Three-center Auger-type nonradiative recombination mechanism in the ZnS lattice

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The occurrence of an Auger-type energy-transfer process from a recombining donor-acceptor pair to a third center (donor or acceptor) is proved directly using the electron-spin-resonance technique. The efficiency of this process is estimated by analyzing the kinetics of the donor ESR signal. The conditions under which the three-center Auger-type energy transfer processes would efficiently quench photoluminescence intensity are discussed.

I. INTRODUCTION

Donor-acceptor pair (DAP) recombination, i.e., radiative recombination of an electron bound on a donor with a hole bound on an acceptor, dominates the visible photoluminescence (PL) of ZnS. The first step in the excitation mechanism of the DAP PL is population of an acceptor due either to its direct ionization or to capture of a hole generated in the valence band (VB) by band-band excitation. An electron induced in the conduction band (CB) by either type of illumination can then be trapped by a donor and, if a populated-donor–populated-acceptor pair is not distant, DAP recombination will follow. This recombination does not necessarily result in visible emission due to a possible energy transfer from the DAP to another impurity (defect).

Among the different energy-transfer processes, the so-called Auger effect is often the most important one. An Auger effect is a cooperative interaction of three carriers (two electrons and a hole or two holes and an electron). In this process the energy of a recombining electron-hole pair is transferred to a third carrier which, in consequence, is excited high into the continuum of CB or VB states. The three carriers participating in the Auger process can be either free or bound at one, two, or three different centers. For example, it is well known that the Auger effect is responsible for the very efficient nonradiative recombination of excitons bound at neutral donors or acceptors[1,2] [Fig. 1(a)]. Similarly, this process was found to reduce the intrashell emission of transition-metal (TM)[3] and rare-earth (RE)[4] impurities [Figs. 1(b) and 1(c)]. The role of the various Auger effects in the nonradiative recombination processes of DAP's has remained, however, practically unknown. The particular case of the three carriers involved in the Auger process (i.e., an electron, a hole, and a recoil particle—either electron or hole) being bound at three different centers has recently been discussed by one of the authors.[5] In the analyzed case, the DAP could decay nonradiatively due to energy transfer to a deep center (iron, chromium), causing its ionization [Fig. 1(d)]. It has been proved that such a three-center Auger recombination (TCAR) mechanism offers a channel competitive to the radiative DAP recombination for samples intentionally doped with TM's. For crystals only contaminated with TM's, or other impurities introducing midgap levels in ZnS, the efficiency of TCAR has been estimated to be too low to significantly affect the PL intensity.

In this paper, we present the first estimation of the efficiency of another TCAR channel. We analyze the case of an Auger process in which the energy of the recombining DAP is transferred to a shallow impurity, i.e., to a donor similar to that involved in DAP transitions. Notwithstanding the fact that such a TCAR process is expected to be less efficient than the one in which a deep center is ionized (as it has been found for the case of Auger recombination of excitons bound at isoelectronic centers[6]), it may significantly reduce the ZnS visible PL owing to the higher concentration of shallow centers involved in this process, since they are often intentionally introduced to get a better luminophor.

This paper is organized in the following way. In Sec. II, the experimental setup is explained and the results obtained are presented. Some details concerning the experimental method employed are also given because of its originality. Since there are several other nonradiative recombination mechanisms leading to PL quenching, we have applied the photo-ESR technique, which has allowed us to separate the contribution due solely to the TCAR process discussed. This could not be achieved by any of the standard optical methods.

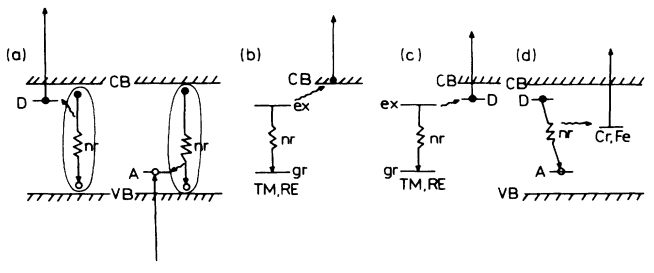

FIG. 1. Auger-type nonradiative (nr) recombination of (a) excitons bound at neutral donors or acceptors; (b),(c) transition metals or rare-earth ions; and (d) a donor-acceptor pair (three-center Auger recombination). ex denotes the excited state, gr the ground state.

In Sec. III, a simple analysis of the experimental results is given and thus, for the first time, a comparison of the efficiencies of radiative DAP recombination and TCAR is made. This has also allowed us to deduce the role played by the TCAR process discussed in the overall efficiency of the ZnS visible PL (Sec. IV).

II. EXPERIMENT

The ESR experiments have been performed on a standard Bruker B-ER 418s spectrometer equipped with a simple optical set for *in-situ* sample illumination, consisting of a high-pressure mercury lamp, a set of interference filters, and quartz lenses. The samples have been mounted in an Oxford Instruments gas-flow cryostat working in the temperature range of 4–300 K.

The optical measurements have been performed on a standard setup consisting of a Carl-Zeiss Jena ILA-120 4-W argon laser, a GDM-1000 double-grating monochromator, an Oxford Instruments CF-100 gas-flow cryostat, a Hamamatsu R636 with a GaAs photocathode, and an Ithaco 393 lock-in amplifier.

The experiments have been carried out on compensated, high-resistivity n-type ZnS samples. The crystals were grown by the iodine transport method and then annealed in molten Zn:Al (2.5 wt. % Al) at 1000 °C for 70 h. Although not intentionally doped with chromium, a small Cr contamination was observed, which is typical of all ZnS crystals.

In these samples, an ESR signal of an unidentified donor has been observed ($g = 1.8844$) "in the dark," i.e., without prior illumination. The occupancy of this donor state has remained practically constant up to room temperature, as deduced from the temperature dependence of the ESR signal intensity. The uv illumination, which generates electrons in the CB, has induced only a slight increase in the intensity of this signal. These results mean that we are dealing with a relatively deep donor with an ionization energy of at least 100 meV, and with the Fermi level close to the energy level of this donor. The same uv excitation has also induced the well-known blue self-activated and green Cu-related emissions with a maximum at 2.62 eV (21 125 cm^{-1}) at 3.5 K, shown in Fig. 2.

The observation of a photosensitive ESR signal of a donor allows us to perform an experiment in which the influence of the DAP recombination on the depopulation of this donor can be directly measured. This donor can be depopulated either in a direct DAP process by recombining with a hole on an acceptor or due to the Auger-type process shown in Fig. 3(a). In the latter case, it is ionized owing to the energy transfer from a recombining DAP involving another donor. During uv excitation both processes are obscured by the capture of electrons from the CB. This is observed as a small rise in the intensity of the donor ESR signal (see Fig. 4). Therefore, we cannot conclude on the role of the DAP and Auger-type recombination processes from these data. This is also not possible from standard PL measurements, which explains the lack of the relevant information. The strong thermoluminescence (TL) observed in our samples enables us, however, to exclude the capture and DAP recombination

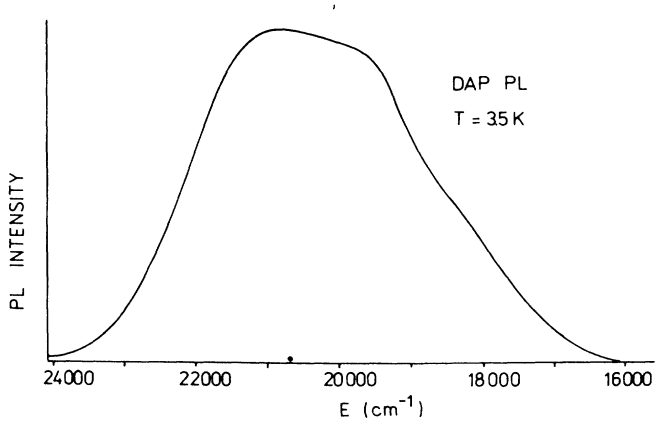

FIG. 2. Photoluminescence spectrum of ZnS crystals annealed in Zn,Al.

channels and to study solely the depopulation of the donor due to the Auger mechanism. We have established first the role played by different donors in the electron-capture and DAP recombination processes. We have found[7] that both these processes for 110–160 K are dominated by a 35-meV donor and that the deep donor observed in ESR plays a fairly unimportant role. This means that in the TL, electrons thermally ionized from one shallow donor are recaptured by another one and, if it is close to the populated acceptor, the DAP transition will follow. Consequently, deep donors do not play any significant role in this process. Therefore, any depopulation of the deep donor, measured as a decrease in the intensity of the relevant ESR signal, is practically due to some indirect processes. To prove that it is due to the Auger-type energy-transfer mechanism [Fig. 3(a)], we should show that the depopulation of the deep donor is thermally stimulated with the activation energy equal to that found in the TL data. The latter comes from the fact that in the case of the energy-transfer mechanism shown in Fig. 3(a) there should be a direct correlation between DAP and Auger recombination efficiencies.

The experiment is done in the following way. The uv excitation is used to populate donor and acceptor centers in the sample. Then, the light is turned off at low temper-

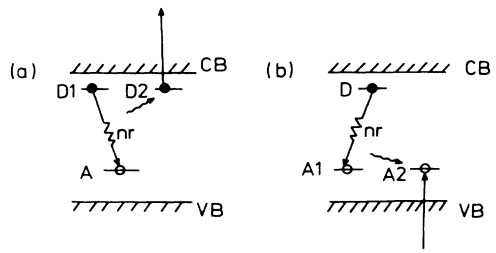

FIG. 3. The model of three-center Auger recombination processes involving a donor-acceptor pair and another (a) donor or (b) acceptor.

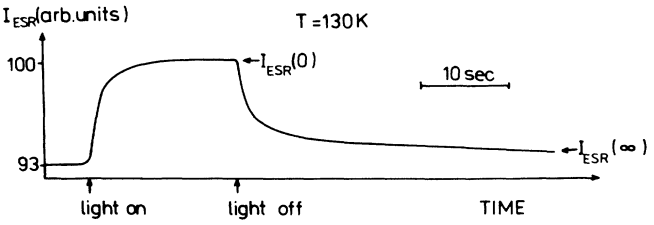

FIG. 4. The sequence of steps in the photo-ESR experiment. It shows the kinetics of the ESR signal intensity of the $g=1.8844$ donor under uv illumination.

ature (~ 100 K). Under such conditions the DAP recombination can be induced thermally (TL). The kinetics of the ESR signal of the donor ($g=1.8844$) has been measured after the uv excitation was turned on and then when it was turned off. These measurements were performed in the temperature range of 110–160 K, in which strong blue–green TL has been observed, characterized by a 35-meV activation energy.

The sequence of steps in the photo-ESR experiment is shown in Fig. 4. For each temperature studied, we have monitored first the small increase (light on) and the subsequent decrease (light off) of the donor ESR signal. The initial drop of the ESR signal intensity after the light was turned off consisted of two easily separable exponential components, characterized by different temperature dependences. The temperature dependences of the decrease rates for the "slow" and "fast" components of the decay are shown in Fig. 5(a). They yield activation energies of 35±8 meV and 85±12 meV, respectively. These energies are equal, within the experimental error, to the thermal ionization energies of two shallower donors

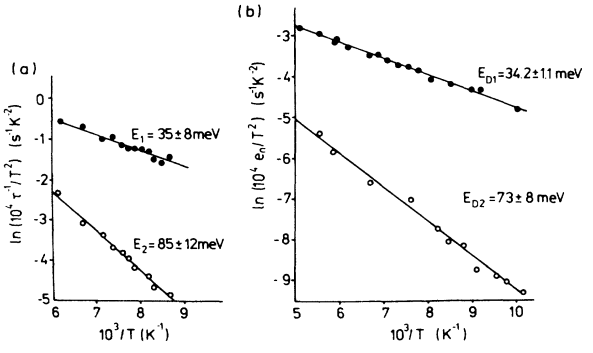

FIG. 5. (a) Temperature dependence of the decrease rates (τ^{-1}) of the donor ESR signal. The data were obtained from the initial decay of this signal after the uv light was turned off. This part of the kinetics consists of two exponential components: a fast one (closed circles) and a slow one (open circles). (b) Temperature dependence of the electron emission rates (e_n) from 35-meV (●) and Al (○) donor centers. These data were obtained by measuring the kinetics of the Cr^+ ESR signal rise (chromium recaptures electrons thermally ionized from these donors).

present in the crystal studied, i.e., the 35-meV native donor of ZnS and the 75-meV donor, probably related to aluminum [see Fig. 5(b)]. These two donor species, although not directly observed in ESR, could be identified indirectly by observing the electron redistribution between chromium and the donors in a process induced by $h\nu > 1.3$ eV illumination. The idea of the appropriate experiment has been explained in detail in previous papers.[7–9] The relative occupancies of these two donors (n_D^1, n_D^2) were deduced from the ratio of "slow" to "fast" components in the rise of the Cr^+ESR signal (the electrons thermally ionized from the donors are recaptured by chromium) to be almost equal [$n_D^1(0)/n_D^2(0)=1\pm0.1$]. Their absolute concentrations have remained unknown, but we estimate the number of them populated metastably by prior uv light to be in the range of 10^{14}–10^{15} cm^{-3}. This information was obtained indirectly, by measuring the number of occupied acceptors (equal to the concentration of the occupied shallower donors). We have utilized in this the fact that the Cr^+ state has a much larger hole-capture cross section than the ZnS acceptors. This means that the majority of the holes ionized to the VB from acceptors by secondary infrared illumination are recaptured by chromium. Therefore, by measuring the efficiency of the light-induced quenching of the Cr^+ ESR signal, we can estimate the acceptor occupancy and hence the concentration of the occupied donors. The details of the applied experimental procedure can be found elsewhere.[8]

III. DISCUSSION

A. Analysis of the experimental results

The observation of the ESR signal of such a donor center, characterized by a small electron-capture cross section and slow DAP recombination, enables us to estimate directly the efficiency of the postulated TCAR process. In fact, the decrease of the intensity of this signal after the light is turned off, characterized by the activation energy equal to that found in the TL experiment, can only be accounted for by the Auger-type mechanism, as was explained above. This cannot be due to a capture process which should lead to an increase rather than a decrease of the ESR signal. Moreover, the direct DAP process can also be excluded, bearing in mind that the experiment was performed after the light was turned off. The mechanism can be summarized as follows.

(1) The initial uv light ionizes the acceptors. Electrons induced in the CB are captured by shallow donors. The less distant DAP's recombine immediately. The distant pairs, however, remain practically metastably occupied after the light is turned off at low temperatures.

(2) The metastably occupied shallow donors (of concentration n_D) are depopulated only due to thermally induced redistribution of the electrons from the distant DAP's to the closer-lying ones, followed by DAP recombination.

(3) The thermal ionization itself does not lead to any change of n_D since the electrons induced in the CB are immediately recaptured by other empty shallow donors. All the other deep electron traps are occupied in our crystals and hence do not take part in this retrapping.

(4) DAP recombination may be radiative (TL) or nonradiative. One of the possible nonradiative recombination channels is energy transfer to the deep donor observed in ESR, followed by its ionization [TCAR process shown in Fig. 3(a)]. Another one, which may also contribute to small depopulation of the deep donor, is an analogous TCAR process involving, however, another acceptor [Fig. 3(b)]. The free hole created in the VB can either recombine directly with an electron on this donor or can be captured by an acceptor close to this donor, and then recombine.

(5) Owing to the small DAP recombination rates for the donor species observed in ESR and to the large hole-capture rates by the Cr^+ centers, we believe that the sole depopulation channel for these donors is the TCAR process shown in Fig. 3(a). The high efficiency of hole capture by Cr^+ [$\sigma_p^{Cr^+}/\sigma_p^A > 10^5$ (Ref. 10), where σ_p^{Cr} and σ_p^A are the hole-capture cross sections for chromium and acceptors, respectively] ensures that the process shown in Fig. 3(b) and explained above should be much less significant due to a preferential hole capture by chromium centers.

An important consequence of the above facts is that the known dependence of the DAP recombination rate (β_{AD}) on pair distance[11,12] is, in our case, averaged by thermally induced electron redistribution between the different donors sites, until the one with a close-lying populated acceptor is found, and DAP recombination can then proceed at this site. We expect, therefore, that the following important simplification is justified. We approximate the β_{AD} parameter by some averaged parameter $\langle\beta_{AD}\rangle$, which leads to a simple form of the kinetics equation:

$$dn_D/dt = -\langle\beta_{AD}\rangle n_D n_A \ . \tag{1}$$

This can be readily solved analytically by taking into account the detailed balance condition

$$\sum_i n_A^i = \sum_j n_D^j \ . \tag{2}$$

The above equation means that the light-generated occupancy of the acceptor centers (n_A) is practically equal to the concentration of the occupied shallower donors. This is true in our samples, since all other electron traps are compensated and the concentration of the deeper donor is only slightly affected by uv illumination.

The solution of Eqs. (1) and (2) then takes a simple bimolecular form:

$$n_D(t) = n_D(0)/[1+n_D(0)\langle\beta_{AD}\rangle t] \ , \tag{3}$$

where $n_D(0)$ is the concentration of the shallow donors at the moment when the light is turned off. In the above equations, we did not specify if the DAP recombination is radiative or nonradiative. It will be shown later that radiative recombination dominates in DAP processes.

The appropriate equation describing the depopulation of the deeper donor due to the TCAR process can be simplified in a manner similar to that introduced in Eq. (1). For analogous reasons we approximate the TCAR recombination rate T_{TCAR} by an averaged parameter ($\langle T_{TCAR}\rangle$), which does not depend on the distance between the recombining DAP and the donor to which the energy is transferred:

$$dn_D^d/dt = -\langle T_{TCAR}\rangle n_D n_A n_D^d \ , \tag{4}$$

where n_D^d is the occupancy of the deeper donor.

A solution of Eq. (4) is easily found from (2) and (3):

$$n_D^d(t) = n_D^d(0)\exp\left[-\frac{\langle T_{TCAR}\rangle n_D(0) n_A(0)}{1+\langle\beta_{AD}\rangle n_D(0) t}\right] . \tag{5}$$

Equation (5) can be used to analyze the experimental results. It is easily seen that at first [i.e., shortly after the light is turned off, when $n_D(0)\langle\beta_{AD}\rangle t \ll 1$] the concentration of the deeper donor decreases exponentially:

$$n_D^d(t) = n_D^d(0)\exp(-t/\tau) \ , \tag{6}$$

$$\tau^{-1} = \langle T_{TCAR}\rangle n_D(0) n_A(0) \ . \tag{7}$$

We have already explained that $n_D(0)$ decreases at elevated temperatures, due to the thermally stimulated redistribution of the electrons from distant to closely spaced DAP's, which means that the decay rate should depend on temperature with an activation energy equal to the ionization energy of the shallower donors (E_D):

$$\tau^{-1} \sim \exp(-E_D/kT) \ . \tag{8}$$

Then, for $t \gg [n_D(0)\langle\beta_{AD}\rangle]^{-1}$ the occupancy of the deeper donor reaches a saturation value described by the following equation:

$$n_D^d(t) = n_D^d(0)\exp[-\langle T_{TCAR}\rangle n_A(0)/\langle\beta_{AD}\rangle] \ . \tag{9}$$

Although the present approach pertains to a rather specific case, we believe that the averaged TCAR and DAP rates obtained from an analysis of the experimental results with Eqs. (5)–(9) will give us a good insight into the efficiency of the radiative and nonradiative (TCAR) recombination rates of the DAP's in the ZnS lattice. Before proceeding further, we should extend Eqs. (1)–(9) to cover the case observed in the experiment, i.e., we should include the possibility of thermal ionization of another shallow donor (of n_D^2 concentration) in the temperature range studied. This leads to the initial decay rate (τ) in the form

$$\tau^{-1} = \tau_1^{-1} + \tau_2^{-1} \ , \tag{10a}$$

where

$$\tau_1^{-1} = \langle T_{TCAR}^1\rangle n_D^1(0) n_A(0) \sim \exp(-E_D^1/kT) \ , \tag{10b}$$

$$\tau_2^{-1} = \langle T_{TCAR}^2\rangle n_D^2(0) n_A(0) \sim \exp(-E_D^2/kT) \ . \tag{10c}$$

B. Estimation of the TCAR efficiency

Equations (5)–(9) mean that if the $n_D^1(0)$, $n_D^2(0)$, and $n_A(0)$ concentrations are known, the appropriate TCAR transfer rates can be estimated. The TCAR transfer rates obtained from the experimental results presented in Fig. 5 are of the order of 10^{-29}–10^{-31} cm^6/s ($\langle T_{TCAR}^1\rangle$) for energy transfer from the 35-meV donor-acceptor pairs and 10^{-30}–10^{-32} cm^6/s ($\langle T_{TCAR}^2\rangle$) for Al-related donor-acceptor pairs. Their ratio $\langle T_{TCAR}^1\rangle/\langle T_{TCAR}^2\rangle$ is there-

fore 10 and is estimated with a higher accuracy (half an order of magnitude).

The relative importance of the TCAR processes can be estimated from the ratio of $\langle T_{\mathrm{TCAR}} \rangle / \langle \beta_{AD} \rangle$. In our experiment, this ratio is given directly by the value of $\ln[I_{\mathrm{ESR}}(0)/I_{\mathrm{ESR}}(\infty)]$ [see Eq. (9)], where $I_{\mathrm{ESR}}(0)$ and $I_{\mathrm{ESR}}(\infty)$ are the intensity of the donor ESR signal at the moment when the light is turned off and the saturation intensity of this signal, respectively ($I_{\mathrm{ESR}} \sim n_D^d$). The experimental results yield

$$\langle T^1_{\mathrm{TCAR}} \rangle n_A(0)/\langle \beta^1_{AD} \rangle = 0.04 \tag{11a}$$

and

$$\langle T^2_{\mathrm{TCAR}} \rangle n_A(0)/\langle \beta^2_{AD} \rangle = 0.02 \ . \tag{11b}$$

Moreover, the $\langle \beta^i_{AD} \rangle$ rates can be found separately [Eq. (7)] since

$$\langle T^i_{\mathrm{TCAR}} \rangle n_A(0)/\langle \beta^i_{AD} \rangle = [\tau_i \langle \beta^i_{AD} \rangle n^i_D(0)]^{-1} \ , \tag{12}$$

where τ is the characteristic time of the donor ESR signal decay.

The average DAP recombination rate for the 35-meV donor obtained from the above relations is of the order of 10^{-14}–10^{-13} cm^3/s, whereas for the 75-meV donor it is approximately one order of magnitude lower. These rates are lower than those found, e.g., for DAP processes in GaAs [9×10^{-10} cm^3/s (Ref. 13)], where both centers in DAP (donor and acceptor) are shallow.

Although the DAP rates determined in our experiment are probably overestimated (when thermal ionization of the donors is fast, the DAP recombination may occur mainly at closely separated pairs), the $\langle T_{\mathrm{TCAR}} \rangle$ parameters are small enough [see Eqs. (11)] to ensure that the TCAR process does not quench the radiative recombination. This is in agreement with the observation of a relatively strong thermoluminescence in our experiments. The ratio of these two parameters indicates, however, that the TCAR process can effectively quench the DAP luminescence for heavily doped samples and high excitation intensities [$n_D^d(0) > 10^{16}$ cm^{-3}].

The efficiency of the TCAR process should increase with an increase of the ionization energy of the "third" center (E_i). According to the simple model proposed by Kudykina *et al.*[14] we have $T_{\mathrm{TCAR}} \sim E_i^{3/2}$. Although the calculations leading to the above dependence were simplified, it can be expected that the TCAR process is more significant for deep centers. Similar experiments performed on ZnS crystals doped with iron[5] and chromium[15] support this conclusion. It was shown that even when the concentrations of these impurities are low enough for them to be treated as contaminants of ZnS, the TCAR process, leading to ionization of the deep transition metal impurities (Fe,Cr), becomes important.[16]

A simple way to minimize the role of the nonradiative recombination processes connected with deep centers in ZnS is to increase the concentration of the donors and acceptors in the sample to well above the contamination level. In accordance with our results, the inevitable TCAR process $DA \rightarrow$donor would affect the PL of these samples only at high excitation intensities. Our results also show that the TCAR process involving a donor and two acceptors [the model shown in Fig. 3(b)] is less efficient.

IV. CONCLUSIONS

On the basis of photo-ESR experiments, we have shown that the DAP transitions in ZnS can be nonradiative owing to the possibility of an energy transfer to a third center (a donor or an acceptor similar to those active in the DAP recombination processes). The energy-transfer process is of Auger-type, i.e., the center to which the energy is transferred (energy acceptor) is ionized in the process. Such a nonradiative recombination mechanism of the DAP's has been previously proposed by Sheinkman and co-workers for CdS (Refs. 17 and 18) and CdI_2 (Ref. 19) and by Dishman for GaP:O (Ref. 20). Due to the very complex nature of the nonradiative recombination processes, the relative importance of this mechanism could not be estimated in previous studies. A new experimental approach, proposed recently by one of the authors,[5,10] enables us to prove directly that the TCAR processes can limit the efficiency of radiative DAP recombination. Transfer rates of the order of 10^{-30} cm^6/s have been found for the case of an energy transfer to a donor. In the case of the deep impurity centers (such as TM ions) acting as energy acceptors, the TCAR process should be more efficient and should compete with the radiative transitions even for samples unintentionally doped with TM's. We have been able to reach this conclusion since the efficiency of the relevant TCAR transitions has recently been estimated.[16]

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