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Photoconductivity relaxation in $ZnIn_2Se_4$

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The photoconductivity relaxation process in $ZnIn_2Se_4$ is studied in the 80–300-K temperature interval, together with optical quenching and thermally stimulated current. In order to study thermal emission, the four-gate technique of photoinduced-current transient spectroscopy (PICTS) is applied to photoconductivity decays. PICTS and thermocurrent results allow us to exclude the typical thermal emission mechanism from single-trap levels. Photoconductivity relaxation is interpreted on the basis of a nonlinear two-channel recombination process: in the 80–170-K range radiative recombination with bimolecular kinetics predominates, while for $T > 170$ K transients show a power-law behavior due to a nonradiative-recombination path. By applying concepts commonly used in dispersive transport theory, an exponential "trap" distribution connecting the nonradiative center with free carriers is deduced. Experimental results allow us to propose a general scheme for the levels and the transitions characterizing photoconductivity relaxation. The general photoconductivity trend also observed in other compounds of the AB_2X_4 family may be related to the intrinsic nature of the involved defects.

I. INTRODUCTION

$ZnIn_2Se_4$ is a ternary semiconductor belonging to the $A^{II}B_2^{III}X_4^{VI}$ defect chalcopyrite family which, in recent years, has received growing interest because of its intrinsic disorder revealed by studies on optical, transport, photoconductive, and memory-effect properties.[1,2] Disorder, indeed, causes the appearance of a large number of electronic levels in the energy gap and is commonly attributed to some exchange in the A and B ions at the sites of the cationic sublattice.[3] Electronic defects may be divided into two groups.

(a) Shallow acceptor and donor levels characterizing the transport properties. In spite of the large number of these levels, low electrical conductivity is often observed due to strong compensation between acceptor and donor centers. This type of defect frequently produces localized gap tail states commonly observed in amorphous semiconductors.

(b) Deep levels which largely determine the photoconductivity and luminescence properties, as well as contribute to charge storage and negative resistance.

It has been found[2,4,5] that different compounds of the AB_2X_4 family show a common behavior in kinetics governing the luminescence and photoconductivity processes. In general, two channels of recombination—one radiative, the other nonradiative—are present. They are in competition with each other; moreover, the recombination process is controlled by shallow levels distributed in energy that act as intermediate centers in either of the two recombination channels, rather than as trapping centers.

Photoconductivity (PC) processes seem the most suitable technique for obtaining information about the states in the gap. In fact, both radiative and nonradiative transitions are important in nonequilibrium photoconduction; whereas, luminescence involves only radiative transitions. On the other hand, the normal role played by shallow levels in PC, specifically thermal emission, can be studied by means of the new technique of photocurrent transient analysis, called photoinduced-current transient spectroscopy (PICTS), which has recently been used to characterize semiconductors.[6,7] PICTS, although commonly limited to photoconductive processes governed by thermal emission, may also furnish information on the whole relaxation process.

The purpose of this work is to determine if in $ZnIn_2Se_4$ the relaxation process is ruled by kinetics similar to those observed in other members of the AB_2X_4 family. The study of the PC properties, together with the knowledge of the luminescence features, can provide the complete picture of the defect levels taking part in the relaxation process.

II. EXPERIMENTAL PROCEDURE

Monocrystals of $ZnIn_2Se_4$, grown by the method of vapor-phase chemical transport using iodine as the carrier, are small prismatic columns $3\times1\times0.2$ mm^3 in size with one good natural face in the $[1\bar{1}2]$ direction. Electrical contacts in a coplanar configuration were obtained by vacuum deposition of indium followed by heating briefly at 180 °C in a nitrogen atmosphere. The contacts were ohmic in a wide temperature range (18–300 K). Dark resistivity was typically 10^6 and 10^8 Ω cm, at 300 and 80 K, respectively. All the samples were n type.

For the PC spectral response, a quartz-iodine lamp together with a McPherson 0.3-m grating monochromator set for a resolution of 0.5 nm was used as light source and for dc detection a 414 S Keitley picoammeter was employed. The optical quenching spectrum was obtained using a He-Ne 5-mW laser as a primary source and the quartz-iodine lamp coupled with the McPherson monochromator as secondary source. All the PC spectra were normalized to the blackbody response of the optical system.

Thermally stimulated current measurements were achieved by exciting the sample, kept at 80 K, for 15 min with He-Ne laser (intrinsic excitation). Subsequently the dark current flowing in the sample was measured, while temperature was linearly raised (about 0.5 $K\,s^{-1}$).

Photocurrent transients excited by the He-Ne laser were obtained by means of an electronic shutter (Computer Electronic M) using a 200-μs closing time for the laser beam. The voltage drop across a low resistor in a series with the sample and a low voltage supply (9 V), was amplified by means of a PAR 113 dc operated amplifier. The amplifier output was fed to a digital acquisition system (2005-S LeCroy Century system, maximum frequency 5 $Msample\,s^{-1}$) coupled with an IBM PC/XT3 computer. The computer operated the shutter opening for a time long enough to reach the PC signal saturation, then the shutter was closed and the PC decay was acquired with a sampling frequency properly set. The entire decay was recorded at various temperatures (about every 2 K) during a very slow thermal scan and the results were stored on a hard disk with all the parameters of the measurements for the subsequent analysis.

III. RESULTS

A. PC spectra

The PC spectral responses at 300, 80, and 18 K are shown in Fig. 1. In order to interpret these spectra it is worthwhile to remark that $ZnIn_2Se_4$ is considered by most authors[8,9] to be a direct-gap semiconductor with an energy gap of 1.83 and 1.93 eV, at 300 and 80 K, respectively. Furthermore, Mekhtief *et al.*[9] recently were able to show some structures at 1.93, 2.02, and 2.08 eV in the PC spectrum obtained at 80 K with polarized light. These structures have been attributed to interband transitions from the split off Γ_{15} sphalerite level to the conduction band, similar to what has been seen in other compounds with thiogallate structure, such as $CdIn_2Se_4$ (Ref. 10) and $CdGa_2Se_4$.[11]

Bearing these remarks in mind, we can attribute the PC peak at 1.68 eV in the 300-K spectrum to extrinsic photoexcitation from an acceptor level to the conduction band. This impurity peak dominates the PC spectrum, due to the high density of the acceptor centers that are ionized at 300 K. The well-defined structure centered at about 1.80 eV can be attributed to interband transitions and is connected to the gap energy (1.83 eV).

The 80-K spectrum is dominated by a step rise starting at 1.87 eV. The energy at half-height of the step rise is 2.01 eV, while at the top it is 2.10 eV. There is good agreement with the energies of the structures observed by Mekhtief *et al.*[9] As may be seen from the inset in Fig. 1, on the low-energy side of the 80-K spectrum, two structures at 1.87 and 1.80 eV are present. By shadowing these structures with raising temperature, it was possible to correlate the 1.80-eV structure to the extrinsic peak at 300 K and the 1.87-eV one to intrinsic photoexcitation. We can then conclude that extrinsic photoconductivity is still present at 80 K, but with a much lower intensity than at 300 K. The presence of a nearly unionized acceptor at 80 K accounts for this strong lowering of the impurity PC peak. From the low-energy tail of the 1.80-eV structure, the acceptor level can be located at 1.68 eV below the bottom of the conduction band.

The PC spectrum at 18 K has features similar to those observed at 80 K, with a low-energy tail of the step rise at 1.93 eV. The optical quenching spectrum at 80 K is shown in Fig. 2. No optical quenching was observed for $T > 150$ K. The quenching signal is defined by the relation

$$Q = (I_P - I_S)/I_0 \, , \tag{1}$$

where I_P is the photocurrent produced by the primary light, I_S that due to both the primary and secondary light, and I_0 is a normalization function which takes into account the spectral response of the optical system. As primary light the 633-nm line of the He-Ne laser was used in order to produce intrinsic excitation of the PC. The laser intensity was reduced to 0.01% to match the secondary light intensity.

As may be seen, quenching starts at 1.68 eV and rapidly rises as the secondary light energy is decreased. For $E > 0.9$ eV quenching decreases almost linearly with energy, the extrapolation energy at zero quenching being 0.67 eV.

It is known[12] that optical quenching can be interpreted

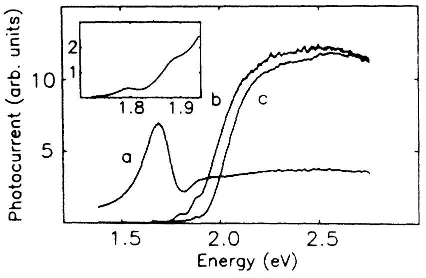

FIG. 1. Photoconductivity spectral response at (a) 300 K, (b) 80 K, and (c) 18 K. The inset shows a detail of the 80-K spectrum.

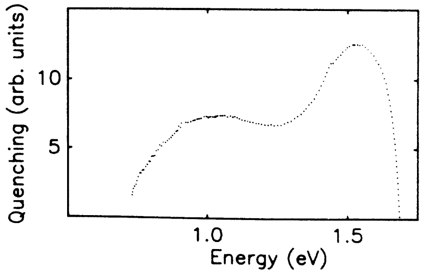

FIG. 2. Photoconductivity optical quenching at 80 K as defined by Eq. (1). Primary excitation was performed through the 633-nm laser line.

as an indication of the presence of two competing recombination centers, class-I (fast) centers and class-II (slow) centers in Rose's model,[13] characterized by two different capture cross sections for majority carriers, electrons in our case. From the quenching spectrum we can derive information about the energy location of the slow center: we obtain 0.67 eV above the valence band. It must be pointed out, however, that the features of the quenching spectrum may suggest a competing process at 1.20 eV (relative quenching minimum).

B. PC transients

In the 80–300 K interval the PC response times range from 0.8 to 5 ms. In Figs. 3 and 4 some PC decays at various temperatures are reported on $\ln(I)$ versus $\ln(t)$ and I^{-1} versus t scales, respectively. Above 180 K decays seem to follow a power law of the type

$$I(t) \propto t^{-\beta} , \tag{2}$$

whereas below 180 K they seem to match a law of the type

$$[I(t)]^{-1} = A_0 + Bt . \tag{3}$$

Exponential behavior is poor in the entire temperature range we examined. The PC saturation signal as a function of temperature evidences a thermal quenching process starting at about 170 K, whose activation energy is 100 meV.

In order to study the thermally activated process involved in the PC decays, we applied the recently developed PICTS technique[7] which can be considered as derived from the well-known DLTS (deep-level transient spectroscopy).[14] Because of the complicated nonexponential relaxation process, we found it convenient to apply the four-gate method of PICTS, which consists in plotting the ratio

$$Y(T) = \frac{I(t_1) - I(t_2)}{I(t_0) - I(t_3)} \tag{4}$$

as a function of temperature, where $I(t)$ is the amplitude of the PC signal at temperature T and at instant t after the shutter has been closed; t_0, t_1, t_2, and t_3 are four reading times appropriately chosen. If we assume a purely exponential decay with a time constant $\tau(T)$ for the thermal emptying of the carrier traps (single-trap case), it can be shown[7] that $Y(T)$ goes through a maximum at a temperature T_m, provided that $t_3 > t_2 > t_1 > t_0$. Assuming $t_3 \gg t_2$, $I(t_3)$turns into the dark current and the time constant at T_m is related in a simple manner to the reading times.[7] Of course, the knowledge of the response time $\tau(T)$ at various temperatures can be used to obtain the physical parameters of the trapping center (Arrhenius plot). The height of the peak in the $Y(T)$ spectrum only depends on the choice of t_0, t_1, and t_2. So, if we assume $t_1 = 2t_0$, $t_2 = 3t_0$, its value is expected to reach 0.25, irrespective of the t_0 value. This can be used as a direct check of the postulated physical model of the relaxation process.

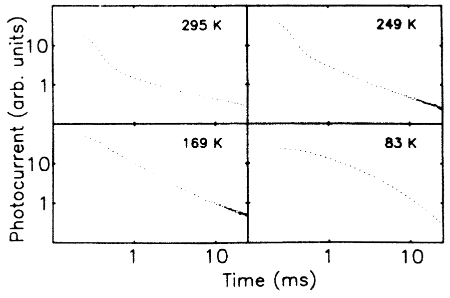

FIG. 3. Photoconductivity decays at different temperatures on logarithmic scales.

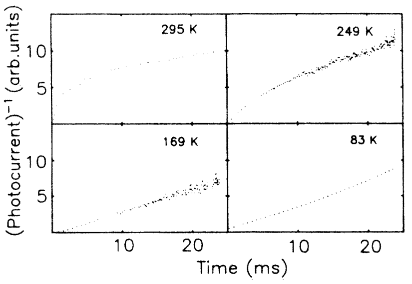

FIG. 4. Inverse photoconductivity decays at different temperatures as a function of time.

Here, the following points are to be remarked as general features of the PICTS technique.

(1) Any information about the initial trapped carrier density is lost by the four-gate method.

(2) The PICTS furnishes the complete spectrum of the various trapping levels active in a given temperature range. In particular, if the trapping levels are continuously distributed within the forbidden energy gap, a broad flat spectrum is obtained from the four-gate method, irrespective of the type of distribution, with an amplitude that may fall considerably below the 0.25 expected value.

In Fig. 5 we show a few PICTS spectra obtained in the case $t_2 = 3t_0$ and $t_1 = 2t_0$. Two broad peaks centered at

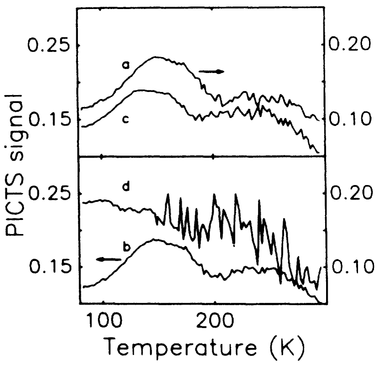

FIG. 5. PICTS spectra for different t_0 values: (a) 0.3 ms, (b) 0.4 ms, (c) 0.7 ms, (d) 5 ms. Note the different right-hand and left-hand scales.

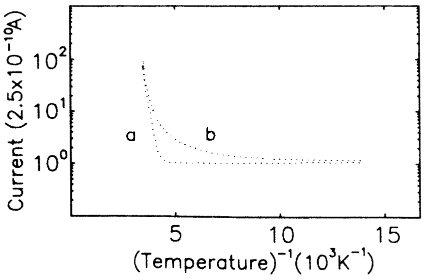

FIG. 6. Dark current as function of T^{-1}: (a) without light excitation, and (b) after excitation with the 633-nm laser line.

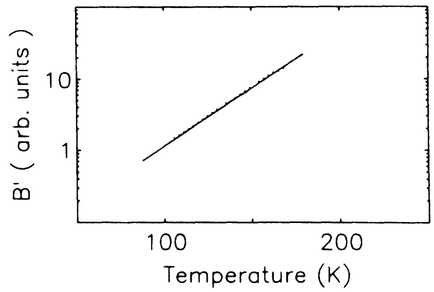

FIG. 8. Plot of B' [see Eqs. (7) and (8)] vs temperature as obtained from photoconductivity decays.

about 160 and 240 K are obtained. The peak amplitude is nearly constant as t_0 is varied, but its value is lower than the expected one for purely exponential decays. Furthermore, for large t_0, the PICTS spectrum is very broad with a flat response. Of course, such PICTS spectra did not allow us to obtain an Arrhenius plot. Therefore, from the analysis of the PICTS spectra we can conclude that in the explored time interval no thermal emission from a well-defined single trap takes place in the relaxation process. The flat response for large t_0 may be attributed to traps continuously distributed in energy within the gap.

As a further check we performed some measurements of thermally stimulated current. In Fig. 6 we show both the dark conductivity and the thermal stimulated conductivity obtained at 80 K with the 633-nm laser line. As may be seen, light excitation produces very little change in the dark conductivity, confirming that the thermal emission model for the PC relaxation is to be excluded. Dark conductivity shows evidence of a single deep-trap level lying at 0.50 eV below the bottom of the conduction band.

It is known[15,16] that in many amorphous semiconductors PC decay follows a power law such as (2) with the β exponent linearly dependent on temperature. This behavior has been interpreted as due to dispersive transport arising from traps distributed in energy or in space. In order to gain information about the relaxation kinetics, we analyzed the temperature dependence of the β exponent in the 170–300-K interval. As may be seen in Fig. 7, β changes linearly with temperature according to

$$\beta=\beta_0-\beta' T \, , \tag{5}$$

where $\beta_0=1.96$ and $\beta'=5.3\times10^{-3}\ K^{-1}$.

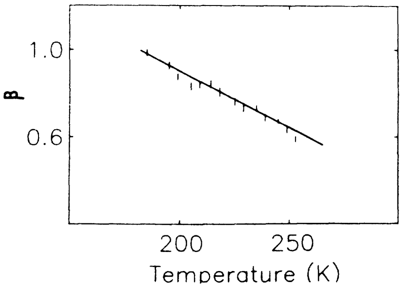

FIG. 7. Plot of the exponent β [see Eq. (2)] vs temperature as obtained from photoconductivity decays.

As is the case in low-temperature decays, it is to be pointed out that behavior of the type of (3) is readily connected with a bimolecular recombination process; that is,

$$\frac{dn}{dt}=-v\sigma_r mn \, , \tag{6}$$

where n and m are the densities of electrons in the conduction band and of holes in the recombination center, respectively; σ_r is the capture cross section of the recombination center for free electrons. Assuming $m=n$, Eq. (6) yields, by integration,

$$i(t)=\frac{I(t)}{I(0)}=\frac{n(t)}{n_0}=(1+B't)^{-1} \, , \tag{7}$$

$$B'=v\sigma_r n_0 \, , \tag{8}$$

where n_0 is the free-electron density at the beginning of decay. B' has been obtained on the basis of (7) by applying the covariance method[17] to the first 50 ms of the decay (that is 1000 digitalized values). Correlation coefficients greater than 0.99 proved the reliability of (7).

B' values are plotted in Fig. 8 versus T. In the hypothesis $\sigma_r=$const, the initial density n_0 of free electrons changes exponentially in the 80–170-K interval, according to a function of the form

$$n_0(T)\propto\exp(\alpha_0 T) \, , \tag{9}$$

with $\alpha_0=3.5\times10^{-2}\ K^{-1}$.

Finally, we should notice that the PICTS spectra are consistent with our interpretation of PC decays for $T<170$ K. Assuming, in fact, a decay of the type of (7), with B' deduced from Eqs. (8) and (9), simulated PICTS spectra according qualitatively with the experimental ones were obtained for $\alpha_0=3.5\times10^{-2}\ K^{-1}$.

IV. DISCUSSION

From our results the following picture can be deduced for the energy levels within the energy gap of $ZnIn_2Se_4$.

(1) An acceptor level, ionized at room temperature, lying at 0.25 eV above the top of the valence band.

(2) A "sensitizing" center (class II of Rose's model) located at 0.67 eV above the top of the valence band.

(3) A "desensitizing" center (class I in Rose's model). The energy location of this center can be deduced by comparing PC and luminescence[18] properties, i.e., the same temperature is observed for the onset of both PC and luminescence thermal quenching and with activation energies of the same order. In addition, optical stimulation of luminescence by means of a secondary light excitation at 1.20 eV appears to be complementary to the optical quenching of PC that at the same energy shows a minimum. On the basis of these observations we can conclude that radiative recombination is to be connected with the sensitizing center of PC. Then it is possible to locate the desensitizing center at 1.20 eV above the valence band, corresponding to the maximum of the luminescence stimulation.

(4) A set of levels distributed in energy, that act very likely as traps on the basis of the PICTS analysis.

Furthermore, the observed decay features indicate that the PC relaxation process follows two different kinetics at low and high temperatures. This suggests that in the low-temperature range, PC relaxation is dominated by a radiative process (slow channel), whereas in the high-temperature range nonradiative recombination (fast channel) predominates. In any case the trap distribution must play an important role.

We first turn our attention to the kinetics of high-temperature decays. Bearing in mind the great amount of intrinsic disorder showed by the AB_2X_4 ternary compounds, we shall apply some concepts commonly used in dispersive transport theory to the PC relaxation of $ZnIn_2Se_4$ in the high-temperature range. We can write, for the fraction of photogenerated electrons in the conduction band,[16]

$$\frac{n}{N_0}=\frac{g(0)}{g(E_d)}\exp(-E_d/kT) , \tag{10}$$

where n and N_0 are the densities of free electrons and of photogenerated electrons, respectively; E_d is the energy of a demarcation level, $g(0)$ and $g(E_d)$ the densities of the trapping states, respectively, at $E=0$ (the bottom of the conduction band) and at $E=E_d$. The demarcation level energy is a function of the relaxation time according to the relation

$$E_d=kT\ln(\nu_0 t) , \tag{11}$$

where ν_0 is a constant of the order $10^{12}-10^{13}\ \mathrm{s}^{-1}$. Namely, we suppose that the distribution of trapped electrons does not follow a quasi-Fermi statistic of steady state, but is dominated both by thermal emission through Boltzmann's factor and by multiple retrapping. For $E<E_d$, trapped electrons are in thermal equilibrium with the conduction band.

If we assume an exponential distribution of traps

$$g(E)=A\exp(-\alpha E) , \tag{12}$$

we obtain, from (10) and (11),

$$n=N_0\frac{g(0)}{A}[\nu_0 t]^{(-1+\alpha kT)} . \tag{13}$$

It is to be pointed out, however, that in our case traps can be directly connected to nonradiative-recombination centers. Besides this, the demarcation energy E_d can also be defined[19] as the energy for which the thermal emission has the same probability as the nonradiative recombination. This requires to introduce a further Boltzmann's factor in (13). Then we have

$$n=N_0\frac{g(0)}{A}[\nu_0 t]^{(-2+\alpha kT)} . \tag{14}$$

This expression fits very well the experimental decays in the 170–300-K range. The value $\alpha=56\ \mathrm{eV}^{-1}$ deduced from Fig. 7 is in good agreement with that reported by Manca *et al.*[20]

As to the PC decays in the low-temperature range, we observe that in this case relaxation is dominated by the radiative recombination (sensitizing center). Relaxation is ruled by the number n_0 of free electrons at the start of the decay [see Eqs. (7) and (8)]. Assuming n_0 to be directly proportional to the density of states at $E=0$ and inversely proportional to the density of trapping states at the quasi-Fermi level energy E_L during excitation, we have

$$\frac{n_0}{N_0}\propto\frac{g(0)}{g(E_L)} . \tag{15}$$

Assuming the energy E_L depending linearly on temperature as the Fermi level energy, we can set[12]

$$E_L=kTr+E_0 , \tag{16}$$

and

$$r=\ln\left[\frac{N_A}{N_D-N_A}\right] , \tag{17}$$

where E_0 is a constant and N_A and N_D are the acceptor and donor densities, respectively. Then, Eq. (15) becomes, assuming the exponential trap distribution of (12),

$$n_0\propto N_0[g(0)/A]\exp(\alpha rkT+\alpha E_0) . \tag{18}$$

This expression of n_0 accounts for the experimental feature shown in Fig. 8. Assuming $\alpha=56\ \mathrm{eV}^{-1}$, as deduced from high-temperature decays, we obtain from (9) and (18) $r=7.3$, that is, the relative autocompensation grade of our material results

$$\frac{N_D-N_A}{N_A}=7\times10^{-4} .$$

V. CONCLUSIONS

Figure 9 shows a summary of the levels and the transitions accounting for the PC relaxation in $ZnIn_2Se_4$. The scheme outlines the picture at 80 K. Intrinsic PC is induced by transition 1, while transition 2 from the A acceptor level to conduction band causes extrinsic PC. Extrinsic peak features in the 80- and 300-K PC spectra give evidence that the A center is to be considered almost unionized at 80 K and completely ionized at room temperature. The L center is responsible for the luminescence emission (transition 3) and is to be regarded as the

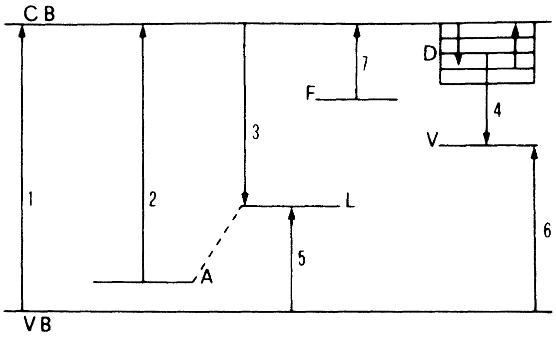

FIG. 9. Scheme of the levels and the transitions involved in the photoconductivity relaxation of $ZnIn_2Se_4$.

sensitizing center for the PC. At this point it is to be outlined that memory effect in $ZnIn_2Se_4$ has been attributed[21] to a recombination center showing a repulsive potential barrier for free electrons. Therefore, we attribute a negative charge to the L center, according to its sensitization effect on the PC. Transition 5, exciting electrons from the valence band to the L center, yields a double negatively charged center corresponding to the A acceptor level. This transition rules the sensitizing center out of the recombination traffic, so accounting for the optical quenching of the PC. Transition 5 can also be thermally activated, giving rise to thermal quenching of both the PC and luminescence. The large difference between optical and thermal activation energies may be associated with the local Coulomb barrier at the charged center.

Optical stimulation of luminescence is produced by transition 6 which lifts electrons from the top of the valence band to the desensitizing center V. This transition accounts for the minimum observed at 1.20 eV in the optical quenching spectrum of the PC. The V center is connected with the distribution of donor levels D through transition 4. This hypothesis is derived from our interpretation of the PC decays for $T > 170$ K; specifically, the PC relaxation arises both from thermalization of the electrons in D levels and from the recombination path 4. For $T < 170$ K decays are dominated by the radiative recombination. The D levels in this case act as centers limiting the free-electron density. Bimolecular kinetics, observed for both the PC and luminescence decays, require that D levels collect as many photogenerated electrons as holes present in the V center. This interpretation agrees with the fact that we failed to observe thermally stimulated current. Finally, transition 7 indicates thermal emission from a deep donor level F which does not seem to take part in the PC relaxation process for $T < 250$ K.

As a concluding remark, we point out that the PC relaxation in $ZnIn_2Se_4$ shows some features that are common to other members of the AB_2X_4 family, such as $CdIn_2S_4$ (Ref. 4) and $ZnIn_2S_4$;[5] i.e., two recombination paths, one radiative, the other nonradiative, which compete and donor levels distributed in energy which are directly connected with one recombination center. Furthermore, similar common features are also observed in the photoluminescence of AB_2X_4 semiconductors. As already proposed by Krausbauer *et al.*,[22] and by Guzzi and Grilli,[2] the general trend of the extrinsic properties can be attributed to the intrinsic nature of the defects in these materials.

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