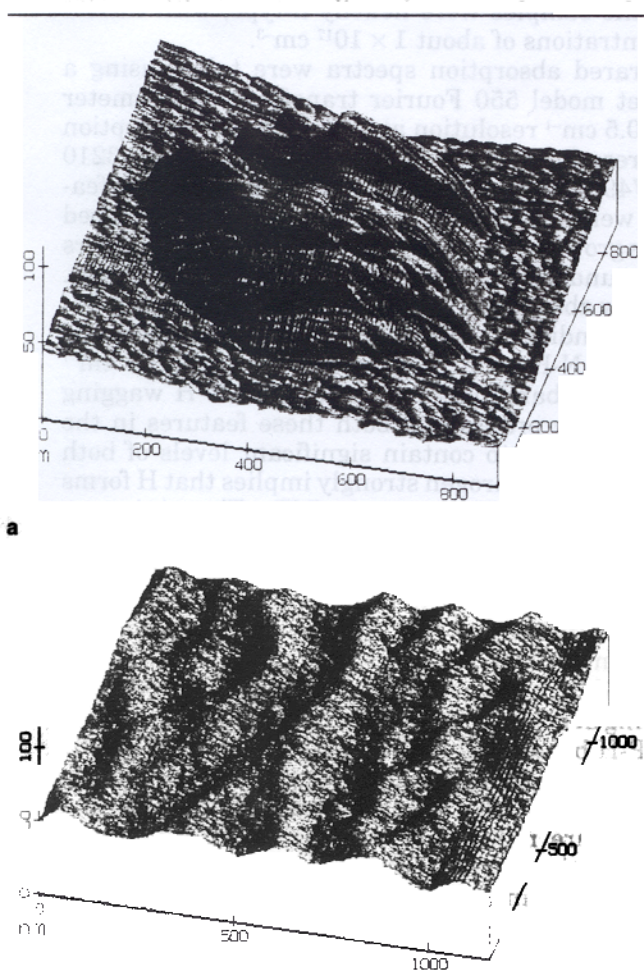


Fig. 4. Atomic force microscopy micrographs of CdTe layers grown under a hydrogen flux: (a) (100)-oriented; (b) (211)B-oriented.

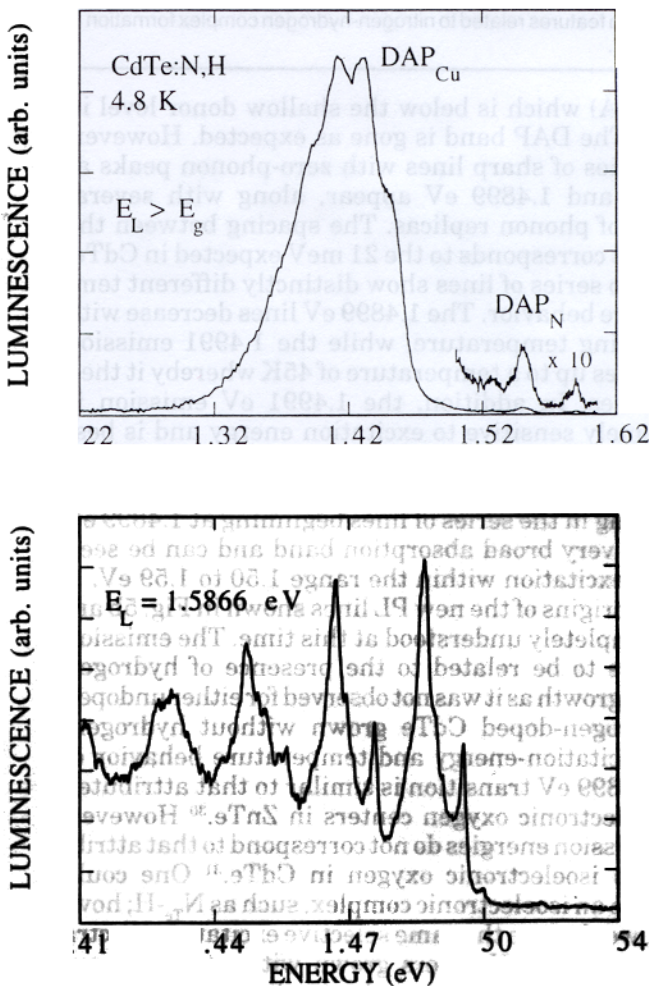


Fig. 5. Selective excitation photoluminescence of CdTe:N,H with (a) above bandgap excitation, and (b) excitation tuned below the shallow donor energy level.

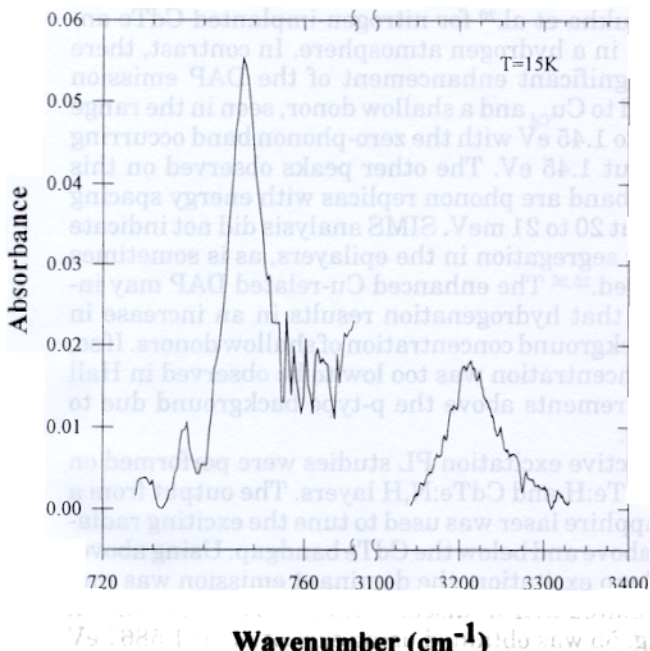


Fig. 6. Fourier transform infrared spectroscopy measurement showing absorption features related to nitrogen-hydrogen complex formation in CdTe:N,H.

(7811.9Å) which is below the shallow donor level in CdTe. The DAP band is gone as expected. However, two series of sharp lines with zero-phonon peaks at 1.4991 and 1.4899 eV appear, along with several orders of phonon replicas. The spacing between the replicas corresponds to the 21 meV expected in CdTe. The two series of lines show distinctly different temperature behavior. The 1.4899 eV lines decrease with increasing temperature, while the 1.4991 emission increases up to a temperature of 45K where it then decreases. In addition, the 1.4991 eV emission is extremely sensitive to excitation energy and is best seen when the Ti:sapphire laser was tuned to about 1.587 eV. In contrast, the recombination mechanisms resulting in the series of lines beginning at 1.4899 eV have a very broad absorption band and can be seen under excitation within the range 1.50 to 1.59 eV.

The origins of the new PL lines shown in Fig. 5b are not completely understood at this time. The emission appears to be related to the presence of hydrogen during growth as it was not observed for either undoped or nitrogen-doped CdTe grown without hydrogen. The excitation-energy and temperature behavior of the 1.4899 eV transition is similar to that attributed to isoelectronic oxygen centers in ZnTe.³⁰ However, the emission energies do not correspond to that attributed to isoelectronic oxygen in CdTe.³¹ One could propose an isoelectronic complex, such as $N_{\text{Te}}\text{-H}$; however, we observe the same selective excitation spectra from the CdTe:H layers grown without a nitrogen flux. The dominant zero-phonon line at 1.4899 eV gives a level about 116 meV below the conduction band. In the previous report of hydrogen interactions with nitrogen acceptors in CdTe,²⁰ no new PL features were observed. We are currently performing a more

detailed study of this interesting transition.

Previous studies have indicated that room temperature PL has significant excitonic contributions.³² Interestingly, the room temperature PL measured for the samples grown under hydrogen flux was comparable to that observed in both undoped and nitrogen-doped CdTe. This indicates that the mechanism quenching excitonic PL at low temperatures is not present at room temperature.

Depth profiles for hydrogen and nitrogen from one of the CdTe:N,H sample were obtained using SIMS analysis. SIMS confirmed nitrogen incorporation at about $1 \times 10^{17} \text{ cm}^{-3}$ in this sample. Although there was again a fairly high background of hydrogen present during the measurement, a distinct increase in hydrogen was observed that correlates with the step in nitrogen concentration. Within the accuracy of the measurement, the increase in hydrogen concentration matched or exceeded that of nitrogen. Thus, the presence of nitrogen is observed to enhance hydrogen incorporation in at least a one-to-one fashion. A similar enhancement has been reported for ZnSe:N,H.²³ Hall measurements made on the CdTe:N,H indicated that the samples were heavily n-type, with carrier concentrations of about $1 \times 10^{17} \text{ cm}^{-3}$.

Infrared absorption spectra were taken using a Nicolet model 550 Fourier transform spectrometer with 0.5 cm^{-1} resolution at 15K. Two new absorption features, shown in Fig. 6, were observed at about 3210 and 749 cm^{-1} for the CdTe:N,H sample. These features were absent for undoped layers, nitrogen-doped layers grown without a hydrogen flux, undoped layers grown under a hydrogen flux, and bare substrates. The new absorption bands are taken as an indication of N-H bonding. The 3210 cm^{-1} peak can be associated with the N-H stretching mode while the 749 cm^{-1} absorption band may be related to a N-H wagging mode.²⁸ The presence of both these features in the sample known to contain significant levels of both nitrogen and hydrogen strongly implies that H forms a complex with N acceptors in CdTe. Thus, it is easy to understand the quenching of PL associated with nitrogen due to complex formation. The presence of the N-H bond removes the N from acting as an acceptor. Indeed, the N-H complex appears donor-like based on the Hall results.

This data, coupled with previous reports of N-H bonding in ZnSe,²¹⁻²³ As-H bonding in CdTe and ZnTe,²⁴ and P-H bonding in ZnTe²⁴ indicates a trend that H will form a strong bond to Group V dopant atoms situated on the chalcogen sublattice. The implication is that care must be taken to minimize hydrogen compensation not only in growth but also in subsequent processing steps. This is particularly true for the reactive ion etching processes currently used for most optoelectronic device processing since they contain a large background of reactive hydrogen. It is interesting that the DAP PL for copper is still present. Thus, H does not complex with Cu, at least not by the same mechanism as with N. A speculation is that p-type dopants on the metal sublattice may not form

compensating bonds with hydrogen. Unfortunately, such dopants typically have large diffusion coefficients precluding layered device fabrication.

SUMMARY

Nitrogen doping in CdTe epilayers grown by photo-assisted molecular beam epitaxy was achieved using an rf plasma source. The layers were characterized using photoluminescence spectroscopy, Hall effect, secondary ion mass spectroscopy, Fourier transform infrared spectroscopy, and atomic force microscopy. PL confirmed the presence of nitrogen acceptors. While p-type carrier concentrations greater than 10^{18} cm^{-3} were easily obtained, SIMS measurements indicated that most of the nitrogen was concentrated near the undoped-doped and epilayer-substrate interfaces which complicates interpretation of activation efficiency.

The effect of the presence of hydrogen during the growth of undoped and nitrogen-doped CdTe has been investigated. Quenching of exciton-related and nitrogen-related PL was observed while Cu-related DAP luminescence was enhanced. New PL features were measured that appear to be related to hydrogenation. Results of PL, SIMS, and FTIR measurements made on CdTe:N,H samples are consistent with N-H complex formation.

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