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Intrinsic trapping and recombination centers in $CdWO_4$ investigated using thermally stimulated luminescence

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An investigation of carrier trapping and recombination processes in $CdWO_4$ crystals undoped and Li doped has been performed by wavelength-resolved thermally stimulated luminescence (TSL) studies following x-ray irradiation at 10 K. Particular focus has been paid to the temperature region from 10 to 100 K where as many as five TSL peaks were detected, with trap depths from 0.07 up to 0.19 eV. Two of them (at 61 and at 75 K) could be ascribed to the thermal detrapping of self-trapped holes (STH) and their subsequent radiative recombination with electrons localized at intrinsic defect sites. This ascription derives from the strong similarity of their energies and frequency factors ($E=0.07$ eV and $s=10^4$ s^{-1}, $E=0.08$ eV and $s=10^4$ s^{-1}) with those of recently studied STH paramagnetic defects. The TSL emission spectrum features both the intrinsic emission at 2.5 eV probably originating from the $(WO_6)^{6-}$ group and a further band at 1.9 eV previously ascribed to a transition within a $(WO_6)^{6-}$ group lacking of an oxygen ion. Our findings allow a detailed discussion on the trapping-recombination processes in $CdWO_4$, also in comparison with those occurring in another tungstate like $PbWO_4$. Moreover, the possible role of the detected traps in the scintillation performance of the crystal is discussed.

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I. INTRODUCTION

$CdWO_4$ crystals have been extensively applied in medical imaging devices, especially in modern computed tomography systems.[1–4] The suitability of this crystal for medical imaging is related to its high density ($7.9\ g/cm^3$), good scintillation efficiency, and radiation hardness, and a scintillation decay time in the microsecond time scale. Besides the growth of single crystals by the Czochralski method, the preparation of thin films by several different techniques and with interesting optical properties was also pursued.[5–8]

Crystalline $CdWO_4$ has the wolframite structure; its electronic band structure has been investigated in detail, also in comparison to that of other tungstates and molibdates.[9–12] In these theoretical studies attention was also paid to oxygen-related defects but only for scheelite structures.[10] The luminescence and scintillation spectrum is composed of a blue-green band at about 500 nm and an additional band at approximately 570–600 nm. It was proposed that the blue-green emission is of intrinsic nature and that it originates from the $(WO_6)^{6-}$ group, similar to other tungstates with the scheelite structure such as $PbWO_4$ and $CaWO_4$, where the intrinsic emission was supposed to originate from the $(WO_4)^{2-}$ tetrahedron.[13] On the other hand, and similar once again to the cases of $PbWO_4$ and $CaWO_4$, the band at longer wavelength was assigned to a transition within tungstate groups which lack an oxygen ion.[13] The assignment of this band is controversial, however, since other authors have attributed it to the presence of Bi^{3+}.[14] Moreover, a third emission at about 1.8 eV (690 nm) was observed in Mo-doped crystals and attributed to a transition within the $(MoO_6)^{6-}$ complex.[15,16]

This paper is devoted to an investigation of charge-carrier traps in $CdWO_4$ competing with prompt electron-hole radiative recombination under irradiation with ionizing radiation. There are numerous reasons that motivate a study of this type for a scintillator material. "Deep" traps (with a lifetime much longer with respect to the scintillation decay time) trap carriers in a stable way and thereby lower the scintillation efficiency, while they can also cause an increase of the scintillation efficiency upon prolonged irradiations.[17] On the other hand, "shallow" traps, with a lifetime longer but comparable to the scintillation decay time, cause slow scintillation tails and thus alter the timing properties of a scintillator. If slow tails extend in the millisecond time scale and longer, the phenomenon is usually called "afterglow" and it is due to traps with characteristics lying between those of shallow and deep traps.

Afterglow is indeed the most critical technological problem with $CdWO_4$. In a recent study aimed at the optimization of $CdWO_4$ growth technology,[18] the phenomenon of afterglow and its dependence upon aliovalent ion doping was investigated. It turned out that crystals doped with trivalent ions (and mostly with Bi) manifest the strongest afterglow while those doped with monovalent ions (for example, Li) have very low afterglow values. Accordingly, the thermally stimulated luminescence (TSL) glow curves of trivalent ion-doped samples display glow peaks at higher temperatures with respect to those of samples doped with monovalent ions. Moreover, the transparency of $CdWO_4$ crystals is worsened by trivalent ion doping.[19,20] In contrast to this, the degradation of optical transmission after ionizing irradiation seems to be lower in the same doped crystals with respect to undoped samples.[19] An interesting comparison was performed between $CdWO_4$ and another important scintillator tungstate, $PbWO_4$.[19] In general terms the two crystalline systems behave in an opposite way since after trivalent ion doping the transparency is reduced in $CdWO_4$ while it is increased in $PbWO_4$; moreover, at variance with $CdWO_4$, TSL peaks above 100 K are reduced in $PbWO_4$ doped by trivalent dopants.[19,21] However, the effect of trivalent ion doping on the radiation hardness is similar for the two crystals and this is a puzzling result in disagreement with the previous mentioned opposite behaviors. Probably, the simultaneous influence of crystal nonstoichiometry, growth atmosphere, undesired impurities, and trivalent ion doping complicates the situation. However, at least the detrimental effects of trivalent ion doping on afterglow and transparency prior to irradiation, together with the positive effect of Li doping on afterglow, appear to be clear results.[19,20] A detailed understanding of the role of dopants can hardly be reached from present data; however the general picture that emerges from the comparison between $PbWO_4$ and $CdWO_4$ is that, starting from different defect configurations in the undoped systems, trivalent and monovalent dopings, respectively, contribute in bringing crystals closer to stoichiometry.

The positive role of trivalent ion doping in $PbWO_4$ was explained by the occurrence of Pb deficiency in the lattice[21] while the opposite effect observed in $CdWO_4$ was justified by the predominance of oxygen deficiency.[19] In Ref. 19 the authors suggest the presence of such vacancies from the analysis of the crystal coloration and from the dependence of the absorption spectra from annealing treatments in different atmospheres. However, no evidence of such defects was found so far from electron-paramagnetic-resonance experiments.

As observed in previous publications,[18,19,22–25] the TSL glow curve of $CdWO_4$ below room temperature (RT) is complex, displaying several overlapping peaks also characterized by different emission spectra. Up to now, major attention was paid to the peaks occurring at temperatures above 100 K even though TSL features below 100 K were also reported in one paper.[25]

An electron-paramagnetic-resonance study revealed the self-trapping of holes at oxygen ions after UV or x-ray irradiation.[15,26] The O^- center and its variant perturbed by Li(O^--Li) were investigated in detail in Ref. 26. The thermal decay of O^--Li centers occurs at about 160 K, and it was proposed that it is correlated with a TSL peak detected in the same temperature region,[22] namely, holes thermally freed from O^--Li centers radiatively recombine at luminescent centers giving rise to a TSL peak. On the other hand, the decay of unperturbed O^- centers occurs below 100 K and could be related to a TSL peak in that temperature region. We have thus undertaken a detailed study of the TSL of $CdWO_4$ from 10 up to 300 K in undoped and Li-doped crystals, with particular focus on the 10–100 K temperature interval; recombination centers have been also investigated by performing wavelength-resolved TSL measurements. Our aim is to identify the possible role of O^- centers in the TSL properties of the crystal; moreover, we discuss the temperature dynamics of intrinsic lattice defects in $CdWO_4$. Our study of intrinsic traps also allows us to discuss their influence on the scintillation performances of the crystal.

II. EXPERIMENTAL CONDITIONS

$CdWO_4$ crystals, undoped and 0.05% Li doped, were grown by the Czochralski method in air using platinum crucibles. Raw materials with either 4*N* purity (sample "undoped 1" and "Li doped") or 5*N* purity (sample "undoped 2") were used. Crystals undoped 1 and Li doped were grown in the Institute for Scintillation Materials, Kharkov, Ukraine while undoped 2 crystal was grown by Furukawa Co LtD, Tsukuba, Japan.

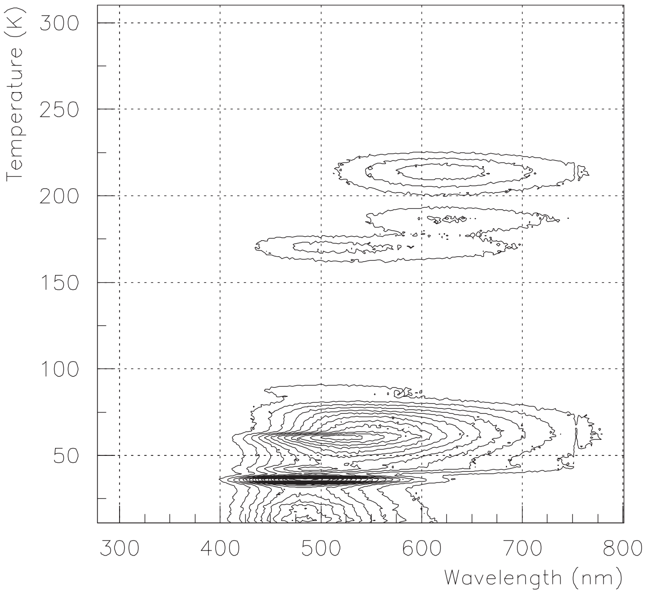

FIG. 1. Contour plot of a wavelength-resolved TSL measurement performed on undoped $CdWO_4$ (undoped 1) following x-ray irradiation at 10 K.

TSL measurements in the 10–310 K range were performed after x-ray irradiation at 10 K by a Philips 2274 x-ray tube operated at 20 kVp. The TSL apparatus consisted of a high-sensitivity spectrometer measuring the TSL intensity as a function of both the temperature and wavelength. The detection system was a monochromator (TRIAX 180 Jobin-Yvon) coupled to a charge-coupled-device detector (Jobin-Yvon Spectrum One 3000) operating in the 190–1100 nm interval. The spectral resolution was about 5 nm. The TSL emission spectra were corrected for the spectral efficiency of the detection system. A 0.1 K/s heating rate was adopted.

III. RESULTS AND DISCUSSION

A. Glow peaks and trap parameters

The contour plot of a wavelength-resolved TSL measurement of the sample undoped 1 after x-ray irradiation at 10 K is reported in Fig. 1. It shows two composite groups of glow peaks, one below 100 K and the second above 150 K. Several emission bands are observed in the visible region. The glow curves of all the considered samples were obtained from integration of wavelength-resolved data in the 300–800 nm wavelength range and they are reported in Fig. 2. Strong peaks are present in all cases below 100 K while the intensity of the glow peaks above 150 K varies significantly from sample to sample. It is lower in the undoped sample of higher purity and in the Li-doped one. So, the peaks above 150 K seem to be favored by the presence of uncontrolled impurities as well as by crystal nonstoichiometry, possibly

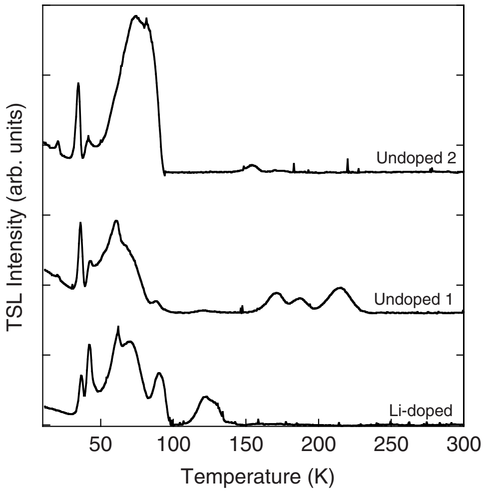

FIG. 2. Glow curves of $CdWO_4$ samples after x irradiation at 10 K. The data are obtained from integration of wavelength-resolved measurements in the 300–800 nm wavelength range.

compensated by the introduction of a monovalent dopant. While erasing the glow peaks above 150 K, Li dopant induces the appearance of a composite peak at about 125 K. The temperature of the Li-related structure is lower with respect to that which was observed in a previous paper[23] and with respect to the temperature stability of O^--Li paramagnetic centers.[26] However, in another study[18] the TSL of a Li-doped sample displayed two peaks at about 125 and 160 K (accompanied by a further one at 200 K). We wonder whether different and sample-dependent variants of such centers could exist with slightly different thermal stabilities.

Since we are mostly interested in the temperature region below 100 K, in Fig. 3 we show an enlargement of all the glow curves from 10 to 100 K. The shape of the curves is very complex, and in this case it is difficult to separate the glow peaks and to evaluate the correct trap parameters for each one. In an effort to extract the maximum information from this complex phenomenology, we performed a detailed series of partial cleaning measurements[27] for all samples; we succeeded to clearly identify five peaks at about 35, 42, 61, 75, and 88 K. The trap depths for such peaks were also determined by the initial rise method[27] applied to partially cleaned curves: an example of this evaluation is reported in Fig. 4 in the case of the undoped 1 $CdWO_4$ sample where the Arrhenius plots of all glow peaks are reported showing the exponential increase in the TSL depending from the term $\exp(-E/kT)$, where E is the trap thermal depth. Moreover, we verified that no significant shifts in the temperatures of the peaks occur upon the dose increasing up to more than two orders of magnitude so that first-order kinetic was supposed to apply to all peaks and the frequency factors s of the traps could be determined by the formula

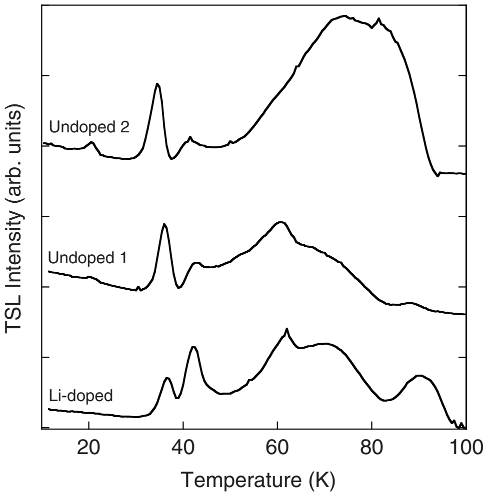

FIG. 3. Enlargement of glow curves of $CdWO_4$ samples after x irradiation at 10 K in the 10–100 K interval. The data are obtained from integration of wavelength-resolved measurements in the 300–800 nm wavelength range.

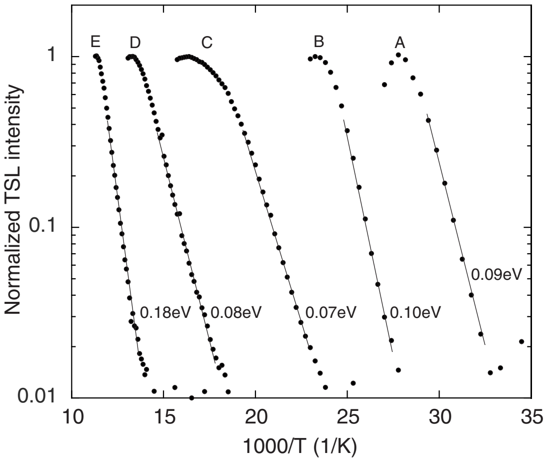

FIG. 4. Arrhenius plots of the glow curves of undoped $CdWO_4$ (undoped 1) following partial cleaning at (a) 34, (b) 40, (c) 50, (d) 0, and (e) 81 K.

$$s=\frac{\beta E}{kT_m^2}\exp\left(\frac{E}{kT_m}\right), \tag{1}$$

where β is the heating rate, T_m is the temperature of the peak maximum, and k is Boltzmann's constant. The RT lifetimes τ were calculated by

$$\tau=\frac{1}{s}\exp\left(\frac{E}{kT}\right), \tag{2}$$

with $T=290$ K. The results of the evaluation are reported in Table I. We note that the values of the RT lifetimes are qualitative due to the lack of information on the temperature dependence of the frequency factor, which can hardly be obtained [Ref. 27 (page 49) and 28]; it should be anyway much weaker with respect to the exponential term appearing in Eq. (2). An example of reconstruction of the glow curve with the parameters obtained is displayed in Fig. 5 in the case of the Li-doped sample. The reconstruction is not completely satisfactory since surely other peaks are present in the 50 K region and in the 70–80 K interval. However they are too close in temperature to the other ones so that they could not be resolved in the partial cleaning procedure.

TABLE I. Thermal depths (E), frequency factors (s), and RT lifetimes (τ) of the TSL traps. The error of the thermal depths is approximately 10%. The values of the frequency factors and of the room-temperature lifetimes are more qualitative (only the orders of magnitude are significant).

T_m (K)	E (eV)	s (s^{-1})	τ (293 K) (s)	
35	0.09	10^{11}	10^{-10}	
42	0.10	10^{10}	10^{-9}	First regime
61	0.07	10^{4}	10^{-3}	
75	0.08	$10^{3}/10^{4}$	$10^{-2}/10^{-3}$	Second regime
88	0.19	$10^{7}/10^{8}$	10^{-6}	Third regime

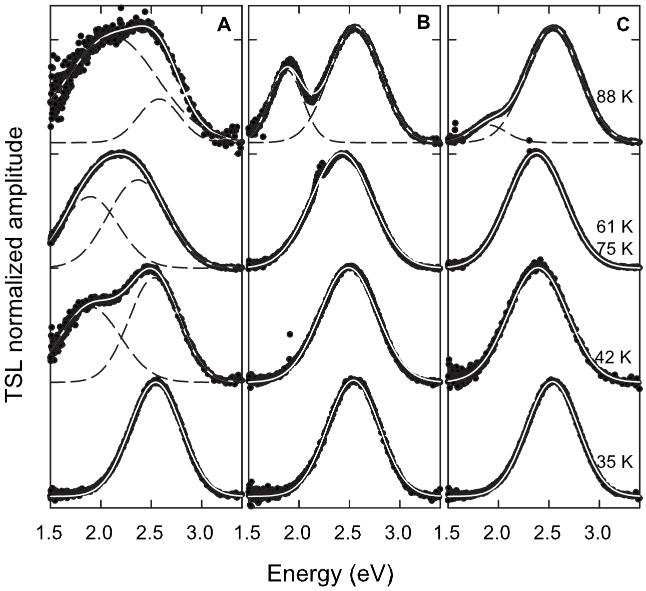

FIG. 6. TSL emission spectra obtained from integration of wavelength-resolved TSL measurements in a temperature interval of +/−3 K around TSL peaks. (A) Undoped $CdWO_4$ (undoped 1); (B) Li-doped $CdWO_4$; (C) undoped $CdWO_4$ (undoped 2). Full circles, experimental data; continuous white line, numerical fit; dashed lines, Gaussian components.

B. TSL emission spectra

The TSL emission spectra of all samples obtained from integration of wavelength-resolved data in a small interval (+/−3 K) around peak maxima are shown in Fig. 6. The undoped 1 sample [Fig. 6(A)] displays a band at 2.5 eV with 0.25 eV full width at half maximum (FWHM), shifted slightly to lower energy (2.4 eV, FWHM=0.3 eV) in the spectra of peaks at 42, 61, and 75 K. The band is again centered at 2.5 eV at 88 K; starting from 42 K, it is accompanied by an additional emission at 1.9 eV with 0.3 eV FWHM. Li doping [Fig. 6(B)] causes the disappearance of the 1.9 eV band at all temperatures except for the highest T peak at 88 K. Finally, undoped 2 [Fig. 6(C)] displays mostly the 2.5 eV emission, shifted at 2.4 eV just in a limited T interval from 40 up to 75 K similarly to the other crystals. For this sample, a weak band at 1.9 eV is detected only in the emission of the 88 K peak. The errors on the energy positions of the bands are lower than 0.05 eV.

From the comparison among the spectra reported in Fig. 6, it is clear that the low-energy band peaking at 1.9 eV is lowered by Li doping in the crystal grown with 4N purity raw material, and its presence in the undoped 2 sample grown with high-purity powders is weak. These findings are in agreement with the previous assignment of the band to a transition within a tungstate group that lacks of an oxygen ion[13] since oxygen nonstoichiometry could be balanced by Li doping, and at the same time it could be reduced in a well controlled and very pure crystal-like undoped 2.

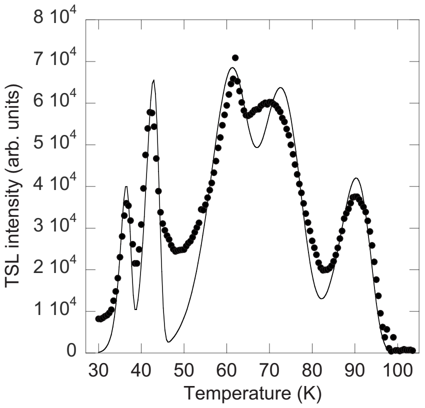

FIG. 5. Reconstruction of the glow curve of $CdWO_4$:Li with five glow peaks. Full circles, experimental data. Continuous line, numerical fit.

The shift of the intrinsic emission from 2.5 to 2.4 eV is a minor but interesting effect observed in all crystals in the T interval from approximately 40 up to 75 K. A similar phenomenon was previously observed for the emission at 2.85 eV in $PbWO_4$:La shifting at about 2.6 eV at around 60 K (Ref. 29); in that case the occurrence of a transition within a tungstate group slightly perturbed by the presence of a nearby trivalent lanthanum ion was suggested, with additionally a strong spatial correlation between the trap and the emitting center. Here, the shift is observed in all samples irrespective from their purity and doping so the presence of a perturbation of the luminescent center by a foreign ion is not likely. Rather, the slight change in the emission band could be related to a local perturbation of the recombination center due to a possible spatial correlation with the traps, as suggested in the next section for the peaks at 61 and 75 K.

C. Trap regimes

From the above presented analysis of trap parameters we classify three "trap regimes" in the TSL pattern of $CdWO_4$ below 100 K (Table I). The first one groups together the peaks at 35 and 42 K, which have approximately the same trap depth and frequency factor. No EPR signals decaying in this region were observed.[26] However the presence of these peaks in all samples suggests that their nature is intrinsic, possibly related to nonparamagnetic forms of hole centers. For example, couples of closely spaced self-trapped holes

(STH) could be created and stabilized in local stress-influenced lattice regions and be thermally disintegrated at around 35 and 42 K. The observation of two peaks is in agreement with the presence of two nonequivalent oxygen sites in the $CdWO_4$ structure.[26] We remark that similar defect states, electron bipolarons, were proposed to exist in $BaTiO_3$.[30] The second regime includes the 61 and 75 K peaks characterized by a slightly lower energy with respect to previous peaks and by very low-frequency factors. Their temperature region is similar to that of O^- centers decays, and energies and frequency factors are not far from those obtained from EPR data[26] so that a correlation between these peaks and the two decays of O^- centers observed by EPR can be proposed. The presence of two bands in the TSL spectrum suggests delocalization of the carriers in the conduction band prior to recombination at two distinct luminescent centers rather than a thermally assisted tunneling process as suggested in Ref. 26. However, thermally assisted tunneling of STH to two recombination centers could occur, where one of which is of defect origin (due to tungstate groups that lack an oxygen ion). In this case a similar thermal energy has to be assumed for the two recombination paths. Alternatively, reabsorption of the light emitted in the 2.4–2.5 eV band followed by excitation of the 1.9 eV transition could occur due to a possible partial overlap between the intrinsic high-energy emission and the excitation spectrum of this lower energy band as observed at RT [Fig. 1 of Ref. 13]. Such reabsorption of the emitted light could justify as well the presence of two distinct emission bands also in a localized recombination process, which is indeed suggested by the low values of the frequency factors of the peaks. The relative intensity of the 1.9 eV band increases by T increasing; at 88 K (third regime of the TSL glow curve) it is higher with respect to that of the 2.5 eV band in the undoped 1 crystal, and comparable to it in Li-doped sample, while it becomes observable also in undoped 2. This fifth peak has a clearly higher trap depth with respect to the other ones and an intermediate frequency factor; it could have a different nature with respect to the lower temperature ones, or represent the thermal escape of STH via valence band, showing higher trap depth and frequency factor with respect to the localized recombination process of the same STH states ascribed above to the 61 and 75 K peaks. Concerning the RT lifetimes of the traps, it can be seen from Table I that they are very short with the exception of the O^--related peaks at 61 and 75 K, for which they are on the order of 10^{-3}–10^{-2} s. Therefore, these intrinsic traps could be responsible for a residual afterglow also in high-purity undoped samples.

D. Considerations on the comparison between $CdWO_4$ and $PbWO_4$

Apart from the above mentioned similarity in the particular effect of low-energy shift of the TSL intrinsic emissions in a limited T interval, the TSL features of $CdWO_4$ and $PbWO_4$ below 100 K present also other common features worth to be underlined. In fact, in the case of $PbWO_4$ the TSL emission spectrum is related to the transition within the tungstate tetrahedron (at 2.85 eV), similarly to $CdWO_4$, where the transition within the $(WO_6)^{6-}$ group is observed as described above. Moreover, in $PbWO_4$ a composite TSL structure was revealed from 50 up to 70 K, which was resolved into three peaks at 50, 55, and 67 K characterized by trap depths of 48, 65, and 104 meV and by very low-frequency factors (10^3 s^{-1}, 10^4 s^{-1}, 10^6 s^{-1}) so that spatial correlation between traps and centers leading to a localized recombination not involving the conduction band (thermally assisted tunneling) was suggested in that case as well.[29] Due to their parameters, those three traps have RT lifetimes on the order of 10^{-2}, 10^{-3}, and 10^{-4} s. Taking into account the qualitative character of the evaluation of frequency factors and RT lifetimes, the TSL features found for $PbWO_4$ resemble those of the second and of the third regime of the TSL glow curve in $CdWO_4$ here presented. In other words, the peaks at 50, at 55, and at 67 K in $PbWO_4$ seem to correspond to peaks at 61, at 75, and at 88 K in $CdWO_4$, respectively. Also the 35 and 42 K peaks of $CdWO_4$ could find corresponding structures in $PbWO_4$ because TSL features below 50 K were clearly observed.[29,31] In $PbWO_4$, the TSL peak at 50 K was assigned to the thermal decay of WO_4^{3-} paramagnetic electron centers.[29,32] Therefore, in both cases the thermal liberation and recombination of self-trapped carriers occurs approximately in the same temperature region from 50 up to 80 K but interestingly they are of opposite sign, namely, self-trapped electrons for $PbWO_4$ and self-trapped holes for $CdWO_4$. Thus it appears that self-trapped carriers of opposite sign have similar recombination dynamics in the two crystals. This coincidence may be fortuitous or have a deeper meaning, which can be just generally outlined here. In fact, it is clear that for the occurrence of a TSL process both electrons and hole trapping sites must be present. Until now EPR data revealed only one kind of trapped carriers in each host—electron traps in $PbWO_4$ and hole traps in $CdWO_4$—while their counterparts were not observed probably because they are in a nonparamagnetic state, for example, because they are stabilized in couples. Couples of holes stabilized by a cation (Pb) vacancy ($O^-V_cO^-$ centers) were suggested to exist in $PbWO_4$ due to its lead deficiency;[33] their existence was indirectly proven by the observation of a hole center in $PbWO_4$:Mo,La caused by the possible coupling between a La^{3+} ion and a Pb^{2+} vacancy since in the case of 1:1 coupling only a single hole can be captured by this complex and a paramagnetic center can indeed be monitored by EPR.[34] Symmetrically, in $CdWO_4$ couples of electrons could be stabilized by oxygen vacancies. In each host the capture of the opposite charge carrier in the vicinity of the self-trapped one could be favored by the Coulombic field of the self-trapped charge, which polarizes its nearest environment.

We also note that in a localized recombination event between two spatially correlated carriers created in pairs by irradiation, like it may be the case for the TSL peaks now discussed, the concepts of trap and of recombination center tend to merge since the detrapping of one charge determines the recombination with the opposite charge in a one-to-one way, and the thermal decays of both defects result to be identical.

IV. CONCLUSIONS

Wavelength-resolved TSL was applied to the investigation of defects — traps and recombination centers — of $CdWO_4$ with major attention to the glow peaks occurring from 10 to 100 K. The intrinsic origin of traps responsible for TSL peaks is suggested by their presence in crystals with different purity, both undoped and Li doped. Li doping reduces the intensity of a defect related emission band at 1.9 eV detected in the TSL spectrum, confirming the previously announced positive role of monovalent dopants in the compensation of stoichiometry defects. Li doping also clearly reduces the intensity of glow peaks above 150 K.

Due to their trap parameters, glow peaks in the 60–80 K region can be ascribed to the thermal liberation and radiative recombination of self-trapped holes in accordance with previous electron-spin-resonance studies; other peaks (below 50 K and at 88 K) did not find, until now, paramagnetic counterparts and can therefore be tentatively attributed to non-paramagnetic defect variants.

The comparison between $CdWO_4$ with another tungstate which was deeply investigated in the past such as $PbWO_4$ reveals several common characteristics between the TSL pattern of the two crystals; this suggests the occurrence of common recombination features in spite of the fact that, from electron-spin-resonance studies, the recombination of self-trapped holes is observed for $CdWO_4$ at low temperatures while that of self-trapped electrons was revealed in $PbWO_4$.

Finally, the analysis of trap parameters of the investigated glow peaks shows that those related to self-trapped holes have room-temperature lifetimes on the order of 1 ms or more; therefore intrinsic centers can contribute to scintillation slow tails or even to afterglow phenomena.

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