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PICOSECOND NONLINEAR RESONANT INTERACTIONS IN SEMICONDUCTORS

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1. Research Objectives and Summary

The objective during the last phase of the research under AFOSR Contract F49620-82 C-0044 (1/1/85 to 12/31/85) was aimed at advancing the understanding and utilization of selected optical properties of novel semiconductors containing magnetic elements. We have carried to succesfull completion our work on semimagnetic semiconductors of the II-VI family (CdMnTe and CdMnSe). As a final new departure, emphasis during this contract period was placed on the infrared properties of a new semiconductor material, (Pb,Eu)Te, which has been shown to be of significance in infrared light emitting applications. The element Eu contains permament magnetic moment from its f-electron shell and we have investigated both the onfluence of these inner shell electrons on the states defining the optical bandgap in the material, as well as studied the magneto-optical properties of (Pb,Eu)Te. Our experimental work is the first to characterize the optical and magneto-optical properties of this mixed crystal over the entire composition range of Eu (0% to 100%). In ultrathin layers of this material, we have also characterized the electronic properties in the so-called 'superlattice structures', to obtain first results about the electronic energy states in PbTe/(Pb,Eu)Te quantum wells and the kinetics on nonequilibrium electronic excitations ...

The research results derived from this AFOSR sponsored research have formed the basis of a number of scientific publications, as enumerated below. In addition to contributions to regular scientific meetings, the principal investigator has also been invited to present the research results in different forums.

2. Research Accomplishments and Results

Optical Properties of a New Infrared Material: $Pb_{1-x}Eu_{x}Te$

In this phase of the AFOSR supported reserach we have focussed on optical studies of a novel semiconductor material, $Pb_{1-x}Eu_{x}$ Te. Recent advances in molecular beam epitaxial growth of $Pb_{1-x}Eu_{x}Te$ have demonstrated the usefulness of this narrow gap material for infrared optoelectronic applications, specifically in the fabrication of PbTe/Pb1__Eu_Te heterojunction diode lasers including quantum well structures (1),(2). The range of Eu-concentrations so far employed in these devices has been relatively low (x< 0.05) and useful information has been obtained about the basic optical properties of $Pb_{1-x}Eu_x$ Te and the PbTe/Pb_{1-x}Eu_xTe multiple quantum wells (MQW) in this concentration range (3),(4). On the other hand, very little is presently known about the basic properties of $Pb_{1-x}Eu_x$ Te for higher Eu-concentrations, limited to an early study on evaporated thin films (5). Among the important issues for experimental measurement is the concentration dependence of the effective (optical) energy gap and associated magneto-optical effects. While for low x-values the bandedge states of $Pb_{1-x}Eu_x$ Te are expected to be PbTe-like (composed primarily of p-orbitals with a direct gap at the L-point in the Brillouin zone), experimentally derived arguments for the bandstructure of the antiferromagnetic EuTe show the importance of the Eu^{2+} -ion f- and d-electron states in defining the optical gap (6). Since Eu enters the alloy as a divalent ion, the half-filled f-electron shell is expected to contribute a magnetic moment from a pure spin state (S=7/2 in the free ion). This raises the question of the spin exchange interaction between the Bloch states and the ground f-electron states as already investigated broadly in the II-VI 'semimagnetic' semiconductors as well as in (Pb,Mn)Te and (Pb,Mn)S. In this section of our report we present results of

luminescence and absorption studies on several thin film samples over a concentration range up to x=0.30, which have allowed us to speculate about the nature of the electronic states which define the effective optical bandgap in this alloy. In addition, we also discuss measurements of the magnetic field dependence of the optical gap on selected samples which we use for an empirical description of the 'p-f' spin exchange effects in $Pb_{1-x}Eu_{x}Te$ at moderate Eu-concentrations (x<0.20). We also briefly note observations on samples of higher Eu-concentration.

The samples used in these studies were MBE-grown single crystal thin films of $Pb_{1-x}Eu_xTe$ on (111) oriented BaF_2 substrates (by Dr. D.L. Partin at General Motors Research Laboratories). No effort was made to control the strain induced by the finite film/substrate lattice mismatch although lattice matching to PbTe substrates with the addition of selenium at low x-values has been demonstrated (1). The thin films varied in thickness from approximately 0.3 um to 30 um and were, in most cases, not intentionally doped. However, nonstoichicmetric growth under conditions of excess Te-vapor leads to p-type material with a hole density on the order of $1-2x10^{17}$ cm⁻³.

Low temperature photoluminescence measurements were performed with a continuous wave Nd;YAG laser as the excitation source ($h\u=1.17 \text{ eV}$) at low excitation intensities. The samples were mounted on the coldfinger of a standard variable temperature dewar for both luminescence and absorption measurements, while a separate superconducting magnet dewar was used for the field dependent studies. Figure 1 shows luminescence spectra for two $Pb_{1-x}Eu_{x}Te$ films of x=0.032 and 0.20 at T= 10K (see comments below about the concentrations). The linewidth of the sample of lower Eu-concentration



Figure 1: Photoluminescence spectra at the optical gap from two MBE grown thin film samples of (Pb, Eu)Te at T= 2K.

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(left panel) is typical for a low density electron-hole plasma recombination spectrum. The sample with x=0.20 shows a pronounced broadening on the low energy side, similar to that observed in other semiconducting alloys at increased concentrations and which has been modelled in terms of alloy potential fluctuations (7).

The optical bandgap as a function of Eu-concentration obtained from both luminescence and absorption measurements on a number of $Pb_{1-x}Eu_xTe$ thin films up to x=0.30 is shown in Figure 2 at T= 10K. The luminescence spectra frequently showed additional emission features at lower photon energies and we studied such spectra carefully (e.g. as a function of excitation density) before extracting values for the optical gaps. The effective gap energies were then defined from the peak photon energies for the luminescenece spectra. The absorption data was used to correlate this information; in particular for x>0.05 corresponding values for the optical gap were obtained from a linear extrapolation of (absorbance)² against the photon energy axis. The choice of this parabolic functional form has no rigorous justification in this case because of the unknown energy dependences of the densities of states, and it was simply chosen as a best fit over a wide concentration range. Using the relatively narrow luminescence linewidths and the rather good linear fits to (absorbance)² vs. photon energy data (over a limited energy range), the two procedures generally agreed to within 5-10 meV on the several samples for which such consistency was checked. The dashed line in the figure refers to the recently derived dependence of $E_q(x)$ at low concentrations (x<0.04), obtained from emission energies of $Pb_{1-x}Eu_x$ Te lasers (1). Furthermore, we verified that up to 77K the empirical temperature dependent expression for $E_{\alpha}(x,T)$ of Ref. 2 was obeyed rather well. However, we also accounted for



Figure 2: Optical gap of (Pb,Eu)Te as a function of the Eu-concentration at T= 10 K as obtained from luminecsence and absorption measurements. The dashed line refers to the empirical expression in Ref. 3.

the finite uncertainties in the composition and strain in our samples by explicitly assuming that the dependence of ${\tt E}_{_{\bf Q}}$ on x is indeed linear in this range. Our data in Figure 2 then clearly shows that while the optical gap opens up rapidly for low x-values ($dE_{\alpha}/dx = 5.788$ eV for the dashed line), the slope decreases strongly for x > 0.05. Furthermore, while for samples with x up to 0.10 the thermal coefficient $dE_{\alpha}/dT>0$, we found that this coefficient had changed sign for the x=0.20 sample (and higher values). Results for three other thin film samples at considerably higher concentrations x>0.3 (x=0.47, 0.71, 1.00) showed evidence that the electronic character of the states defining the optical gap (in absorption) is now further strongly changing (with x) from a PbTe-like system. Among other things, this manifests itself with an increasing Stokes shift between the luminescence and absorption edges, reaching a value of approximately 0.7 eV in the EuTe limit, as will be detailed elsewhere (8).

Magnetic field dependence of the bandedge luminescence spectra for the two $Pb_{1-x}Eu_{x}$ Te thin film samples discussed above in connection with Fig. 1 is summarized in Figure 3, where the peak emission energies are plotted up to a field of 5 Tesla in the Faraday geometry. Note that while the sample with x=0.032 shows predominantly a spectral blueshift with increasing field, the peaks for the x=0.20 thin film display a strongly temperature dependent spectral redshift. The dashed lines correspond to a model calculation discussed below.

We now consider these measurements and their implications on the electronic character of those states which define the effective bandgap in $Pb_{1-x}Eu_{x}Te$. Our observations are largely empirical in nature and have



Figure 3: Peak position of the luminescence emission (in meV) as a function of external magnetic field for two samples of different Eu-concentration. The dashed lines refer to the model calculation discussed in text.

partial difficulty in the fact that the bandstructure for EuTe appears still to be under some theoretical debate. At low x-values (x<0.05), however, it seems clear that the effective gap is composed of conduction and valence band states which are still predominantly 'PbTe-like'. The large slope dE_{α}/dx is assumed to be constant and $dE_{\alpha}/dT>0$. The magnetic field induced shifts in the luminescence emission energy (lower panel in Fig.3) are compatible with optical transitions which occur between the lowest spin split Landau levels of the conduction and valence bands. A smaller (oppositely shifting) contribution from an exhange term is also present as summarized below. The striking feature in Fig. 1 is, of course, the rather precipitous decrease in the slope dE_q/dx for x>0.05. With increasing Eu-concentration, magneto-optical data for the x=0.20 sample and the observation of $dE_{\rm g}/dT{<}0$ support the argument that substantial additional mixing to the initially p-like conduction and valence orbitals is continuing to take place. This hybridization can in principle involve the s-, p-, and f- electron orbitals of the Eu^{2+} -ion but we do not estimate their contributions in this paper any further (in part because of the lack of e.g. useful tight-binding parameters). Finally, the Stokes shifts between the absorption edge and the luminescence spectra which become clearly evident beyond x>0.30 show that in this regime of concentrations, further changes have been introduced by the Eu²⁺-ion to those states which define the optical gap (in absorption). Specifically, the Stokes shifts appear to reflect a strong configurational relaxation (Jahn-Teller) and thus point towards the introduction of the f-states into the gap.

To characterize the magneto-optical effects shown in Figure 3 further, we have taken a first order phenomenological approach to test one possible

description for the exchange interaction between localized paramagnetic Eu^{2+} -ion moments with spins of extended electronic states (which lead to spin splittings of the conduction and valence band states). This approach is well established in the wider gap II-VI compound 'semimagnetic' semiconductors 'with Mn^{2+} as the paramagnetic ion) where the spin exchange is a Heisenberg-like short range interaction (9). Also, in the present case, the relatively low background free carrier concentration makes an RKKY-like exchange mechanism of negligible importance. Accordingly, we have fitted the data in Fig. 3 to the following parametrized expression:

$$\Delta E = \Delta E_{O}(B, m^{\star}, g) - A(x) \times B(g' u \beta / k(T+T_{O}))$$
(1)

Here AE is the spectral shift in the luminescence emission energy (optical gap) and the first term on the right, ΔE_{o} , is the contribution from the spin-split lowest Landau-level (proportional to the applied external field in the range of interest here). The second term, where the Eu-concentration is x, originates from the assumed Heisenberg-like exchange mechanism with a Brillouin function B in whose argument g' is the g-factor for the Eu^{2+} -ion. As for the free ion, we take S=7/2, L=0, and assume that Hund's rule is valid. By using the empirical treatment applied earlier to the II-VI semimagnetic semiconductors (10), we account for the finite antiferromagnetic (AF) spin-spin interactions within the Eu²⁺ system by using a temperature correction T_{O} in the argument of the Brillouin function so that $T_{eff} = T + T_{o}$, and define a concentration dependent interband exchange coefficient A(x) . Note that our present experiment is not able to separate out the valence and conduction band contributions to these exhange coefficients. For evaluation of the exchange effect we can subtract away the 'Landau level contribution' to

lowest approximation by extrapolating from measured magneto-optical constants of PbTe (11) in terms of an effective mass (and g-factor) which have been assumed to have an approximately inverse dependence on the effective mass for the different x-values. Then the fits in Figure 3 are obtained for the following parametrized values of the exchange term in Eq. 1: (i) for the sample with x=0.032, A= 49 meV and $T_0 < 0.5$ K; (ii) for x=0.20 material A= 30 meV and $T_0 = 4.3$ K in the lower temperature case. For the lattice temperature of T= 15 K we find that the best fit requires a somewhat different (lower) value of $T_0 = 2$ K; this may imply that the description of Eq. 1 is not quite correct or show imprecision in our treatment of the Landau contribution. We also note that the forefactor A(x) decreases with x, as expected from increasing AF contributions, and that no spin saturation is evident at the moderate magnetic fields and temperatures employed in these experiments.

These estimates, while based on a limited amount of information and simplified in character, do suggest that even at x=0.20, the conduction and valence band extrema of (Pb,Eu)Te are still still largely composed of extended states. The approximate fitting to a Heisenberg-like spin exchange formulation to account for the magneto-optical shifts is not necessarily a unique solution to the problem but seems to us to be the most likely one. We have some support to these estimates from recent magnetization (and susceptibility) measurements which have yielded more direct information e.g. about the role of the AF interactions (12). These measurements on similar MBE grown (Pb,Eu)Te thin film samples show, for example, that the Eu²⁺-spin system is paramagnetic over the range of compositions (x<0.20) and temperatures considered here (12). Furthermore, fitting the magnetization to the modified Brillouin function yields

temperature corrections T_{o} which are in good agreement with the values obtained above. We note in passing other recent susceptibility measurements for (Pb,Eu)Te in a low concentration range (x from 0.001 to 0.018) which show very large (negative) Curie-Weiss temperatures (13). This has been interpreted in terms of the long range exchange interaction in a narrow-gap system such as (Pb,Eu)Te; however, the concentration range discussed in this paper is much higher and is likely to involve more direct processes. Finally, using the values extracted for the exchange amplitudes A(x) from above, we note that these are between one and two orders of magnitude smaller than corresponding the joint conduction-valence band contributions of exchange in the wider gap II-VI semimagnetic semiconductors. At the same time, magneto-optical effects in the narrow gap materials $Pb_{1-x}Mn_x$ ans $Pb_{1-x}Mn_xS$ have also been measured to be quite small (14), (15).

In summary, we have obtained through infrared luminescence and absorption measurements information about the optical bandgap of $Pb_{1-x}Eu_x$ Te over a wide range of composition. While using additional data from magneto-optical luminescence studies we suggest that in this narrow-gap mixed crystal, the character of the states which define the effective energy gap remains largely extended at least up to x=0.20. The origin of the pronounced change in the slope of dE_q/dx for x>0.05 is clearly a major unresolved theoretical problem in this connection, and from questions of hybridization, may also have a magnetic apart contribution through coupling with the Eu²⁺-ion magnetic moments. The magneto-optical effects at the optical gap have been found to be rather small but seem at least approximately fit to a simple exchange description. Here also a proper theory will require systematic additional

experimental input as a function of concentration x, including detailed correlation between magneto-optical and susceptibility data. Finally, approximately beyond x=0.30 the effective bandgap in (Pb,Eu)Te appears to contain states which are now directly related to the Eu^{2+} -ion f-electron (and possibly d-electron) bands.

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Electron-Hole Recombination Spectra and Kinetics in PbTe/PbEuTeSe Multiple Quantum Wells

In this segment of the AFOSR supported research we have focussed on the optical properties of electronic excitations in new narrow-gap semiconductor ultrathin layer structures, i.e. quantum wells, based on the Pb_{1-x}Eu_x material system. As an example we present here studies of PbTe/PbEuTeSe multiple quantum wells in samples of two different well thicknesses ($L_2=92$ A and 51 A) from measurements of photoluminescence over a range of temperatures and magnetic fields structures grown by Dr. D.L. Partin at General Motors Research Laboratories). From infrared luminescence we obatin transition energies which yield tentative assignments for values of the band offsets. Hot electron effects are pronounced in the $L_2=92$ A sample and show that the electron-LO phonon interaction is substantially reduced in comparison with bulk PbTe. The energies of the lowest Landau levels have been measured in several samples from shifts in luminescence energies in external fields. Finally, measurements of excess electron-hole recombination rates by time-resolved techniques show lifetimes less than 10 nsec in these quantum wells.

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The PbTe/PbEuTeSe multiple quantum well structure has been successfully fabricated for diode laser operation recently (1) and presents a useful system for study of quasi-2D electronic excitations through interband optical transitions because of the very small excitonic corrections. We have investigated basic optical properties of this superlattice for narrow wells $(L_z < 100 \text{ A})$ through photoluminescence measurements as a function of incident excitation level, temperature and external magnetic field. With pulsed laser techniques we have also measured the recombination lifetimes and find them typically to be less than 10 nsec at temperatures below 77 K.

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The samples used in this study were grown by MBE techniques on (100) oriented PbTe substrates (2). Buffer layers of PbEuSeTe of thicknesses 1-2 microns separated the quantum well layers from the substrate. Below, we show results for two samples, a PbTe/Pb $_{1-x}Eu_{x}Te_{1-y}Se_{y}$ structure with PbTe well thickness $L_{z}=92$ A and barrier thickness $L_{b}=940$ A, and a structure with $L_{z}=51$ A and $L_{b}=355$ A. The Eu concentrations for the two samples were x= .048 and x= .040, respectively. The selenium concentrations (y= .059 and .046) were chosen to minimize lattice mismatch and are not expected to appreciably change the near bandgap electronic properties of the ternary PbEuTe. The PbTe layers were assumed to have a backgound hole concentration of about $2x10^{17}$ cm⁻³ from comparison with data obtained for single crystal films of PbTe grown under similar conditions.

Figure 1 shows infrared luminescence spectra for two MQW samples at a nominal lattice temperature T=4K, obtained by photoexcitation from a Nd:YAG laser ($h\u$ =1.17 eV) under moderate power (P< 50 mW). For the wider well sample ($L_2=92$ A) the spectrum consists of a single feature (n=1 to n=1 conduction to



Figure 1: Photoluminescence from two PbTe/PbEuTeSe multiple quantum wells at T=4 K under moderate cw excitation. The amplitudes for each spectra are normalized to their respective peaks.

valence band transition) which acquires a nearly Boltzmann like tail at higher temperatures and excitation levels. We have shown recently how the excitation intensity dependence of the spectra for such MQW samples leads to readily observable hot carrier effects which have been interpreted in terms of a significantly reduced electron-optical phonon interaction of the photoexcited carriers (3). Furthermore for these samples, the low energy portion of the lineshape fits well with a model in which well width fluctuations of one monolayer contribute to the observed finite broadening. In contrast, as the well width decreases to approximately 50 A, a distinct low energy shoulder appears in low temperature luminescence (Fig.1). The shoulder (whose origin is not understood at present) decreases in amplitude and disappears with increasing temperature (T>20 K), but even in its absence only a poor fit can be made to the lineshape by only considering well width fluctuations. Comparison with another MQW sample with comparable dimensions (L_2 =52 A) but grown at a higher temperature (390 C vs. 320 C) shows nearly identical spectral behavior; this shows that interdiffusion is not a dominant factor in the thin well samples. From the spectral positions of the luminescence for the two samples in Fig. 1, estimate that the conduction band offset ratio in the PbTe/PbEuTe heterojunction $\Delta E_c / \Delta E_q = .90$ or .10.

The influence of an external magnetic field on the luminescence spectra is shown in Fig. 2 for the 51 A MQW sample. The lower part of the figure shows the dependence of the spectral position of the main peak at 2K for two orientations of the field vs. the superlattice growth axis z (parallel to [100] direction). The optical transition is assumed to correspond to lowest spin allowed transition between the conduction and valence band quantum well Landau levels. Clear anisotropy in the field induced shifts $\Delta E(B)$ is evident, as expected for a quasi-2D transition involving free electrons and holes. The upper part of the



Figure 2: Influence of an external magnetic field on the position of the luminescence peaks for a sample of 51 A PbTe quantum well width. Lower trace: Dependence on field orientation vs. superlattice axis at T=2 K. Upper trace: Lowest resonance and the appearance of the next spin/Landau transition at T=60

figure shows the field induced shift at 60K in Faraday geometry where, in addition to the lowest resonance, a next spin/Landau level transition has sufficient thermally excited population to be clearly resolved in the luminescence spectra at higher field values. The dashed straight lines are to guide the eye only. The absence of any strong temperature dependence in the slope $\Delta E/\Delta B$ shows that little contribution is made by any spin exchange effects of the electrons and holes with the Eu-ion f-electrons in the PbEuTe barrier (in spite of the substantial penetration of their wavefuctions into this region). At the same time, comparison with reported interband magneto-optical data in bulk PbTe (4) shows that the field induced shifts in our thin well MQW samples are substantially smaller. Details of a model which accounts for the magneto-optical behavior in the (100) PbTe/PbEuTeSe superlattices will be discussed elsewhere.

Finally, we have also made measurements of the recombination lifetime in several MQW samples by time-resolved photoluminescence. The source of excitation in these experiments was a cw modelocked Nd:YAG laser (t_p = 100 psec). At low excitation levels where transient luminescent signals are weak for direct detection, a useful experimental technique is based on a phase-shift approach to characterize the luminescence decays (5). Briefly, the high repetition rate pulsed output (100 MHz) of the laser was additionally amplitude modulated at 10 MHz and the relative phase of the photodetected luminescence from the MQW samples was compared with that directly emitted by the laser through a high frequency lock-in amplifier. As an example, at 77K our measurements indicate approximately an electron-hole lifetime for the L_z =92 MQW sample of 6 nsec and for an L_z = 51 A sample a lifetime of 4 nsec. These lifetimes become somewhat longer towards lower temperatures (to about 4K). Considering the high radiative efficiency and the moderate excitation levels (excess electron density < 1×10^{17} cm⁻³) our measurements indicate that a substantial shortening in the radiative

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lifetime in the quantum well structures has occurred when compared with reported values in bulk PbTe under comparable conditions (6). Additional experiments are now under way to examine the lifetimes in the PbTe/PbEuTeSe quantum well structures at higher carrier densities in order to examine the characteristics of Auger recombination in the quasi-2D case.

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- 3. <u>Scientific Publications and Presentations Resulting from the AFOSR</u> <u>Supported Research</u>
- "Picosecond Excitonic Phenomena in Semimagnetic Semiconductors", X.-C. Zhang, S.-K. Chang, and A. V. Nurmikko, SPIE Proceedings vol. 533, p. 18 (1985).
- "Photoluminescence in PbTe-PbEuTeSe Multiquantum Wells, W. Goltsos, J. Nakahara, A. V. Nurmikko, and D. L. Partin, Appl. Phys. Lett. 46, 1173 (1985).

- "Electron-Hole Recombination Spectra and Kinetics in PbTc/PbEuTcSc Multiple Quamtum Wells", W. Goltsos, J. Nakahara, A. V. Nurmikko, and D. Partin, Proc. Int. Conf. Modulated Semiconductor Structures, Kyoto, Surface Science (in press).
- "Optical Bandgap and Magneto-Optical Effects in (Pb,Eu)Tc", W. C. Goltsos, A. V. Nurmikko, and D. L. Partin, submitted to Solid State Comm.

In addition to these scientific publications, the AFOSR supported work has been presented in numerous scientific conferences.

- Topical Meeting on Semimagnetic Semiconductors, MIT, Cambridge, Oct. 1984.
- Philips Research Laboratories, Colloquium, Briarcliff Manor, NY, Nov. 1984.
- Gordon Research Conference (International) on Nonlinear Optics, Brewster, NH, July 1985.
- International Symposium on Semimagnetic Semiconductors, Aussois, France, Sept. 1985.

The following have had direct support from this AFORSR grant: Professor A. V. Nurmikko, Principal Investigator. William C. Goltsos, Ph.D. candidate.

5. Patents

No patents have been filed in connection with this AFOSR sponsored research.

6. <u>Remaining Funds</u>

No remaining funds exist.

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