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Defect specific luminescence dead layers in CdS and CdSe

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Abstract

CdS and CdSe are often used in optoelectronic devices whose effectiveness may be dictated by defects in the near surface region. Luminescence is one of the main tools for studying such defects. The energy dependence of the x-ray excited optical luminescence (XEOL) spectra of these materials enables the extraction of the depth-dependence of the defect distribution. Normal and time-gated XEOL spectra were obtained from these materials in the energy range 600 to 1500 eV. We find that the results can best be understood in terms of a luminescence dead layer whose width depends on the position of the defect level in the band gap.

Keywords: CdS, CdSe, luminescence, dead layer, x-ray

Introduction

One of the main tools for studying defects in electronic materials is luminescence. Besides near band gap and higher lying energy transitions in semiconductors and insulators, luminescence at energies below the minimum energy band gap arises from defect levels located in the band gap. X-ray excited optical luminescence (XEOL) has been shown to be a powerful tool for investigating the local chemical environment of a site that gives rise to a particular luminescent band.¹ Recent studies on ZnO nanowires and single crystal SrTiO₃ have demonstrated how XEOL may be used to determine the depth distribution of defects in these materials.²⁻³ The depth dependence arises from the energy and angle dependence of the x-ray attenuation length. In the absence of any core level excitations, the attenuation length (AL), defined as the depth into the material measured along the surface normal where the intensity of x-rays falls to 1/e of its value at the surface, increases with energy. We define the penetration length (PL) as the AL at a particular angle. For CdS (CdSe) at a grazing angle of 6 degrees the PL varies between 16 - 72 nm (9 – 45 nm) as the x-ray energy increases from 700 to 1400 eV.⁴

II-VI semiconductors are well-known luminescent materials, which have many applications in optoelectronics ranging from solar cells⁵ to gas sensing.⁶ Nanostructures based on these materials have found numerous applications,⁷ and, due to their small size, knowledge of the depth dependence of defects is of particular importance. Practical device implementation is often limited by the nature of their intrinsic or extrinsic defects. In the present paper we examine the x-ray energy dependence of the

luminescence from commercially available single crystal CdS and CdSe in the range 600 to 1500 eV. In addition to band gap exciton structure we find peaks associated with defects at lower energies. The x-ray energy dependence of the luminescence peaks shows significant variation among the various channels. By using a simple “dead layer” model we are able to posit an explanation for these differences.

Experimental

Low resistivity, single crystal CdS(0001) and CdSe(0001) samples were obtained from MTI Corporation and used without any further processing. Although the doping type is unknown, it is almost certainly n-type as it is very difficult to make low resistivity p-type materials.⁸ The sample was mounted on a manipulator located in an ultrahigh-vacuum (UHV) end station. Measurements were performed at a temperature of 40 K. The XEOL experiments were performed using soft x-rays from beamline 4-ID-C at the Advanced Photon Source (APS). This facility provides intense, pulsed, tunable radiation in the range 500 to 3000 eV. The $\sim 0.2 \times 0.4 \text{ mm}^2$ x-ray beam irradiated the sample at an angle of 6 degrees with respect to the sample surface. This angle was chosen to maximize surface sensitivity, while still being sufficiently above the critical angle (2 degrees) so refraction effects are minimal. The emitted optical photons were extracted at 45° with respect to the x-ray beam using a condenser lens and then focused on the entrance slit of a 0.3-m monochromator. A cooled photomultiplier tube was used to detect the dispersed photons. Time-gated measurements were performed using the pulsed nature of the electron bunches in the APS, which provides $\sim 80 \text{ psec}$ x-ray pulses, spaced by 153 ns. Details of the method have been described previously.⁹

X-ray absorption spectroscopy (XAS) was performed by measuring the total-electron yield (TEY) from the sample. Since the x-ray flux varies with energy the TEY and luminescence signals were normalized to the electron yield from a gold mesh inserted into the beam.

Characterization

To characterize the samples XAS measurements were performed at the S K edge for CdS and the Se L edge for CdSe. Results are shown in Fig. 1. These spectra are similar to previously published results for these materials.¹⁰⁻¹¹ XAS measurements using TEY detection are surface sensitive (~top 10 nm). These results indicate that there is no significant anion oxidation in the surface region.

Time-gated luminescence spectra, taken with 900 eV x-rays are shown in Fig. 2. Ungated spectra, as well as those taken with short (0-10 ns) and long (20-120 ns) time gates are displayed. For CdS (Fig. 2(a)) there is a short lifetime, near band gap (NBG) peak located at 2.54 eV and longer lived defect structure found between 2.2 – 2.5 and 1.4 – 2.1 eV. These peaks have been assigned to exciton emission, donor-acceptor pair (DAP) transitions, and a vacancy-type, self-activated defect complex, respectively.¹² The short lived NBG emission for CdSe (Fig. 2(b)) lies at 1.82 eV and defect structure with a mixture of short and long lived components is evident between 1.4 – 1.77 eV. The high energy peak has been assigned to an exciton bound to an ionized donor, while the structure in the 1.6 – 1.77 eV region is due to donor-acceptor-pair (DAP) transitions and their phonon replicas.¹³ It is not clear as to the identity of the

lower energy (<1.6 eV) region, but structure in this region has been observed in ion irradiated materials and tentatively assigned to defects associated with Cd vacancies.¹⁴ The insets show measurements of lifetimes at selected energies along with fits to one or two exponentials. For both samples the lifetime of the NBG emission was too short to be measured, in agreement with prior work.¹⁵⁻¹⁶ For CdS the lifetime measured at 2.4 eV showed a single component with a value of 94 ns, while measurements taken at 1.62 eV were fit with a single, 38 ns component. These values are consistent with previous lifetime measurements.¹⁷⁻¹⁹ In the case of CdSe measurements made at 1.73 eV revealed a double exponential with fitted values of 6 and 54 ns. Note that for CdS the background level in the lifetime curve was $\sim 65\%$ while for CdSe it was $\sim 22\%$. Since the bunch spacing of the x-ray pulses is 153 ns these background levels may indicate the presence of very long lived components (>1000 ns).

X-ray energy dependent luminescence

Luminescence spectra taken at 600 eV and 1410 eV are shown in Fig. 3. The spectra are scaled so that the maximum intensity is 1 at both energies. For CdS (Fig. 3(a)) the main difference is that the defect structure between 1.4 and 2.1 eV becomes enhanced at the lower energy. More dramatic changes are seen in the case of CdSe (Fig. 3(b)). At 1410 eV the main structure is the NBG peak at 1.82 eV and broader structure between 1.5 – 1.78 eV. At 600 eV structure between 1.4 – 1.7 eV dominates the spectrum. To better characterize the evolution of these features, spectra were taken at 30 eV intervals, starting at 600 eV. The results are shown in the image plots in Fig. 4(a) for CdS and 4(b) for CdSe (note the scale difference between the left and right sides of

the images). For both samples, the lower energy, deeper-lying defects have an x-ray energy dependence showing a significant enhancement at lower x-ray energies as compared to the higher-energy luminescence.

Discussion

The x-ray energy dependence of the luminescent yields results from the material-dependent attenuation length of x-rays. In the absence of significant reabsorption of the emitted photon and reflectivity, the energy (depth) dependent yield, $\eta(E_x)$ is given by the following expression:

$$\eta = SQ(E_x/E_e)(1-\exp(-d/PL(E_x))) \quad (1)$$

where S is the energy transfer efficiency; Q is the quantum yield; $PL(E_x)$ is the angle and energy dependent x-ray penetration length; E_x/E_e is the number of electron-hole pairs created per x-ray photon, where E_x is the x-ray energy and E_e is the average energy required to create an e-h pair; and d is the material thickness.^{1, 20} For thick samples $d \gg PL(E_x)$ for all E_x and the exponential term vanishes, so

$$\eta = SQ(E_x/E_e) \quad (2)$$

and the yield should simply be proportional to E_x .

For semiconductors the effect of the depletion layer must be considered. A “dead layer” results because electron-hole pairs photoexcited in this region have a reduced density of carriers for radiative recombination, and thus an increased lifetime, enhancing the probability of nonradiative recombination.²¹⁻²³ An

additional factor stems from band bending which can cause bulk states to become depopulated in the depletion region.²⁴⁻²⁶ To account for the effects of the “dead layer” equation (2) is modified to:²⁷

$$\eta = SQ(E_x/E_e)\exp(-DL/PL(E_x)) \quad (3)$$

where DL is the width of the dead layer.

To utilize this model the individual components of the luminescence were extracted using curve fitting techniques. The data were modeled with the minimum number of Gaussian peaks that produced a reasonable fit throughout the entire x-ray energy range. For CdS 10 components were required while for CdSe the number was 8. Results of the fitting process for 900 eV x-rays are shown in Fig. 5(a) for CdS and Fig. 5(b) for CdSe. The energy-dependent yields were modeled using Eq. (3) and the results of the fitting procedure for the data are shown in Figures S1 and S2 and summarized in Tables 1 and 2. Since the data were normalized using the photoyield signal from a gold mesh a correction was made for its energy dependence using previously published results.²⁸ It is interesting to note that in the long (20 - 120 ns) time-gated spectra of CdSe in Fig. 2(b), the peak at 1.74 eV, along with some lower energy features, are enhanced. This may be due to the 1.74 eV peak having a relatively longer lifetime, which is mirrored in the phonon replica(s) at lower energy.

Inspection of Tables 1 and 2 show that the maximum dead layer thickness is ~120 nm for CdS and CdSe. In general the dead layer thickness is slightly less than the depletion width,²¹ which is unknown for these samples, but previous measurements

have shown to be ~300 nm for CdS and 200 nm for CdSe.²⁹⁻³⁰ Tables 1 and 2 reveal a general trend that the lower the energy of a transition, the shorter is its corresponding dead layer. Previously it has been shown for ZnO that band bending can cause depopulation of defect levels that exist in the flat band region, i.e. a V_O^+ level is converted to V_O^{++} in the depleted region.²⁴⁻²⁶ Analogous processes may be responsible for the dead layer thickness trend seen in Tables 1 and 2: states that are populated in the flat band region become depopulated as a result of band bending. The cause for the shorter dead layer thickness with decreasing emission energy can be seen by the schematic diagram in Fig. 6. Lower lying levels will cross the Fermi level and become depopulated at shorter distances from the surface. Thus, the higher energy level in Fig. 6 gives rise to a dead layer width DL_1 , while the lower energy level results in DL_2 . Note that the position of the levels shown in Fig. 6 does not necessarily correspond to the energy of the emitted luminescence; this will depend on the position of the lower lying states of the transition. Transition to the valence band will have the maximum energy, while transitions to higher energy, acceptor states will be lower. For instance, the 1.74 eV and 1.70 eV transitions in CdSe are very close in energy, yet their dead layer thicknesses differ by a factor of two. This could arise from a transitions akin to those designated 2 and 3 in Fig. 6, that originate from well separated upper states but make transitions to lower states separated by similar amounts. In contrast, the 1.93 eV and 1.51 eV transition have dead layer thicknesses that are fairly close, yet their energies differ substantially. This could arise from a situation similar to transitions 1 and 2 in Fig. 6 (neglecting any core interactions³¹). However, transitions from the conduction band to acceptor states will not follow this trend.

It is possible that band flattening could occur as a result of excess carriers being created by the exciting x-rays as has been observed in previous work utilizing electrons or lasers. However, the maximum flux of x-rays used in the present work only resulted in power densities of $\sim 10 \text{ mW/cm}^2$, so band flattening should not be a factor.

Examination of the fitted data in Figs. S1 and S2 reveals that some of the results match the simulation better than others. This is not surprising. Although every effort was made to effectively fit the spectral data shown in Fig. 5 with the optimum components, it is not certain that these components accurately depict the transitions leading to the luminescence. In addition the defect population in the surface region is often greater than the bulk,²⁻³ and it was not possible to include this factor in the modeling without making unwarranted assumptions. For CdS a broad transition centered at 1.7 eV has previously been identified with surface states.³² This could account for the poor agreement with the model for the 1.70 eV and 1.46 eV transitions in Table 1. Similarly in CdSe previous work has indicated that many deep levels are associated with surface states,³³ which would explain the poor modeling behavior for the levels at 1.64 eV and lower energies. Another possibility is that these states only become populated after conversion from states that were populated in the flat band region and who become ionized in the depletion region, as was observed in studies of ZnO.²⁵⁻²⁶

To summarize, we have measured the x-ray energy (depth) dependence of the luminescence from single crystal CdS and CdSe. The results were interpreted in terms

of a “dead layer” model and showed, in general, that lower energy transitions have shorter dead layers than higher energy ones. This may arise from the relative positions of the energy levels in the band gap, which, as a result of band bending, causes them to intercept the Fermi level at different distances from the surface.

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Table 1. Peak positions, FWHM, dead layer thicknesses, peak area percentages and tentative assignments^a for CdS

Peak Energy (eV)	2.54	2.42	2.39	2.35	2.34	2.31	1.93	1.70	1.51	1.46
FWHM (eV)	0.03	0.03	0.03	0.03	0.09	0.19	0.43	0.30	0.13	0.07
Dead layer (nm) ^b	98±3	108±7	111±6	121±7	123±6	126±8	88±9	-	60±8	-
% Area, 600 eV	1.2	11.6	13.6	3.4	16.4	7.5	12.0	26.1	5.5	2.7
% Area, 1200 eV	2.4	19.5	21.1	5.7	23.0	10.2	6.0	9.5	2.0	0.6
Assignments ³⁴⁻³⁷	E _x	DAP	LO	LO	DAP	I _s	I _{Cd}	V _{Cd}	V _{Cd}	V _{Cd}

^aE_x=band gap exciton; DAP=donor-acceptor pair; LO=longitudinal optical phonon loss replica; I=interstitial; V=vacancy. ^bThe dashes indicate that the fitting error was larger than the fitted value.

Table 2. Peak positions, FWHM, dead layer thicknesses, peak area percentages and tentative assignments^a for CdSe

Peak Energy (eV)	1.82	1.76	1.74	1.71	1.70	1.64	1.58	1.52
FWHM (eV)	0.02	0.06	0.02	0.03	0.06	0.08	0.08	0.12
Dead layer (nm) ^b	101±8	123±4	50±8	48±6	25±6	-	-	-
% Area, 600 eV	1.5	0.7	2.9	1.7	16.6	24.4	24.4	28.8
% Area, 1200 eV	17.5	9.1	17.5	10.7	27.7	2.5	2.5	12.9
Assignment ³⁸	E _x	?	DAP	LO	DAP	DL	DL	DL

^aE_x=band gap exciton; DAP=donor-acceptor pair; LO=longitudinal optical phonon loss replica; DL=deep level. ^bThe dashes indicate that the fitting error was larger than the fitted value.

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Figure Captions

Figure 1. (a) S K edge x-ray absorption spectrum from CdS. (b) Se L edge x-ray absorption spectrum from CdSe.

Figure 2. Ungated (bottom), 20 – 120 ns time gated (middle) and 0-10 ns time gated (top) luminescence spectra from (a) CdS and (b) CdSe. The insets show lifetime curves obtained at the energies displayed in the legend where the dots are the data and the solid lines are exponential fits.

Figure 3. Luminescence spectra obtained at 600 eV (red, solid line) and 1410 eV (black, dashed line): (a) CdS (b) CdSe.

Figure 4. Image plots displaying luminescence spectra as a function of x-ray energy for (a) CdS (b) CdSe. Note the difference in scaling between the left and right sides of the plots.

Figure 5. Results of curve fitting luminescence spectra taken at 900 eV x-ray energy for (a) CdS (b) CdSe.

Figure 6. Schematic energy level diagram for a n-type semiconductor with Fermi level, E_f , showing the effects of band bending on the conduction (E_c) and valence (E_v) bands, as well as three defect levels shown by dotted (red), dashed (blue) and dot-dash (purple) lines. DW is the depletion width. Where the upper two defect levels cross the

Fermi level gives rise to dead layers with widths DL_1 and DL_2 . Also shown are three different transitions: 1 and 3 go from two different donor levels to the valence band and 2 is donor-acceptor pair transition.

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Figure 1

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Figure 2

Figure 3

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Graphical Abstract

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