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Thermoluminescence kinetic study of binary lead-silicate glasses

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ABSTRACT

This paper describes a detailed experimental study of the thermoluminescence (TL) properties of four binary lead-silicate glasses, with PbO concentrations ranging from 32% to 62% in mole percent. The TL glow peaks between room temperature and 300 °C were analyzed using a systematic thermal cleaning technique. The T_{\max} - T_{stop} and E - T_{stop} methods of analysis were used to identify the number of peaks under the glow curves, and to obtain the activation energy E for each TL trap. A computerized glow curve fitting analysis is used to fit the experimental data to four first-order peaks with maxima at temperatures of 54, 80, 110 and 210 °C, as measured with a heating rate of 2 °C/s. The kinetic parameters of the glow-peak at 210 °C were confirmed by using phosphorescence decay methods of analysis. The TL traps associated with the low-temperature TL peak at 54 °C are found to depend strongly on the PbO concentration of the samples, while the higher-temperature TL peaks show a behavior independent of the PbO concentration. The activation energy E and frequency factor s of the low-temperature TL trap associated with the peak at 54 °C are consistent with a trap involving a delocalized transition through the conduction band. However, the activation energies and frequency factors for the higher-temperature TL traps are consistent with traps involving localized transitions via an excited state below the conduction band. The data suggest that these higher-temperature TL traps are associated with the common silicate matrix in these binary silicate glasses.

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1. Introduction

Heavy metal oxide glasses, such as lead silicates, have been the subject of numerous studies due to their importance in the photonics industry. Detailed knowledge of the physical properties, particularly electronic, and internal structure of these glasses is critical if they are to be used in all-optical switches. The present study concerns glasses of the lead-silicate family, which have been the subject of several recent experimental and theoretical studies [1–13]. Previous studies have presented two differing views on the nature of the glass structure as a function of lead concentration. In this paper, we provide another approach to study lead silicates, namely, thermoluminescence (TL), to aid in the understanding of how its structure and electronic properties are related.

Mizuno et al. [14] studied the formation of a plumbate network in binary lead-silicate glasses based on the leaching behavior of Pb^{2+} over a wide composition region. They found a steep increase of the effective diffusion coefficient of Pb^{2+} at 40 °C for 35–50 mol% PbO concentrations. They proposed that these changes can be interpreted as the lead ion playing the role of a network former over the entire glass. Rybicki et al. [15] performed molecular dynamics simulations and X-ray absorption fine

structure (EXAFS) investigations of the structure of lead-silicate glasses. Their results showed that the PbO_4 groups are the dominant structural units in lead-silicate glasses for any concentration and that at lower lead concentrations the co-existence of the PbO_4 and PbO_3 groups is possible. Meneses et al. [16] studied seven binary lead-silicate glasses by infrared spectroscopy. These authors provided an explanation for the observed structural evolution of the glass matrix and the drastic changes that occur around a concentration of 45% lead content. Below this 45% threshold, lead cations act only as modifiers of the silicate network, while above it the glass structure is modified by the developing lead network. The NMR experiments of Fayon et al. [17,18] showed that Pb forms covalent PbO_4 and PbO_3 pyramids over a large compositional range. At larger Pb contents they observed the existence of a PbO-based network. For concentrations less than 60 mol% PbO, however, they concluded that Si is the main glass former and as the Pb content increases, the silicate network is broken.

There have been no previous published reports of the thermoluminescence properties of binary silicate glasses. This paper describes a detailed experimental study of the TL properties of four such glasses with PbO concentrations ranging from 32% to 62% in mole percent. The TL glow curves up to 300 °C were analyzed using several standard analysis techniques, namely a systematic thermal cleaning procedure, the initial rise technique, the phosphorescence decay analysis, and a computerized glow

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curve-fitting procedure. The experimental data is fitted to four first-order peaks with maxima at temperatures of 54, 80, 110 and 210 °C, as measured with a heating rate of 2 °C/s. The activation energy E and frequency factor s of the low-temperature TL trap associated with the peak at 54 °C is consistent with a trap involving a delocalized transition through the conduction band. On the other hand, the activation energies and frequency factors for the higher-temperature TL traps were found to be consistent with traps involving localized transitions through excited states below the conduction band.

2. Experimental

The four binary silicate lead samples were fabricated by D.H. Blackburn at the National Institutes of Standards and Technology (NIST) and polished at the Oklahoma State University

Table 1

Physical properties of the four binary silicate glasses n = index of refraction, α = absorption coefficient, ρ = density and c = specific heat.

(% PbO)	(% SiO ₂)	n (514.5 nm)	α (cm ⁻¹) (514.5 nm)	ρ (kg/m ³)	c (J/kgK)
32	68	1.715	0.439	4510	560
42	58	1.829	0.539	5320	465
52	48	1.922	0.556	6030	438
62	38	2.080	1.150	6830	353

(OSU). Table 1 shows some of their physical properties measured at OSU. Each sample was approximately 20 mm high and 15 mm wide. Prior to any measurements, we measured the zero-dose TL signal and found it to be of the order of the background. We used a conventional Daybreak TL equipment, with Corning 7–59 and a HA-3 infrared-rejecting filter to reduce thermal noise, and a 9635QB photomultiplier tube for photon counting. All TL glow curves were recorded with a linear heating rate of 2 °C/s under constant nitrogen flow. The glass samples were irradiated on plate using a 100 mCi Sr-90 source with a nominal dose rate of 2 Gy/min. We digitally subtracted the black-body radiation from the TL glow curves.

3. Experimental results

3.1. The TL glow curves for different PbO% concentrations and different beta doses

Fig. 1a–d illustrates the TL glow curves for the four samples using the same beta dose of 1 Gy and with a linear heating rate of 2 °C/s. There are small variations in the exact location of the various TL peaks as well as in the relative TL peak heights, but three peaks are clearly identified around 54, 110 and 210 °C. The glow curve for the 52% PbO sample in Fig. 1c also shows a smaller peak around 80 °C. The presence of these four peaks in the

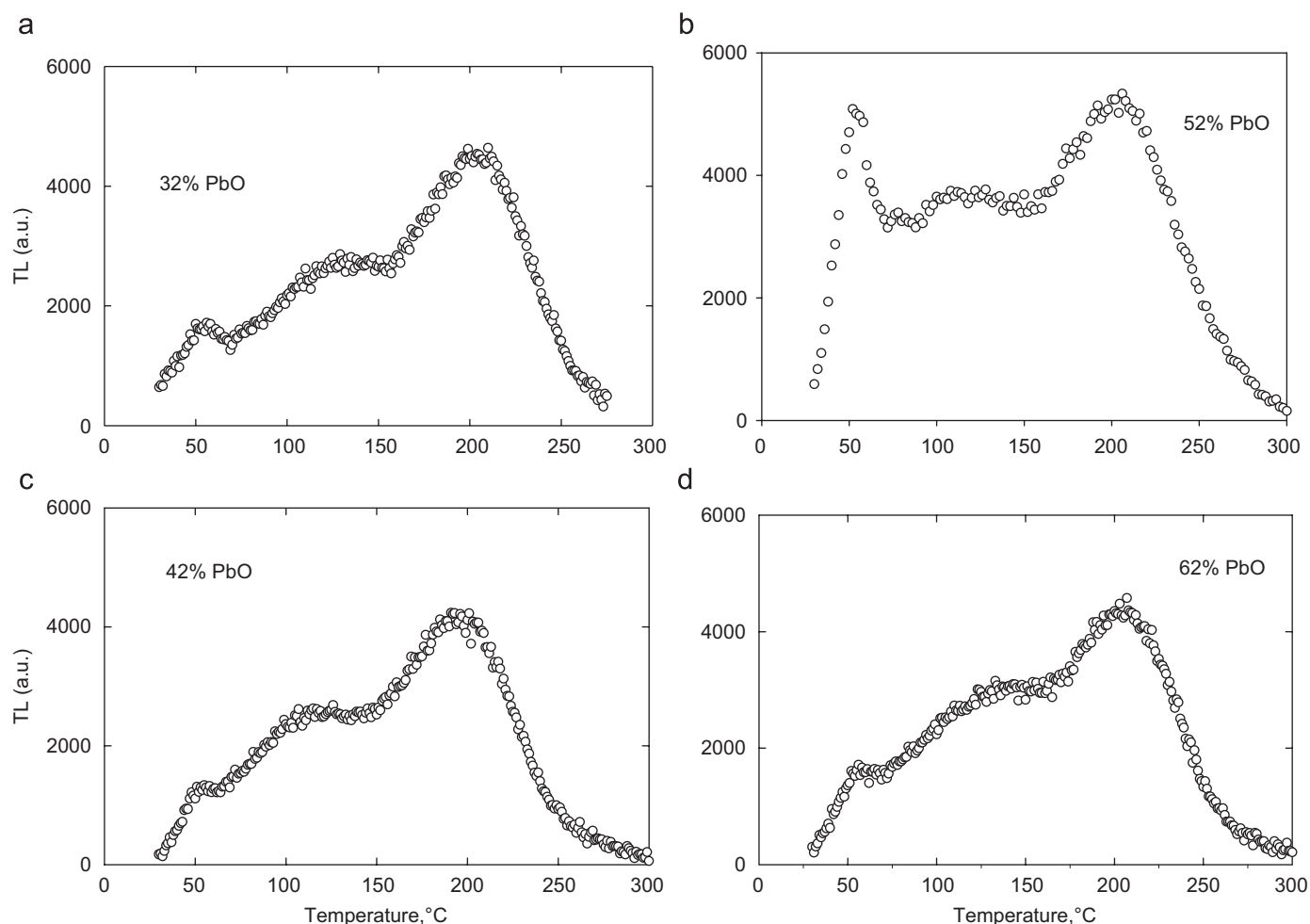


Fig. 1. Comparison of the TL glow curves of the four samples containing 32%, 42%, 52% and 62% PbO, measured with a linear heating rate of 2 K/s. The samples were given a beta dose of 1 Gy.

glow curves in Fig. 1 is confirmed in the next section, where a detailed computerized glow curve fitting procedure is carried out.

By comparing the data for the 32% and 42% PbO samples in Fig. 1a and b, these appear to be almost identical except for small differences in the location and height of the low-temperature TL peak at 54 °C. In contrast, a comparison of the 42% and 52% PbO samples in Fig. 1b and c indicates significant differences between the two samples, for both low- and high-temperature peaks. Finally, a comparison of the 52% and 62% samples in Fig. 1c and d again demonstrates large differences between the glow curves. These results are in agreement with previous studies by Meneses et al. [16] and Fayon et al. [17,18] on lead-silicate glasses, which

found that significant changes take place in the structure of these materials at PbO concentrations above 45 mol%.

Fig. 2 shows the TL response for the 32% PbO sample for higher beta doses in the dose region 2–24 Gy. The TL glow curves contain apparent peaks at 54, 110 and 210 °C. The data in Fig. 2 also shows that the temperature of maximum TL intensity (T_{\max}) changes very little within the dose range studied, which is a strong indicator of first-order kinetics for the TL process. The error bars in Fig. 2 represent the average of at least three runs on the same glass sample. Similar results to those in Fig. 2 were obtained for the other three samples studied in this paper.

3.2. Thermal cleaning and peak analysis of the TL glow curves

We used the well-known T_{\max} – T_{stop} method [19] to determine the number of overlapping TL peaks within the TL glow curve. In this method, the glass sample is irradiated with a beta dose and subsequently heated to a temperature T_{stop} , while recording the TL signal. Immediately after cooling the sample to room temperature, the sample is heated to the maximum temperature of 300 °C, and the temperature T_{\max} of maximum TL intensity is recorded. The whole experiment is repeated by re-irradiating the glass sample and gradually increasing the temperature T_{stop} in small steps of 7–10 °C. In this manner, the glow curve is thermally cleaned in a systematic fashion as shown in Fig. 3 with T_{stop} values ranging from 35 to 205 °C in steps of 8 °C. The lowest graph in Fig. 3 indicates the size of the black-body signal, representing the background which is subtracted from the TL signal. We obtained a total of a minimum 18 different glow curves similar to the ones shown in Fig. 3 for each sample. The results for the other three samples studied were very similar to Fig. 3.

Fig. 4a shows the results of using the T_{\max} – T_{stop} method for 52% PbO sample and it verifies the presence of at least three distinct TL peaks in the glow curves located at ~54, 130 and

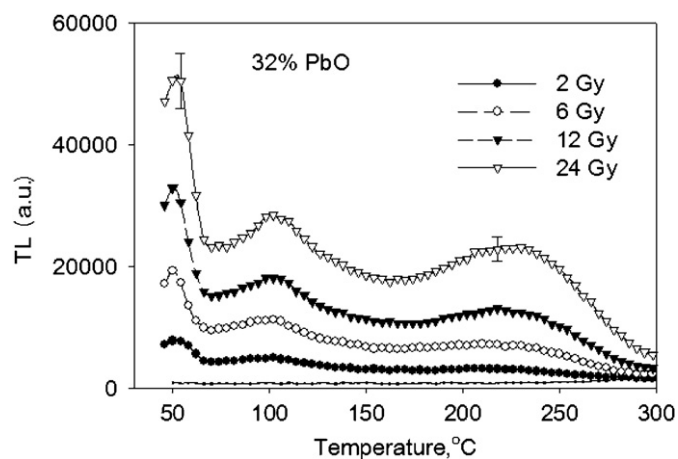


Fig. 2. The TL response for the 32% PbO sample for doses in the region 2–24 Gy, measured with a linear heating rate of 2 K/s. Similar dose responses were obtained for the other three samples studied. The bottom plot in this graph represents the background signal which is subtracted from the TL glow curves.

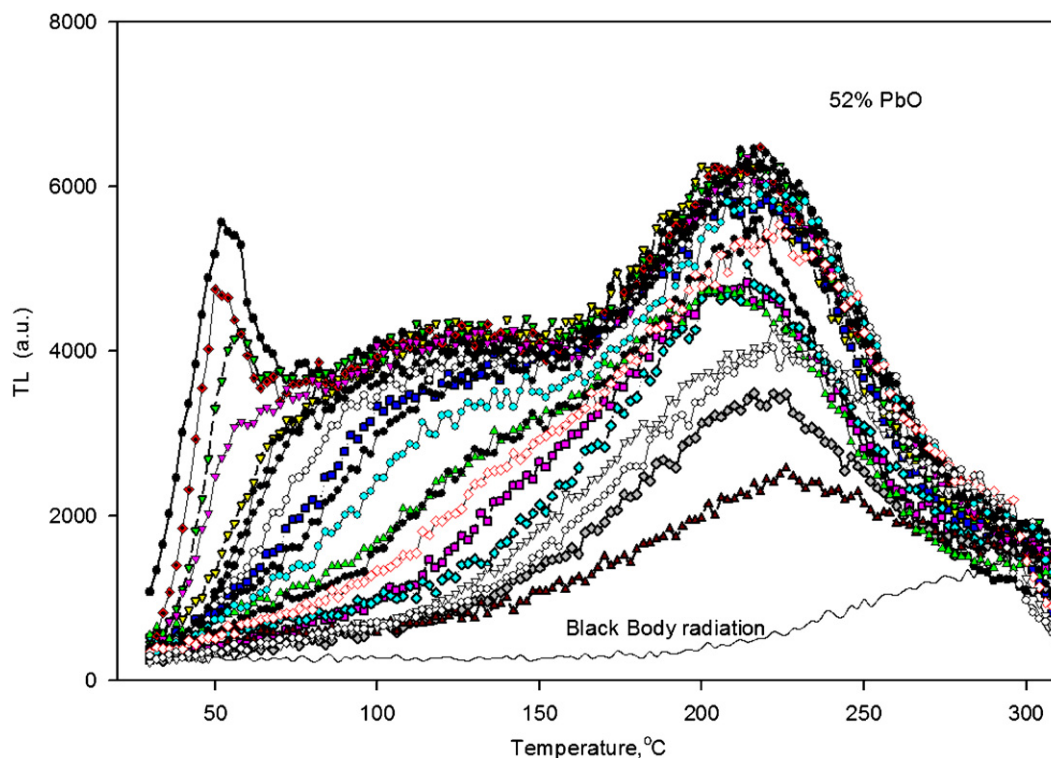


Fig. 3. Gradual thermal bleaching of the glow curves using the T_{\max} – T_{stop} method yields 21 different glow curves for the 52% PbO sample. The T_{stop} values range from 35 to 205 °C in steps of 8 °C. Similar results were obtained for the other three samples. The samples were given a beta dose of 1 Gy.

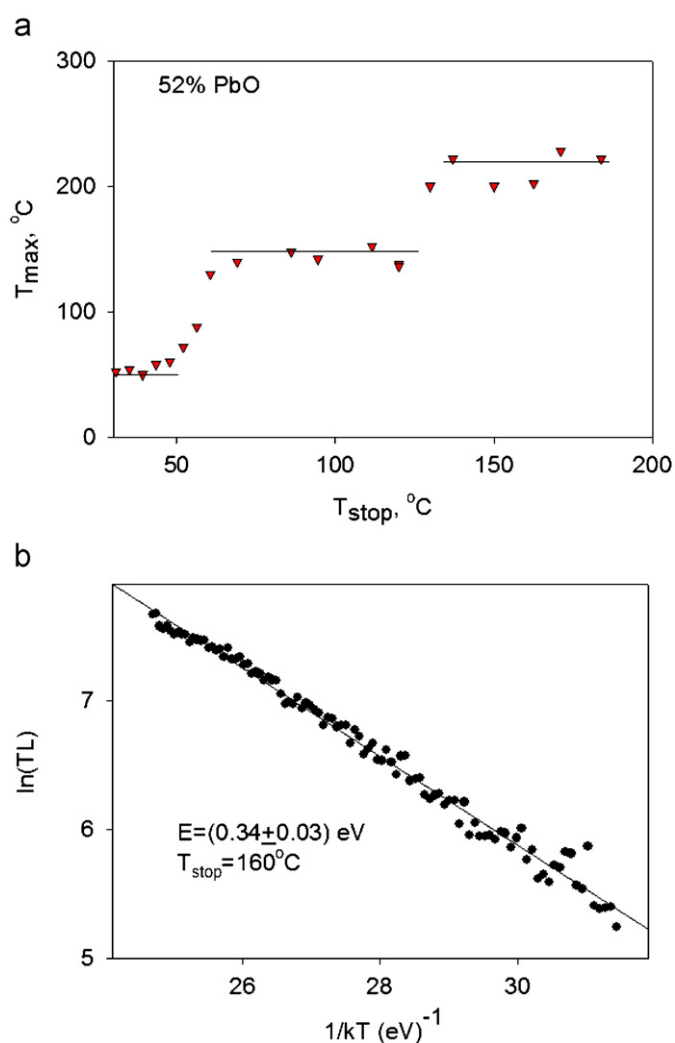


Fig. 4. (a) The results of using the $T_{max}-T_{stop}$ method for the 52% PbO sample. (b) A typical example of calculating the E -value by using the initial rise method of analysis. The natural logarithm of the TL intensity is plotted against the quantity $1/k_B T$, with the slope equal to the activation energy E . The T_{stop} value used was $30^\circ C$.

$220^\circ C$, resulting in a “staircase structure” typical of this method. For each of the partial TL glow curves in Fig. 3, we used the well-known initial rise method of analysis to estimate the activation energy E of the TL traps. In this method the natural logarithm of the TL intensity is plotted against the quantity $1/k_B T$, where k_B is the Boltzmann constant, to obtain the E -values corresponding to these individual peaks. A typical example of this procedure applied to one of the glow curves in Fig. 3 is shown in Fig. 4b. The slope of the linear graph in the inset of Fig. 4b represents the activation energy E of the corresponding TL trap. We applied the initial rise method of analysis to all the data of Fig. 3, and Fig. 5a, b shows the resulting $E-T_{stop}$ graphs for all four samples studied. The error bars in Fig. 5a are approximately equal to the size of the experimental symbols, and represent the statistical uncertainty (± 1 s.d.) of the regression line fitting to the initial rise experimental data for the 52% PbO sample.

The data in Fig. 5a suggest that the activation energy E for the low-temperature TL peak at $54^\circ C$ is $E \sim 1.2 eV$, while the E -value of the higher-temperature TL peaks is much lower, $E \sim 0.35 eV$. This is a rather unusual result, since in most materials higher-temperature TL peaks correspond to higher E -values. The detailed analysis of the next section will demonstrate that the low

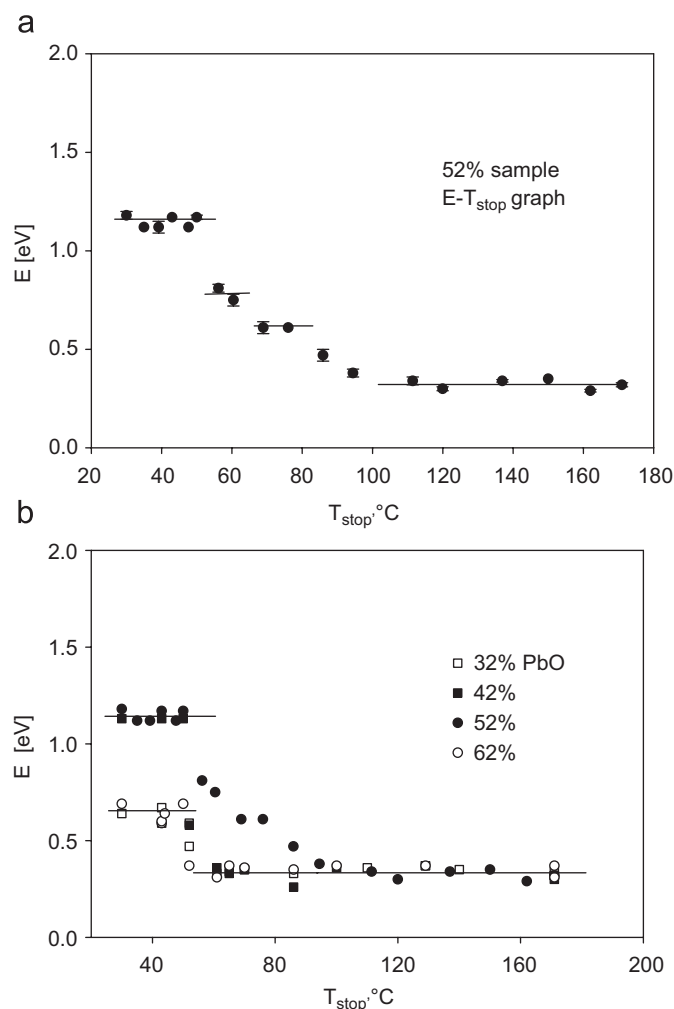


Fig. 5. (a) The activation energy E as a function of the temperature T_{stop} for the 52% PbO sample, as obtained from the partial glow curves shown in Fig. 3(a). (b) The activation energy E as a function of T_{stop} for all four samples studied. Two distinct regions are clearly seen for temperatures $T_{stop} < 50^\circ C$, and for $T_{stop} > 50^\circ C$, with different behaviors. The solid lines are a guide to the eye.

$E \sim 0.35 eV$ values are consistent with the higher-temperature TL peaks corresponding to delocalized electronic transitions.

The data in Fig. 5a shows that the value of the activation energy E decreases steadily between T_{stop} values of 40 and $100^\circ C$, indicating the possible presence of several overlapping TL peaks in this temperature region. This is indeed verified in the next section.

Fig. 5b presents the results of the $E-T_{stop}$ method for different percent of PbO concentrations, with clear statistical differences between the four samples. The E -values for the 32% PbO and 62% PbO samples (open squares and open circles in Fig. 5b), are seen to be almost identical, with a value of $E \sim 0.65 eV$ for $T_{stop} < 50^\circ C$, and a value of $E \sim 0.35 eV$ for $T_{stop} > 50^\circ C$. The behavior of the 42% and 52% samples (solid squares and solid circles in Fig. 5b) is very different, with a value of $E \sim 1.2 eV$ for $T < 50^\circ C$, a value of $E \sim 0.35 eV$ for high T_{stop} values, and a steadily decreasing value of E at intermediate temperatures.

We note that the first TL peak at $54^\circ C$ of the 32% and 62% samples has a lower E -value $\sim 0.65 eV$, while the 42% and 52% samples have a higher E -value $\sim 1.2 eV$; this difference indicates that network formation plays a key role in determining the activation energy. Network formation due to bond overlap is expected to give rise to a larger spread in the available energy

levels and, therefore, to lower activation energies. Our data is consistent with the idea that there is strong network formation at both 32% and 62%, with the network former at 32% mostly being due to the silicates, while in the 62% sample it is primarily due to lead oxides. A lower E -value for the 62% sample also suggests the PbO network is softer than the silicate network. The weakly

networked or fragmented systems of 42% and 52% have high E -values, with the structurally softer 52% sample having an activation energy that is lower than the 42% sample. On the other hand, for larger values of $T_{\text{stop}} > 90^\circ\text{C}$, the E -values of the four samples are identical and independent of the PbO concentration. This suggests that the traps that are activated at higher

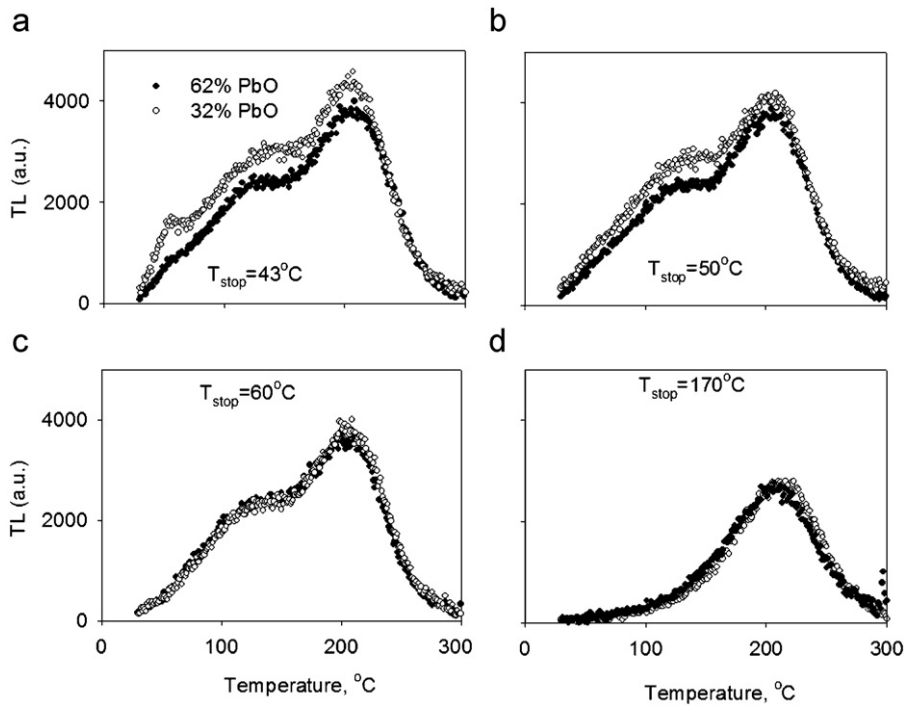


Fig. 6. Comparison of the partial TL glow curves obtained for the 32% and 62% samples when heated to four different T_{stop} temperatures of 43, 50, 60, and 170°C . Clear differences are seen between the two samples in (a, b), while in (c, d) the TL glow curves are identical within the accuracy of the experimental data. Similar results were obtained for the other samples studied. The samples were given a beta dose of 12 Gy.

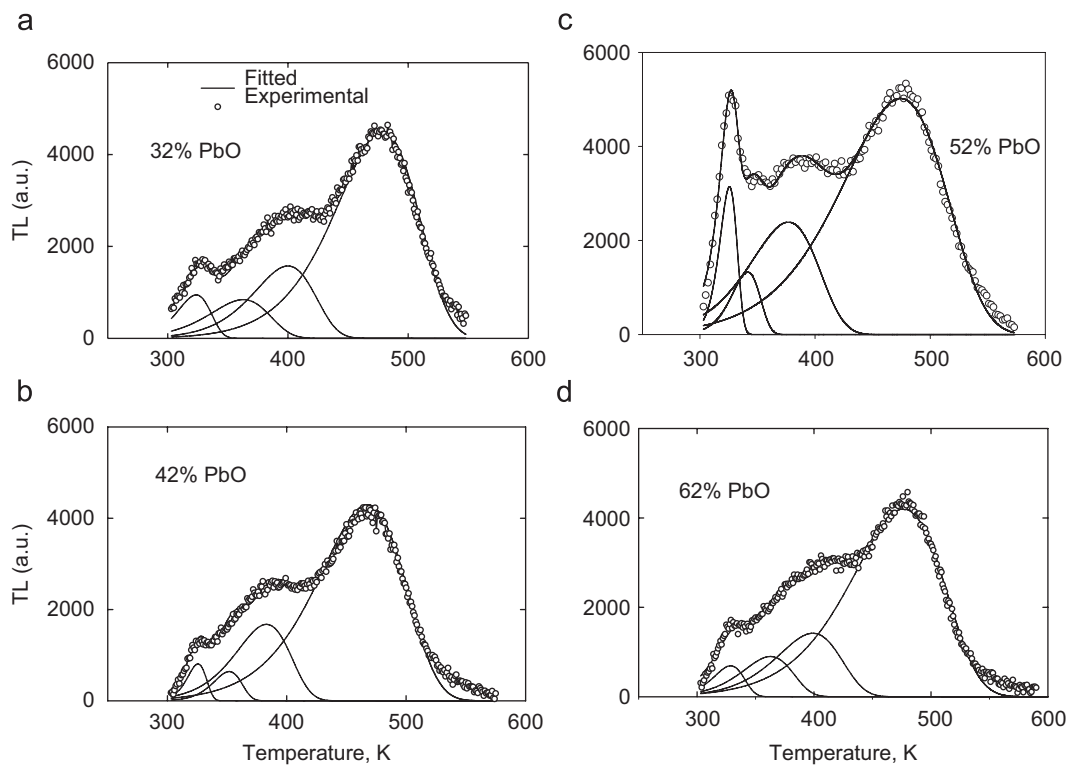


Fig. 7. The TL glow curves for all four samples studied are fitted to a sum of four first order TL peaks.

temperatures are associated with the silicates. The average E -value for the higher-temperature TL peaks for $T_{\text{stop}} > 90^\circ\text{C}$ is $E = 0.350 \pm 0.006\text{ eV}$.

Fig. 6a–d shows a comparison between the partial TL glow curves obtained for the 32% and 62% samples when heated to four different T_{stop} temperatures of 43, 50, 60, and 170°C . Clear differences are seen between the two samples in Fig. 6a, b, while in Fig. 6c, d the TL glow curves are identical within the accuracy of the experimental data. Similar results were obtained for the other samples studied, with differences in the TL glow curves disappearing as the samples are heated to higher T_{stop} temperatures.

The above results indicate that the activation energy of the shallow TL traps associated with the peak at 54°C is strongly affected by the varying PbO concentrations in the samples (Fig. 5b). On the other hand, the deeper TL traps above $\sim 90^\circ\text{C}$ shown in Fig. 6c, d stay largely unaffected by the PbO concentration of the samples, indicating that they are most likely associated with intrinsic traps in the silicate matrix. Such intrinsic traps in the silicate matrix would be expected to be present in these glasses and to behave in the same manner in all four samples.

3.3. Computerized glow curve fitting procedure

We have applied a computerized glow curve fitting procedure to the glow curves of Fig. 1, by assuming first-order

kinetics. The assumption of first-order kinetics was supported by the dose dependence data already presented in Fig. 2.

The intensity of the TL signal at temperature T is given by the well-known first-order expression

$$I(T) = n_0 s \exp\left(-\frac{E}{k_B T}\right) \exp\left[-\frac{s}{\beta} \int_{T_0}^T \exp\left(-\frac{E}{k_B T'}\right) dT'\right], \quad (1)$$

where n_0 is the concentration of trapped electrons at temperature T , β the constant heating rate used during measurement of the TL glow curve, T_0 the temperature at time $t = 0$, E the activation energy of the electron trap (eV), s the frequency factor for the trap s^{-1} , and k_B the Boltzmann constant.

The commercially available program PEAKFIT is used to find the best possible fit to the experimental data, with E , s and n_0 being the adjustable parameters. The values of the activation energy E obtained from the initial rise method are used as initial values in the least-squares algorithm used in PEAKFIT. The results of this fitting procedure are shown in Fig. 7, and the corresponding E , s and T_{max} values for the four samples studied are summarized in Table 2.

Typical values of the frequency factor s for electronic transitions taking place via the delocalized conduction band (or valence band in the case of holes), are of the order of $s = 10^7\text{--}10^{14}\text{ s}^{-1}$. The data in Table 2 show that the E -values for the 54°C TL peak are within these broad limits, while the two higher-temperature

Table 2

Results of applying a computerized glow curve analysis technique to the experimental TL glow curves for the 4 samples studied.

Peak	32%			42%			52%			62%		
	E (eV)	T_{max} ($^\circ\text{C}$)	S (s^{-1})	E (eV)	T_{max} ($^\circ\text{C}$)	S (s^{-1})	E (eV)	T_{max} ($^\circ\text{C}$)	S (s^{-1})	E (eV)	T_{max} ($^\circ\text{C}$)	S (s^{-1})
1	0.64	50	1.4E+09	1.13	53	7.3E+16	1.19	52	7.4E+17	0.69	56	5.5E+09
2	0.44	91	9.5E+05	0.88	79	6.6E+11	0.79	68	7.4E+10	0.35	89	4.6E+03
3	0.41	127	8.7E+03	0.53	110	7.9E+05	0.39	104	10418	0.36	126	1.9E+03
4	0.47	206	4.2E+03	0.49	195	9.8E+03	0.40	202	720	0.42	204	1.2E+03

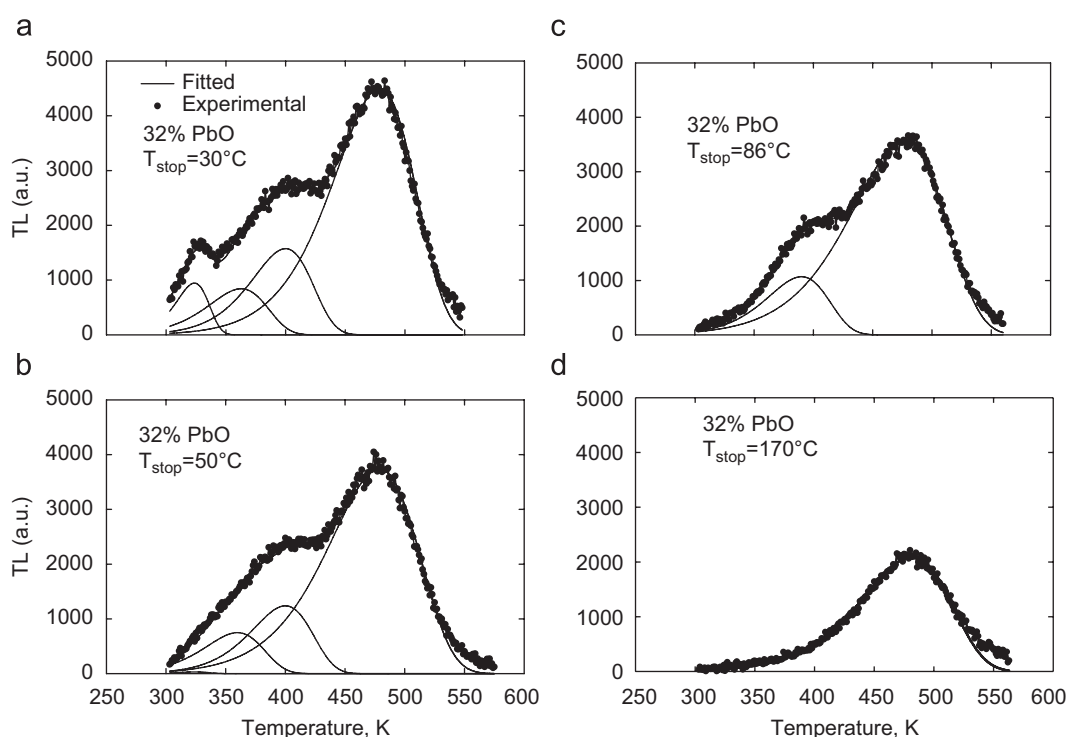


Fig. 8. The partial TL glow curves for the 32% PbO sample is fitted to a sum of first-order TL peaks.

TL peaks at 110 and 210 °C correspond to much lower s -values outside the above limits. The E -values for the intermediate 80 °C TL peak are outside the limits for the 32% and 62% samples, while they are inside these limits for the 42% and 52% samples.

McKeever [19, p. 113] and Chen and McKeever [20, p. 65] discussed how localized transitions can lead to very low values of the frequency factors. In such localized transitions the electron is thermally stimulated into an excited state below the conduction band, and subsequently recombines directly with a hole in the recombination center. The TL intensity due to localized transitions will be given by

$$I(T) = \left[\frac{vs}{v+s} \right] m \exp\left(-\frac{E_e}{k_B T}\right), \quad (2)$$

where E_e is the energy difference between the trap and the excited state, m the concentration of trapped holes, $v = mA_{me}$ is a constant probability (s^{-1}) and A_{me} representing the recombination probability for the electron in the excited state. The solution of Eq. (2) has the same form as a first-order TL peak, hence localized transitions are commonly assumed to involve first-order kinetics. Chen and McKeever [20, p. 65] discuss how such localized processes can lead to very low values of s , and therefore can produce very broad TL peaks even at high temperatures. Furthermore, they suggested that analysis of the experimental data in such localized processes by any standard method of analysis, will always lead to such very low values of s .

These authors further note that such high-temperature–low- s TL peaks can be produced not only by nearest-neighbor interactions, but also by overlapping wavefunctions for next-nearest neighbors, etc.; therefore under such circumstances one may expect to have a second and possibly a third such TL peak at higher temperatures. The experimental data presented for the binary silicate glasses in this paper seems to be consistent with the existence of at least two such broad TL peaks located at 110 and 210 °C, both involving localized transitions via an excited state located below the conduction band.

Fig. 8a–d shows typical results of the computerized curve fitting procedure for the 32% PbO sample, and for partial glow curves containing 4, 3, 2, 1 peaks correspondingly. The same E - and s -values are used to fit all four partial glow curves in Fig. 8. The results of Figs. 7 and 8 provide strong evidence for the presence of four first-order TL peaks in all four samples, and in all partial glow curves examined in this paper.

3.4. Isothermal decay of TL signal at different temperatures

In order to verify the unusually low E -values obtained for the higher-temperature TL peaks, a separate isothermal TL decay experiment was undertaken and is described in this section. The four glass samples were irradiated at a constant irradiation temperature with a dose of 12 Gy and the phosphorescence from the samples was measured immediately after the end of the irradiation for a time period of 500 s, by keeping the temperature of the sample constant. The experiment was repeated at different temperatures of 87, 104, 113, 121, 130 and 138 °C and the TL intensity is shown in Fig. 9a and b as a function of the time t elapsed after the end of the irradiation, for the six different temperatures used in this experiment. The temperatures are chosen so that the TL signal measured will correspond only to the higher-temperature TL peak at ~210 °C, with minimal contributions from the lower-temperature TL peaks at 54, 80 and 110 °C.

The graphs in Fig. 9a, b are fitted with a single-exponential law $I = I_0 e^{-\lambda t}$ and the fits are shown as solid lines. We conclude that the phosphorescence of this TL trap associated with the peak at 210 °C obeys an exponential decay law with a decay constant λ

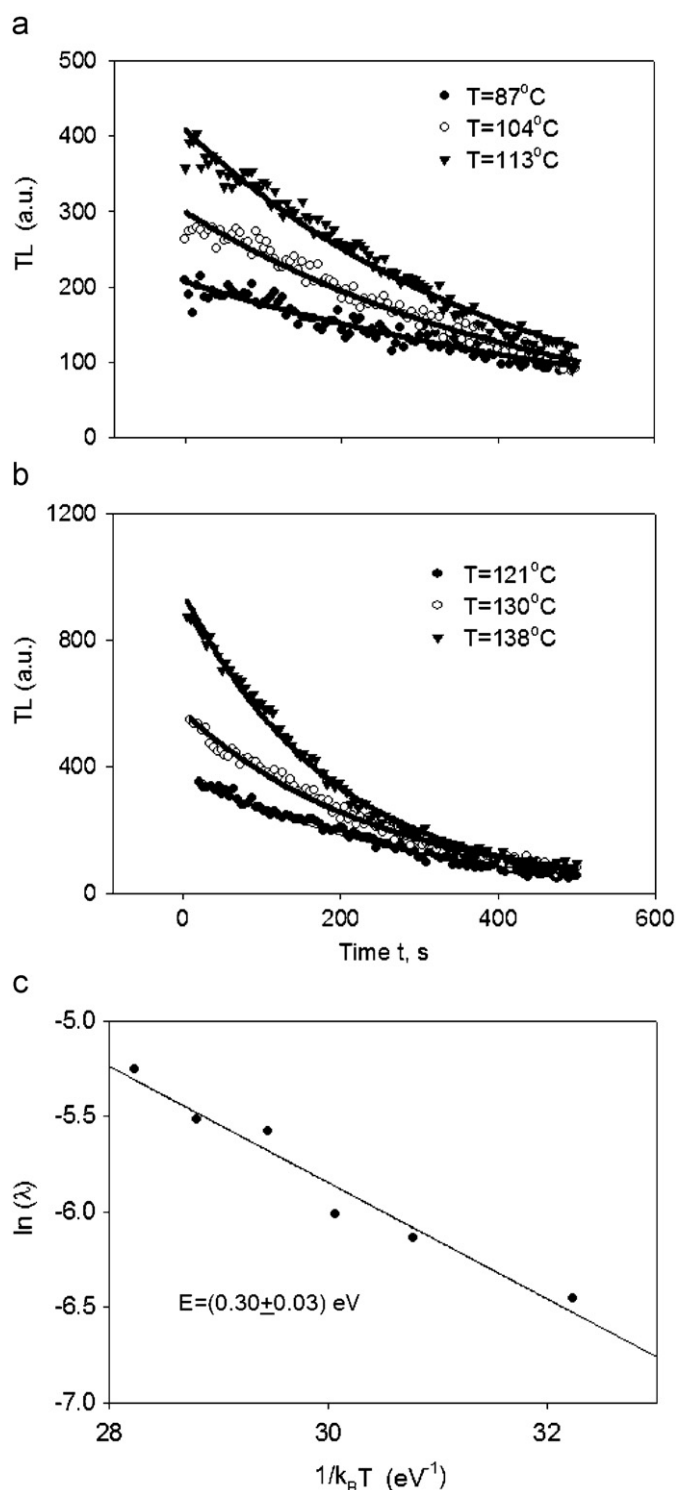


Fig. 9. (a) and (b) The results of the isothermal decay at temperatures of 87, 104 and 113, 121, 130 and 138 °C for the 52% PbO sample. The solid lines represent single exponential law fits to the experimental data. (c) The logarithm of the decay constant λ obtained by the exponential decay fits in (a) and (b) is plotted as a function of the inverse irradiation temperature $1/k_B T$. The slope gives the activation energy $E = 0.31 \pm 0.03$ eV for the TL peak at 210 °C.

(s^{-1}), consistent with the first-order kinetics assumption for the TL peaks.

Fig. 9c shows the logarithm of the decay constant λ obtained from the exponential fits as a function of the inverse irradiation temperature $1/k_B T$. The slope of this line gives the activation energy $E = 0.31 \pm 0.03$ eV for the TL peak at 210 °C. This value of E

is in agreement with the value of $E = 0.350 \pm 0.006$ eV obtained from the initial rise technique. It was not feasible to carry out reliably a similar isothermal decay analysis of the other three TL peaks present in the glow curves, because of the very strong overlapping between them.

We conclude that the results of the isothermal decay analysis are consistent with the TL peak at 210 °C corresponding to a localized transition.

4. Conclusions

The TL glow curves of four binary lead-silicate glasses with PbO concentrations ranging from 32% to 62% were found to contain four first-order peaks at 54, 80, 110 and 210 °C and were analyzed using a systematic thermal cleaning technique, the initial rise method and a computerized fitting procedure. Furthermore, the kinetic parameters of the high-temperature glow peak at 210 °C were verified using the phosphorescence decay method of analysis. The low-temperature TL peak at 54 °C exhibited very different behavior from the three higher-temperature peaks at 80, 110 and 210 °C. The activation energy of the TL peak at 54 °C was found to depend strongly on the PbO concentration of the samples, while the TL peaks at 110 and 210 °C show a behavior independent of the PbO concentration. The TL traps responsible for the 54 °C peak in the glow curves are most likely associated with the lead network developing for higher concentrations of PbO, and therefore depend strongly on these concentrations. However, the TL traps corresponding to the higher-temperature peaks are most probably associated with the common silicate matrix in these binary silicate glasses.

The calculated E - and s -values for the four TL peaks also support the conclusion that the 54 °C TL peak is associated with delocalized transitions taking place via the conduction band, while the higher-temperature TL peaks are associated with localized transitions taking place via an excited state below the

conduction band. Additional experimental evidence is required to ascertain these facts with complete confidence. Such experiments should examine the glow curves of the samples at much higher doses (in which case the statistical accuracy would be improved), and also the behavior of these glasses in a wider range of PbO concentrations.

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