Luminescence and Electroluminescence Properties of Nd, Tm, Yb Doped GaAs and Some II-VI Compounds

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by:

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This report describes the progress accomplished during the first year of research on luminescence and electroluminescence properties of Nd, Tm, Yb doped GaAs and some II-VI compounds. The photoluminescence study of GaAs:Yb shows no 4f emission. The PL spectra of CdS:Nd were recorded and about 20 sharp emission lines were observed. This indicates that in CdS, Nd³⁺ occupies different symmetry sites. The PL of CdS:Yb at 9.3 K reveals five sharp lines in the 985 nm - 990 nm range and a strong broader line at 998.3 nm. Electroluminescence of ZnS:Tm embedded in a Boric matrix was observed for the first time, Strong emission was observed at room temperature as well as at low temperature revealing only five groups of strong sharp lines which are assigned to transitions within the 4f shell of Tm³⁺. EL intensity was investigated as a function of voltage, temperature and frequency. The voltage dependence of the EL intensity shows that the direct impact excitation mechanism is a dominant one. Photoluminescence spectra of InP:Yb at different temperatures consist of sharp peaks related to Yb³⁺ transition. Time-resolved PL spectra indicate that the observed emissions originate from one Yb³⁺ center. Rise time kinetics reveals the fact that the excitation process of the Yb³⁺ is indirect through the Yb²⁺ isoelectronic trap. Electric field quenching of photoluminescence was observed for the first time.
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PERSONNEL

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SCIENTIFIC PAPERS

• The following scientific papers have been presented at the Ohio Section of the American Physical Society, Athens, Ohio, April 1991:

1. A. K. Alshawa and H. J. Lozykowski, "Electroluminescence of Tm doped ZnS".


• The following scientific papers have been prepared for publication as a result of the research performed under Air Force sponsorship:


INTRODUCTION

In recent years the luminescence (PL and EL) properties of semiconducting materials such as Si, II-VI and III-V doped with rare earth ions have been studied intensively because of their potential application to light emitting devices. The emitted sharp lines with emission wavelengths insensitive to temperature variation results from RE\(^{3+}\) ion 4f transition.

The purpose of this research is to study the photoluminescence (electroluminescence) of GaAs doped with Nd\(^{3+}\), Tm\(^{3+}\), Yb\(^{3+}\), and some II-VI compounds (ZnS:Tm\(^{3+}\), CdS:Yb\(^{3+}\), CdS:Nd\(^{3+}\), ZnSe, ZnTe, and Cd\(_{1-x}\)Zn\(_x\)Te:Tm\(^{3+}\)). In addition, we study InP:Yb because the luminescence spectrum of this crystal does not depend on the sample preparation method and the ytterbium ion has a simple spectrum because the Yb\(^{3+}\) 4f\(^{13}\) shell has only one excited state. The deep understanding of excitation mechanism in such a simple system can lead to better understanding of excitation mechanism in other more complicated RE\(^{3+}\) systems in semiconductors.

Figure 1a shows the energy levels scheme of the Nd\(^{3+}\) ion in cubic (T\(_d\)) and noncubic symmetry (C\(_{v3}\), C\(_{v2}\), C\(_s\)) environment. In cubic symmetry 4\(F\)\(_{3/2}\) level is not split (\(\Gamma_8\) fourfold degenerated crystal field state). Therefore, in the cubic hosts, any splitting of the 4\(F\)\(_{3/2}\) level is indicative for association of the Nd\(^{3+}\) ion with other defects. The same argument holds for the multiplicities of the ground state. The ground state 4\(I\)\(_{9/2}\) in cubic symmetry is split into three states (\(\Gamma_8\), \(\Gamma_8\), \(\Gamma_7\)) and the first excited state 4\(I\)\(_{11/2}\) into four crystal field states as in the left part of Fig. 1a. In noncubic symmetry, the fourfold degenerate \(\Gamma_8\) states of Nd\(^{3+}\) ion are split into two doublet each and the energy level structure is shown in the right part of Fig. 1a.

The emission spectra for the 4\(F\)\(_{3/2}\) \(\rightarrow\) 4\(I\)\(_{9/2}\) transitions of Nd\(^{3+}\) in noncubic symmetry should exhibit all ten transitions. At low temperature, when only the lower level of 4\(F\)\(_{3/2}\) is occupied, only 5 lines should be observed in luminescence emission. In absorption at low temperature such that only the lowest component of 4\(I\)\(_{9/2}\) state is occupied, only two lines should be observed. In Fig. 1b we show the possible absorption and emission transition with wavelength (in \(\mu\)m) for Nd\(^{3+}\) in cubic symmetry in semiconductors with band gap \(E_g \geq 2.14\) eV. Figure 1c shows energy level schemes for Yb\(^{3+}\) ion in cubic (T\(_d\)) and noncubic symmetry, and the luminescence transition for Tm\(^{3+}\) ion in ZnS is shown in Fig. 1d.
Figure 1 a,b,c,d
EXPERIMENTAL

Photoluminescence & Electroluminescence

The photoluminescence measurements were carried out using a Laser Ionic Model 553A Ar+ laser as the excitation source. A Spectra Physics Model 3900 CW Ti:Sapphire laser was used for excitation spectra. For time-resolved spectra and time measurements, the pulsed excitation was obtained by an IntraAction Model AOM-125 acousto-optic light modulator with rise and fall times around 2 nsec. The drive power for the light modulator was provided by an IntraAction Model ME-125 signal processor. The pulsed excitation was detected by an Antel Model AR-S2 picosecond photodetector and monitored on an HP Model 54503A 500 MHz digital oscilloscope. In electroluminescence experiments, a sinusoidal signal generator and a DC power supply were used to perform total emission measurements. For pulsed excitation EL, an AVTECH Model AVR-4B-C pulse generator was used. The samples were mounted on a cold finger cooled by a closed-cycle helium cryostat down to 8.5 K.

The emission was dispersed by a Jarrel Ash Model 78-490 0.75m scanning monochromator equipped with a 1180 grooves/mm grating. The signal was detected by photomultipliers Hamamatsu R632-01 and R925 with spectral response in the range of 400-1200 nm and 400-930 nm, respectively. The second photomultiplier was also used to detect the pulsed excitation signal in the PL time measurements. The signal from the photomultiplier is passed through a fast ORTEC 474 preamplifier to a dual channel gated photon counter Model SR-400. Finally, the output signal is transferred to a computer for analysis and plotting.

Polarization

For photoluminescence (electroluminescence) polarization measurements we modified our digital spectropolarimeter to operate at the spectral range from 450 nm to 2000 nm (in digital mode the range from 450 nm to 1200 nm is limited by the photomultiplier spectral response.) The modified optical range was accomplished utilizing Oriel Fresnel rhomb achromatic retarders. The quarter wave or the half wave achromatic retarders are in series with a Glan-Thompson crystal polarizer in the excitation channel to provide $90^\circ \pm 2^\circ$ ($\sigma^+$ or $\sigma^-$) or $180^\circ \pm 2^\circ$ (linear polarization)
phase retardation. The heart of the setup consists of a photo-elastic modulator (PEM-80) and a dual channel gated photon counter which has the capability to compensate the variation of excitation intensity with time and wavelength. The second important modification is automatic wavelength tracking of the modulator to insure the constant retardation phase ($\pi/2$ or $\pi/4$) when the wavelength of the analyzed light is scanned in wide range. The angular aperture of PEM-80 modulator comprises a 50 degree full-cone angle.

Unlike the conventional lock-in or other analog systems, where the difference of two oppositely polarized light components (AC) and the average of these components (DC) are extracted electronically, and the ratio AC/DC is recorded as degree of polarization, our setup directly records the $\sigma^+$ and $\sigma^-$ components (or, in the case of linear polarization, $I_\parallel$ and $I_\perp$ components) of the light signal in the same run (of wavelength, temperature, etc.) The block diagram of the instrument for the measurement of the degree of circular or linear polarization of luminescence light and excitation polarization spectroscopy is shown in Fig. 2. A detailed description of the spectropolarimeter system is under final preparation for publication.

Sample Preparation

- **Implantation**

The ion implantation of rare earth was done by Dr. I. G. Brown from Lawrence Berkeley Laboratory at the University of California, Berkeley (because of technical problems, we received the samples at the end of May 1991.) Implantation of Nd, Yb, and Tm was performed with an unconventional implantation technique using a new kind of high current metal ion source developed at Berkeley by Dr. Brown et al. This MEtal Vapor Vacuum Arc (MEVVA) ion source makes use of a dense metal plasma generated directly from the metal cathode material as the medium from which ions are extracted. The source is repetitively pulsed and the implantation is done on a broad beam mode, with a direct line-of-sight from the ion source to the target. High current beams of a wide range of multiply-charged metal ions can be produced. Beam extraction voltage is up to 100 kV, for a mean ion energy of up to several hundreds of keV by virtue of a mean ion charge state that is typically 2 to 3. The beam current can be up to several amperes peak and around 10 mA time averaged delivered onto target. The source has been operated with virtually all of the solid metals of the periodic table. The high energy implants of 1 MeV
Figure 2  Block diagram of the spectropolarimeter
(sample 15) were performed at Universal Energy Systems (UES), Dayton, OH. The list of III-V and II-IV semiconductor compounds, their implantation parameters and post implantation annealing conditions is summarized in Table I.

- **Annealing**

The samples were cut to small pieces (4 mm x 4 mm squares) and then cleaned by standard methods. Thermal heat-pulse using a strip heater was used to anneal the samples up to 900°C for 10-15 sec. The annealing process was performed using the apparatus shown in Fig.3 which we designed and built in our laboratory. A flat graphite plate of ≈ 3mm thickness is placed on the tantalum strip heater. The samples are placed face down on the graphite plate and covered with a thin graphite lid of ≈ 1mm thickness. The temperature is measured and monitored by OMEGA Model 650 microprocessor controlled temperature indicator through a thermocouple placed inside the graphite plate. The variation of annealing temperature with time is recorded by an x-y recorder as shown in the inserts of Fig. 3. The rise time of the temperature was controlled by the AC voltage applied to the strip heater. For temperatures up to 900°C, our system maintains a rise time of 6 sec and a decay time from 900°C to 500°C of 22 sec.

![Figure 3 Thermal-Heat-Pulse Annealing System](image-url)
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* SI: Semi-insulating Cr-doped (0.1-2x10^16 cm^-3) GaAs
  p: p-type Zn-doped (p=1x10^17 cm^-3) GaAs
  n: n-type Si-doped (n=1x10^18 cm^-3) GaAs
** p-type Be-doped GaAs epilayer on a p'-type Zn-doped GaAs
*** Three samples with three different Zn concentration, x, 10%, 22%, and 31%
RESULTS AND DISCUSSION

Photoluminescence of GaAs:Yb, CdS:Nd, CdS:Yb

The photoluminescence (PL) study was performed only on some of the samples listed in table I because we received the samples from implantation at the end of May 1991, and we are still waiting for cooled Germanium infrared detectors EO-817P and EO-817S from North Cost Optical Systems and Sensors.

The PL study of GaAs implanted with Yb$^{3+}$ ions shows no 4f intracenter emission. Similar results were reported by G. S. Pomrenke et al and K. Takahei et al on MOCVD-grown Yb doped GaAs despite the Yb incorporation being confirmed by SIMS. The absence of 4f Yb$^{3+}$ emission in GaAs is unknown and required more investigations. The direct excitation of Yb$^{3+}$ ions by photons, impact excitation in high electric field, or by electron beam excitation will give the answer for the question: are the incorporated Yb$^{3+}$ ions optically active in GaAs or not? This type of experiment is planned in the near future in our laboratory.

Figure 4 shows the characteristic sharp emission spectra of Nd$^{3+}$ implanted CdS and rapid thermal annealed (RTA) at 900°C for 15 sec in forming gas. The excitation was performed using a 488 nm Ar$^+$ ion line at 9.3 K. The observed emission lines are due to $^4F_{3/2} \rightarrow ^4I_{9/2}$ transitions of Nd$^{3+}$ ions in noncubic symmetry. At temperature 9.3 K, only the lower level of $^4F_{3/2}$ is occupied, and we should observe only 5 lines in luminescence emission (Fig. 1a left). Our spectrum shows about 20 lines. This observation indicates that in CdS, Nd$^{3+}$ ions occupied different symmetry sites. The preliminary kinetics study of decay of emission lines at 898.7 nm and 903 nm shows decay times 340 µs and 318 µs, respectively.

The EPR study (Watts et al) shows that isolated RE$^{3+}$ ions are found to be substitutional for the metal ion in zinc blende (ZB) crystals. Spectra of RE$^{3+}$ ions in wartzite (W) lattices are coming from three types of sites: (1) the RE$^{3+}$ ions form several types of associates with charge-compensating ions; (2) the substitutional RE$^{3+}$ ions with no local charge compensation (the possibility of a compensating defect along the C-axis cannot be excluded); (3) another axial and orthorhombic spectrum are observed. Identification of observed emission centers required additional investigation such as time-resolved, polarization and excitation spectroscopy which are in progress in our laboratory.
Implantation Sample: CdS:Nd
- Energy 150 keV
- Temp. 9.3K
- Dose 7.5E13 cm
- Exc. 488 nm Ar laser
Annealing:
- Power 100 mW
- Temp. 900 C
- Time 15 sec

Figure 4

Implantation Sample: CdS:Yb
- Energy 140 keV
- Temp. 9.3K
- Dose 7.5E13 cm
- Exc. 488 nm Ar laser
Annealing:
- Power 100 mW
- Temp. 900 C
- Time 15 sec

Figure 5
Sample: CdS:Yb
Exc. 488 Ar⁺ laser
Power 100 mW
Temp.:
(a) 8.5K (b) 30K
(c) 60K (d) 120K

Figure 6
As seen in Fig. 5, for CdS:Yb the characteristic sharp RE emission lines were observed at a spectral range from 895 nm - 1010 nm for transitions between the crystal field split spin orbit level $^{2}F_{5/2} \rightarrow ^{2}F_{7/2}$ of Yb$^{3+}$ ($4f^{13}5s^25p^6$). The emission spectrum contained five sharp lines in the wavelength region of 985 nm - 990 nm and a strong broader line at 998.3 nm with broad, not well resolved peaks between the 990 nm - 1020 nm region. Figure 6 shows the temperature dependence of $^{2}F_{5/2} \rightarrow ^{2}F_{7/2}$ emission lines. The emission intensities increased as temperature increased from 8.5 K until they reached their maximum value at about 30 K, then they started to decrease with further increases of temperature. At a temperature of 60 K, the sharp emission lines in the 985 nm - 990 nm region are temperature broadened and merged and become unresolved at higher temperatures.

**Electroluminescence of ZnS:Tm**

The electroluminescence of thulium doped ZnS embedded in boric matrix, silicon rubber and some organic binders was investigated in comparison to photoluminescence. The power efficiency of the ZnS:Tm was determined under cathode-ray excitation as 0.216 W/W, the highest yet reported for any rare-earth-activated phosphor. The EL cell was prepared using transparent conducting glass (In$_{0.9}$Sn$_{0.1}$O$_2$) as shown in Fig.7. The Electroluminescence spectra were investigated as a function of temperature, voltage and frequency. The emission spectra show only strong sharp emission lines which are assigned to transitions within the 4f shell of Tm$^{3+}$. The

![Figure 7 The ZnS:Tm Electroluminescence Cell](image_url)
emission spectra revealed five groups of lines at 483 nm (transitions $^{1}G_{4} \rightarrow ^{3}H_{6}$), 662 nm (transitions $^{1}G_{4} \rightarrow ^{3}H_{4}$), 711 nm (transitions $^{3}F_{3} \rightarrow ^{3}H_{6}$), 785 nm (transitions $^{1}G_{4} \rightarrow ^{3}H_{4}$) and 806 nm (transitions $^{3}F_{4} \rightarrow ^{3}H_{6}$). Strong emission was observed at room temperature as well as low temperature. The voltage dependence of the EL intensity and kinetics were studied under sinusoidal, pulsed and DC excitations. The temperature dependence of the emission intensity was investigated in the range of 8.5 K-290 K, as shown in Fig. 8 A, B, C. The emission around 483 nm and 660 nm decreased as the temperature increased. The emission intensities around 805 nm increased with temperature increase until they reached their maximum value at 210 K, then they started to decrease with a further increase in temperature. However, a strong sharp peak at 805 nm can still be observed at room temperature. The voltage dependence of the EL intensity and the kinetics were studied under sinusoidal, pulsed and DC excitation. Analysis of the emission intensity of the applied voltage indicates that the direct impact excitation is a dominant one. Strong DC EL was observed at room temperature at a low voltage of 38 V. The investigation of the emission intensity as a function of frequency was performed in the frequency range of 1-16 KHz. The intensities of all lines increased to a maximum at 7 KHz and then decreased by a further increase in the excitation frequency. A detailed discussion on electroluminescence of ZnS:Tm is under final preparation for publication.
Figure 8 A, B, C
Luminescence Properties of InP:Yb

The photoluminescence of Yb-implanted InP was investigated under CW and pulsed excitation using Ar⁺ ion laser. Figure 9 shows the PL spectra of the different samples of InP:Yb listed in Table I. The spectra consist of a sharp peak at 1001 nm and two broader peaks at 1005.5 nm and 1007 nm (transitions ²F₅/₂ → ²F₇/₂). The time-resolved PL spectra were recorded for the three samples as shown in Fig. 10. The intensities of all emission lines decreased uniformly with increasing the time delay after excitation. This indicates that the emission originates from the same Yb³⁺ center. The study of the rise time and decay time processes (intrinsic excitation) shows that the rise time (9.3 μs) is smaller than the decay time (11.7 μs). This reflects the fact that the excitation process of the Yb³⁺ is indirect through the Yb³⁺ isoelectronic trap (the isoelectronic trap model was proposed by Witney et al). By assuming the isoelectronic trap model, the kinetics study shows that if the excess electron relaxation time is much shorter than the center-filling relaxation time, the RE³⁺ luminescence response to a square pulse excitation will be exponential assuming uniform excitation. This is fulfilled in our case because of the small thickness of the Yb³⁺ implanted layer (≈ 300 nm). The rise of the luminescence intensity is an exponential function with a time constant that depends on the excitation intensity. For generation rate (excitation intensity) the rise time will be the capture electron lifetime on the RE isoelectronic trap. Therefore, with increasing the excitation intensity, the rise time will decrease which is confirmed by our experimental results. PL excitation spectra for the three emission lines of 1000.1 nm, 1005.5 nm, and 1007 nm were recorded using a tunable Ti:Sapphire laser. No emission is observed for excitation below the band gap due to the very small thickness of the Yb-implanted layer. The temperature dependence of InP:Yb was investigated in the 9K - 100K range. From Fig. 11 we can see that the intensities of all emission lines decreased when increasing the temperature. We studied the effect of the electric field on the PL of InP:Yb. To perform this experiment, a semi-transparent gold electrode (100Å) was evaporated on the implanted surface of InP and a second thick gold electrode (500Å) was deposited on the opposite side of the sample. Figure 12 shows the quenching of PL of Yb³⁺ emission as a function of the electric field. The insert of Fig. 12 shows the electric field in the surface layer under the semi-transparent gold electrode. Assuming the uniform absorption coefficient of 10⁵ cm⁻¹ for InP at 9 K, 95% of the excitation photons are absorbed within 300 nm of the surface layer where a strong electric field exists. The quenching of PL of Yb³⁺ can be explained by two possible mechanisms: (1) exciton bound to the isoelectronic
trap disassociation by hot electron impact ionization or (2) swept of electrons or holes to the Au-InP interface where they will recombine nonradiatively. The electric field quenching of photoluminescence presented in Fig. 12 needs more investigations to exclude the possibility of temperature quenching.

**Figure 9**

**Figure 10 A**

**Figure 10 B**

**Figure 10 C**
Figure 11

Figure 12
REFERENCES

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