

Hyperbolic decay of photo-created Sb²⁺ ions in Sn₂P₂S₆:Sb crystals detected with electron paramagnetic resonance

Appl. Phys. Lett. 110, 052903 (2017); https://doi.org/10.1063/1.4975684





ABSTRACT

In this paper, we employed a method that overcomes the known limitations of electron paramagnetic resonance (EPR) to monitor charge trap dynamics over a broad temperature range not normally accessible due to the lifetime broadening of the EPR lines at higher temperatures. This was achieved by measuring the decay of the EPR intensity after thermal annealing by rapid cycling back to low temperatures for the EPR measurement. This technique was used to experimentally demonstrate interesting physics in the form of a *direct* measurement of the *hyperb decay* 1/(1+t) of a charge trap population, which previously was only considered theoretically. The nontrivial effects of bimolecular recombination are demonstrated in the Sn₂S₂P₆:Sb crystals, providing an explanation of the optical sensitization process observed in photorefractive Sn₂P₂S₆:Sb used for dynamic holography.

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Availability and characteristics of free carrier traps are of major

Hyperbolic decay of photo-created Sb2+ ions in Sn2P2S6:Sb crystals detected with electron paramagnetic resonance: Applied Physics L... ionization radiation detectors, photorefraction, thermoluminescence (TL),

phosphorescence, etc. The most common method of studying the traps is thermoluminescence: monitoring of the radiative recombination of the carriers trapped during an initial irradiation (pre-illumination) and released during the course of heating.^{1,2} In systems with radiative recombination, this method is inherent and informative, but it cannot be used when recombination is nonradiative. For example, in most photorefractive systems, the photo-population and decay of trap states are crucial, but luminescence is absent and even undesirable.

For such systems, electron paramagnetic resonance (EPR) could be a good alternative. It provides a tagged detection of a particular defect center in a particular charge state (usually with an uneven number of electrons) and thus allows for *direct* monitoring of the trap population and decay. The EPR spectra from the individual traps are often easily distinguished by their unique hyperfine patterns. One of the highly appreciable advantages of EPR is that the trapped carrier concentration itself is measured rather than measuring its time derivative as in TL. This allows for isothermal decay measurements that are significantly more straightforward in evaluating the kinetics and corresponding trap depth compared to the PDF standard TL methods. However, there are only a few examples of such Help studies in the literature, and the most probable cause is the following: shorter lifetime caused by spin-lattice relaxation (i.e., spin-phonon interactions) leads to a broader resonance, as described by the Heisenberg uncertainty principle ($\Delta E \Delta t \ge \hbar/2$). This in turn often hampers EPR detection in the temperature range of interest, where depopulation of traps occurs at a reasonable rate for a given system.

limitation of EPR. The ability of this EPR method, to monitor trap dynamics in a broad temperature range not normally accessible by the EPR technique due to temperature lifetime broadening of the EPR lines, is demonstrated on the $Sn_2P_2S_6$:Sb crystals (a promising visible/infrared photorefractive system). Applying this unconventional technique (i.e., annealing at higher temperatures followed by a rapid cycling back to low temperatures for the EPR measurement) to $Sn_2P_2S_6$:Sb has also demonstrated interesting physics in itself, i.e., a hyperbolic decay of the charge trap population. A hyperbolic 1/(1+t) decay of photo-generated Sb²⁺ ions was directly measured and interpreted in terms of a "bimolecular recombination," where the numbers of extra electrons on Sb ions (Sb²⁺) and trapped holes are equal. The nontrivial effects of bimolecular recombination are directly demonstrated in the EPR-detected isothermal trap dynamics and compared with the results of holography studies. This is a *direct* detection of a hyperbolic decay of the trap population, which was only considered theoretically.

Although a hyperbolic decay was not anticipated, Sn₂P₂S₆:Sb was initially chosen for an in depth study of the thermal decay of photo-created Sb²⁺ ions, which was in part motivated by the recently found optical sensition of photorefraction in Sn₂P₂S₆:Sb used for dynamic holography.^{3,4} Preillumination in the visible increases the photorefractive gain in beam coupling measurements; the enhancement is transient, and at room temperature it disappears in tens of seconds after removal of the preillumination source. Elucidation of the decay character of the photocreated Sb²⁺ ions in this paper was anticipated to answer the question the ther Sb²⁺ ions are the "secondary photorefractive centers" in

The Sn₂P₂S₆:Sb crystals were grown at the Uzhgorod National University, Ukraine, by the chemical vapor transport method.⁵ 1% of Sb was added to the starting material. The results of photo-EPR studies of Sb ions in Sn₂P₂S₆ have been reported in Ref. 6. In thermal equilibrium, in the dark, all Sb ions are incorporated in the $Sn_2P_2S_6$ lattice as nonparamagnetic Sb^{3+} ions with the same electron configuration $(5s^2)$ as the Sn²⁺. In the as-grown state of $Sn_2P_2S_6$:Sb, the EPR signal is absent. Visible optical irradiation causes photorecharging of Sb³⁺ ions to a paramagnetic charge state Sb²⁺ ($5s^25p^1$) with a characteristic EPR spectrum dominated by the well-resolved hyperfine lines from the ¹²¹Sb and ¹²³Sb nuclei. No decay of the photocreated Sb^{2+} ions is observed in the temperature range up to 100 K; warming a previously irradiated crystal to 250 K for 3 min eliminates nearly all the Sb²⁺ ions.⁶ The defect center that traps the hole formed in the valence band (VB) after the $Sb^{3+} \rightarrow Sb^{2+}$ photorecharging and thus enables a thermal stability of Sb²⁺ has not been identified. A Sn²⁺ vacancy has been mentioned as a likely candidate,⁶ where the hole is localized on a sulfur ion adjacent to a Sn²⁺ vacancy.

There is only a small number of the published reports where photo-EI has been utilized for isothermal trap decay measurements.⁷ In those p studies, the EPR line intensity was monitored directly in the course of a thermal decay of the traps at a temperature range, where the EPR line broadening caused by spin-lattice relaxation was still of minor significance, and the decay of the EPR detected traps occurred at a reasonably high rate. These are necessary conditions for the straightforward EPR technique; it cannot be applied to systems that have an appreciably fast

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broadened beyond recognition. $Sn_2P_2S_6$:Sb is an example of a material with this "unfortunate" but common behavior: the Sb²⁺ EPR signal disappears even at 150 K due to thermal broadening, but annealing the sample for hours at this same temperature causes absolutely no reduction in the preillumination created Sb²⁺ concentration. After pre-illumination at 85 K and a subsequent thermal annealing in the dark at 150 K, the sample temperature was returned to 85 K, yielding exactly the same amplitude of the Sb²⁺ EPR lines (i.e., same Sb²⁺ concentration before and after annealing). Even though 150 K is high enough to eliminate the EPR *signal* due to thermal broadening, it is not high enough to cause any decay of the Sb²⁺ *concentration*.

The method devised in this paper that allows the abovementioned issue to be eliminated is illustrated in Fig. 1. A large steady-state concentration of Sb²⁺ ions was achieved by optical irradiation (532 nm diode laser) of the sample located in the EPR microwave resonator at a temperature low enough for an unperturbed detection (i.e., no line broadening) and no measurable decay of the Sb²⁺ concentration (85 K). After the removal of the irradiation source, it was verified that no noticeable change of the initial EPR line intensity I(t=0) occurred during the period of time necessary PDF measure the Sb²⁺ line with high precision (low noise). Subsequently, a ' Help run" was performed with a rapid heating to the "test temperature" followed by a rapid cooling back to 85 K immediately after the test temperature was achieved. If no change in the EPR line intensity is detected (i.e., the time to approach the elevated test temperature and cool back down caused no decay of the Sb²⁺ ions), then the test temperature is suitable for the isothermal decay measurement. The thermal (heating-cooling) cycle was

temperature, followed by a measurement of the EPR line intensity at 85 K after the anneal time Δt_1 . A successive thermal cycle was repeated with a different anneal time Δt_2 , and the EPR line intensity $I(\Delta t_1 + \Delta t_2)$ was measured. The cycles were repeated with varying dwell times until the EPR line vanished. To measure the decay at a different test temperature, the entire sequence was repeated starting from the same steady-state concentration of Sb²⁺ ions created by the pre-illumination. The thermal cycling was achieved using a temperature controller (model number ITC-503) with an Oxford helium-gas-flow cryostat operated with nitrogen. The proportional-integral-derivative (PID) settings on the temperature controller were tuned for minimizing the time to approach the test temperature, while keeping the "overshooting" below 0.5 K. The EPR data were taken with a Bruker EMX spectrometer operating near 9.6 GHz.



FIG. 1. Diagram of the method for monitoring the trap dynamics over a broad temperature range not normally accessible by the EPR technique due to temperature lifetime broadening of the EPR lines. Green color: optical irradiation of the sample. Black color: sample in the dark.

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The results of the Sb²⁺ thermal decay measurements are shown in Fig. 2 for three annealing temperatures: 190, 200, and 210 K (the upper, middle, and lower X-axes, respectively). Temperatures below 190 K were not used since the decay time exceeded days, and temperatures greater than 210 K were not suitable for the isothermal decay measurement (see "dry run" discussion in the previous paragraph). The measured decay traces are strongly non-exponential (note the log scale on the Y-axis). Proper adjustment of the time scales for different annealing temperatures (three X-axes on Fig. 2) shows that the *shape* of the decay curves is the same: within the experimental error, all three traces are exactly on top of each other. The shape of these curves is accurately described by the hyperbolic function, 1/(1 + t), shown as the solid line in Fig. 2. For each of the fits with the hyperbolic function $1/(1 + t/\tau_{hyp})$, the coefficient of determination (adjusted R-squared) was larger than 0.99. The fitting resulted in the following values of τ_{hvp} : 3100 ± 300, 850 ± 40, and 263 ± 8 s for T = 190, 200, and 210 K, respectively.



annealing temperatures: 190 K (red circles, upper X-axis), 200 K (green diamonds, middle X-axis), and 210 K (blue squares, lower Xaxis). The Y-axis values (peak-to-peak amplitude of the line at 5132 G; annealing did not affect the line shape and width) were normalized to unity with the same normalization factor for all three temperatures. Solid line: hyperbolic function 1/(1 + t). Fitting the decay traces with the hyperbolic function $1/(1 + t/\tau_{hyp})$ resulted in the following values of τ_{hyp} : 3100 ± 300, 850 ± 40, and 263 ± 8 s for T = 190, 200, and 210 K, respectively. For each of the fits, the coefficient of determination (adjusted R-squared) was larger than 0.99. Inset: the Arrhenius plot for the reciprocal decay time τ_{hyp}^{-1} . Dashed line: linear fit to the data points.

In principle, a non-exponential trap decay could be explained by the presence of several types of traps with different activation energies. However, in that case, the shape of the decay curves would never be the same for annealing temperatures with significantly different decay rat therefore, a multi-trap cause should be disregarded. The actual origin of the hyperbolic decay in Fig. 2 is revealed in the following paragraphs.

Due to the complexities of thermoluminescence applications, the thermal decay of the trap states has been a subject of extensive theoretical studies over many decades (Refs. 1, 2, and references therein). On the other hand, applying those results to our case (i.e., isothermal trap decay where n(t) is

with the time derivative dn/dt in the course of heating the sample. The intermediate step, calculations of n(t), is usually omitted in the TL literature (Refs. 1, 2, and references therein). Below, we provide an example of such a consideration performed in the spirit of Ref. 8, but with the use of real material parameters with direct meaning (e.g., electron capture crosssections, trap concentrations, etc.), rather than electron transition probabilities.

Fig. 3 shows the energy levels of the trap states in the band gap with the electron pathways involved in the thermal decay of traps. Providing that the concentration of electrons thermally excited to the conduction band (CB) from the Sb²⁺ ions is infinitesimal compared to the concentration of Sb²⁺ ions, and then the thermal decay of optically created Sb²⁺ ions (decay of electrons trapped on Sb³⁺ ions) can be described by the following rate equation:

$$\frac{dn_{Sb^{2+}}}{dt} = -s \, \exp\left(-\Delta E/kT\right) \, n_{Sb^{2+}} \frac{\sigma_{X^+} n_{X^+}}{\sigma_{X^+} n_{X^+} + \sigma_{Sb^{3+}} n_{Sb^{3+}}},\tag{1}$$

where $n_{Sb^{2+}}$ is the concentration of Sb²⁺ (trapped electrons), $n_{Sb^{3+}}$ is the concentrations of Sb³⁺ (empty electron traps), and n_{X^+} is the concentr of X⁺ (X centers with a trapped hole; the effective charges of X and X⁺ and X⁺ are the electron capture cross-sections of Sb³⁺ unknown). $\sigma_{Sb^{3+}}$ and σ_{X^+} are the electron capture cross-sections of Sb³⁺ and X⁺, respectively. *s* is a pre-exponential multiplier ("frequency factor" that includes a number of system parameters, e.g., the effective density of states in the CB), ΔE is the separation of the trap levels from the CB bottom, *k* is the Boltzmann constant, *T* is the temperature, and *t* is the time. The term $\frac{\sigma_{X^+}n_{X^+}}{\sigma_{Y^+}n_{Y^+}+\sigma_{Y^+}n_{Y^+}}$ describes the "branching ratio" of \checkmark PDF X-center (X⁺) or be re-trapped by an Sb³⁺ trap. Eq. (1) is practically the same as Eq. (4) in the pioneering paper by G. F. J. Garlic and A. F. Gibson (Ref. 8) – except for notations and their assumption of the electron capture cross-sections, $\sigma_{Sb^{3+}}$ and σ_{X^+} , to be equal.



FIG. 3. Energy diagram of the levels in the band gap and the electron transfer processes. The pathways taken are determined by material parameters, either the case of negligible retrapping (a) or strong retrapping (b). Solid arrows: thermal ionization of trapped electron (Sb²⁺); dashed arrows: capture of electron by Sb³⁺ (retrapping) and X⁺ (recombination).

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The rate Equation (1) can only be analytically solved for two extreme cases: (1) negligible re-trapping and (2) strong re-trapping. In the case of negligible re-trapping ($\sigma_{X^+}n_{X^+} \gg \sigma_{Sb^{3+}}n_{Sb^{3+}}$), Equation (1) reduces to

$$\frac{dn_{Sb^{2+}}}{dt} = -s \, \exp\left(-\Delta E/kT\right) \, n_{Sb^{2+}} \tag{2}$$



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$$n_{Sb^{2+}}(t) = n_{Sb^{2+}}(t=0) \exp(-t/\tau_{\exp}), \tau_{\exp} = [s \exp(-\Delta E/kT)]^{-1}.$$
 (3)

The solution for this isothermal decay, n(t), is a single exponential decay that corresponds to the "first order" kinetics of TL, dn/dt, under the increasing sample temperature.^{1,2,9} The decay time τ_{exp} in Eq. (3) is solely determined by the trap characteristics: trap depth ΔE and frequency factor s. Other material parameters are irrelevant. With τ measured at several constant temperatures, the values of ΔE and s can be found through the Arrhenius plot: a straight line fit for $\ln(\tau_{exp}^{-1})$ vs. 1/T. ΔE is the slope of the line, and s^{-1} is Y-axis intercept (at 1/T = 0).

In the case of strong re-trapping, $\sigma_{X^+} n_{X^+} \ll \sigma_{Sb^{3+}} n_{Sb^{3+}}$, with an additional assumption $n_{Sb^{2+}} = n_{X^+}$ (equal concentrations of trapped electrons and holes, consistent with charge neutrality, also referred to as bimolecular recombination¹), Equation (1) reduces to:

$$\frac{dn_{Sb^{2+}}}{dt} = -s \exp\left(-\Delta E/kT\right) \frac{\sigma_{X^{+}} n_{Sb^{2+}}^2}{\sigma_{Sb^{3+}} n_{Sb^{3+}}}$$
(4)

with the solution

$$n_{Sb^{2+}}\left(t\right) = \frac{n_{Sb^{2+}}\left(t=0\right)}{t/\tau_{hyp}+1}, \quad \tau_{hyp} = \left[s \exp\left(\Delta E/kT\right)\right]^{-1} \frac{\sigma_{Sb^{3+}} n_{Sb^{3+}}}{\sigma_{X^{+}} n_{Sb^{2+}}\left(t=0\right)}$$

The solution for this isothermal decay, n(t), is a hyperbolic decay that corresponds to pure "second order" kinetics (b = 2) for dn/dt in TL (see the kinetic order (b) value in Refs. 1 and 2, where 1 < b < 2 is considered mixed

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material parameters, in contrast to the single exponential decay case in Eq. (3). The presence of the term $n_{Sh^{3+}}$ makes the hyperbolic decay rate τ_{hyp} dependent on the Sb³⁺ concentration (i.e., different concentrations of Sb³⁺ yield different decay rates). Even more important, the hyperbolic decay rate depends on the *initial concentration* of optically created Sb²⁺ ions, unlike the case of negligible retrapping (i.e., single exponential solution, Eq. (3)). This conclusion was verified through the thermal decay measurement taken at 210 K with pre-illumination at a different wavelength, 544 nm (green He-Ne laser). The steady-state concentration of Sb²⁺ ions (the initial concentration for the decay measurements) achieved by 544 nm light is 2.3 times *smaller* than that created using 532 nm light. With this change, the hyperbolic decay parameter τ_{hyp} became 2.2 times *longer* (590 ± 20 s at 544 nm vs. 263 ± 8 s at 532 nm); this result is in good agreement with Eq. (5). The extraction of ΔE from the decay traces taken at different temperatures (using the same pre-illumination wavelength) through the Arrhenius plot procedure is still possible, but care must be taken to keep the same initial Sb²⁺ concentration for all decay measurements (see data in Fig. 2 at t = 0). The decay parameters τ_{hyp} for each decay trace on Fig. 2 were found through fitting the data with the hyperbolic function in Eq. (5). The PDF corresponding Arrhenius plot (inset in Fig. 2) provided the trap depth value (the distance between the Sb^{2+/3+} level and the CB bottom), $\Delta E = 0.42 \pm 0.01$ eV. This agrees with the results in Ref. 10, which also show the activation of 0.42 eV for Sb²⁺. In terms of the frequency factor s, the presence of the initial Sb²⁺ concentration and several (generally unknown) material parameters ($\sigma_{Sb^{3+}}, \sigma_{X^+}$, and $n_{Sb^{3+}}$) in the expression for τ_{hyp} in Eq. (5) make timation of the avalue difficult without linewing the values of these PDF

solution in Eq. (3)). Note that the standard assumption applied to thermoluminescence theory considers equal cross-section for different centers,^{8,9} although the electron capture cross-sections for different centers may vary by many orders of magnitude.¹¹

Although an "electron" decay mechanism is considered throughout the paper (thermal ionization of Sb²⁺ with the formation of free electrons in the CB that recombine with the X⁺ centers—solid and dashed arrows in Fig. 3), it should be noted that a "hole" mechanism is also possible. Such a hole mechanism would be comprised of thermal excitation of holes from the X⁺ centers to the VB, with a subsequent re-trapping of those holes on the X centers, followed by a recombination of the holes with electrons localized on Sb ions (Sb²⁺). Regardless of the mechanism, either electrons or holes or a combination of the two, Eq. (1) gives a hyperbolic decay providing a strong re-trapping and bimolecular recombination. The assignment of the decay mechanism for the particular system of Sn₂P₂S₆:Sb is outside the scope of this paper.

Although in this paper we do not provide direct evidence that the electron decay mechanism is strongly dominating, the results outside the scop this paper strongly suggest this to be the case. The Sb²⁺ "photo-creatic presented in this work) and the Born-Haber cycle suggest a significantly higher activation energy of the trapped holes (energy separation between the X/X⁺ level and the VB). Furthermore, the exact same activation energy as found using EPR (190–210 K; see the inset of Fig. 2) has been measured with a holographic technique over a broader temperature range (230–320

plot ensures a strong domination of just one mechanism (otherwise, a noticeable nonlinearity would be observed). It is worth noting that it is possible that two activation energies (for trapped electrons and holes) could exist with similar (yet indistinguishable) values on the Arrhenius plot values. Although not impossible, it is rather far-fetched. Although these additional results that will be provided in a future publication support the electron decay mechanism, the omission of such results does not detract from the fact that a hyperbolic decay is present in $Sn_2P_2S_6$:Sb.

An experimental technique has been devised to overcome the seeming limitation of EPR, allowing for the measurements of trap dynamics in a broad temperature range not normally accessible by the EPR technique due to temperature broadening of the EPR lines. This technique was utilized to perform direct measurements of the Sb²⁺ concentration decay in the S₂P₂Sn₆:Sb crystals. The unexpected observation of a hyperbolic decay of Sb²⁺, as a result of the direct isothermal EPR measurement, is a direct detection of the theoretically considered hyperbolic decay (b = 2) of the trap population (directly measuring n(t) rather than dn/dt followed by the intermediate steps to calculate n(t), as is the case of thermoluminescence). Furthermore, this isothermal EPR technique provides the more reliabl determination of ΔE compared to various thermoluminescence and holographic methods.¹³

Evidence of this hyperbolic decay allowed us to determine the photorecharging process of both the Sb²⁺ and the silent X center decay rates. It provided information on the important features of the X center: in thermal equilibrium only X centers are present, and the optical irradiation creates

vial concentrations of Ch²⁺ and V⁺ (condition for himolocular

in the dark. It is this creation of a "donor density" that provides the means for optical sensitizing of photorefraction in $Sn_2P_2S_6$:Sb.^{3,4}

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