**Particle temperature measurements in closed chamber detonations using
thermoluminescence from $\text{Li}_2\text{B}_4\text{O}_7\text{:Ag,Cu}$, $\text{MgB}_4\text{O}_7\text{:Dy,Li}$ and $\text{CaSO}_4\text{:Ce,Tb}$**

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Abstract

The present work describes the procedures and results from the first temperature measurements in closed chamber detonations obtained using the thermoluminescence (TL) of particles specifically developed for temperature sensing. $\text{Li}_2\text{B}_4\text{O}_7\text{:Ag,Cu}$ (LBO), $\text{MgB}_4\text{O}_7\text{:Dy,Li}$ (MBO) and $\text{CaSO}_4\text{:Ce,Tb}$ (CSO) were tested separately in a total of 12 independent detonations using a closed detonation chamber at the Naval Surface Warfare Center Indian Head Explosive Ordnance Disposal Technology Division (NSWC IHEODTD). Detonations were carried out using two different explosives: a high temperature plastic bonded explosive (HPBX) and a low temperature plastic bonded explosive (LPBX). The LPBX and HPBX charges produced temperatures experienced by the TL particles to be between ~550 – 670 K and ~700 – 780 K, respectively, depending on the shot. The measured temperatures were reproducible and typically higher than the thermocouple temperatures. These tests demonstrate the survivability of the TL materials and the ability to obtain temperature estimates in realistic conditions, indicating that TL may represent a reliable way of estimating the temperature experienced by free-flowing particles inside an opaque post-detonation fireball.

Keywords: thermoluminescence, thermometry, agent defeat tests, particle temperature sensors

1 INTRODUCTION

There is currently no technique able to measure the temperature experienced by free-flowing particles heated inside an opaque post-detonation fireball. This information is relevant, for example, to understand the efficacy of different explosive formulations in targeting biological agent stockpiles [1-6]. Current thermometry techniques, such as thermocouple thermometry [7, 8]), pyrometry [4, 8-10], atomic and molecular spectroscopy [1, 2, 4, 10], Coherent-Anti-Stokes-Raman Scattering [11], and thermographic phosphor thermometry [12-16], are limited to measurements at fixed points, along a line of sight, or as *in situ* probes. Moreover, techniques based on *in situ* emissions (e.g., atomic and molecular spectroscopy) are generally biased towards the brightest (i.e. hottest) particles in the outer regions of the fireball.

Thermoluminescence (TL) is one property among others proposed to enable *passive* particle temperature measurements of a post-detonation fireball [17-23]. Particles heated during a detonation are collected and analyzed in laboratory, providing *ex-situ* estimation of the maximum temperatures experienced by the particles during the entire process.

TL has a long history of application in the field of measurement of ionizing radiation (e.g. gamma rays, X-rays, neutrons, etc.) [24, 25]. In radiation measurements, detectors made of wide band-gap crystalline insulators with specific dopants (e.g. LiF:Mg,Ti, CaF₂:Dy, CaSO₄:Dy, Al₂O₃:C, Li₂B₄O₇:Mn [26]) are produced and packaged, for example, in personal dosimeters. Ionizing radiation creates charge carriers (electrons and holes) within the crystal lattice, which are trapped at localized energy levels within the crystal band-gap. This gives rise to a latent signal in the crystal, which can be subsequently read in laboratory by heating the detectors. The energy provided by heating releases the trapped charges, leading to electron-hole recombinations that result in the observed TL signal. The TL curve (TL signal versus temperature) typically

shows several peaks, related to trapping centers characterized by different thermal stabilities that are subsequently emptied as the detectors are heated. These peaks are not observed without exposure of the detectors to ionizing radiation, or if the detectors are heated to temperatures high enough to empty these trapping centers.

The concept of using TL for temperature measurements, described earlier in other contexts [27-29] and more recently in the context of particle thermometry [17, 18], is the reverse of the radiation measurement concept. For temperature measurements one starts with *irradiated* particles and exposes those to the temperature environment to be measured (e.g. a detonation). After that, the particles are collected and brought to the laboratory, where the TL curve is recorded under controlled heating. If, during the detonation, the particles were heated to a temperature insufficient to affect the trapped charges within the material, the TL curve will be similar to that of control particles, prepared in a similar way but not exposed to the detonation. If the particles were heated to a temperature sufficiently high, the TL curve will be altered to a degree that depends on the time-temperature profile. Using knowledge of the thermal stability of the trapping centers in the specific material, it is in principle possible to determine the temperature the particles experienced during the detonation.

The concept of particle thermometry using TL has been demonstrated using a commercial TL material developed for radiation dosimetry (LiF:Mg,Ti) [19]. Since then, considerable effort has been made to develop new TL materials specifically designed for temperature sensing, having a combination of several properties: high luminescence intensity, multiple TL peaks with different thermal stabilities covering a wide temperature range in the TL curve, and short-wavelength TL emission (blue, UV). Moreover, as opposed to radiation dosimetry, which

requires materials with effective atomic number (Z_{eff}) close to water, Z_{eff} is not relevant for temperature sensing [30].

Based on these considerations, three materials were developed for temperature sensing [31], $\text{Li}_2\text{B}_4\text{O}_7\text{:Ag,Cu}$ (LBO), $\text{MgB}_4\text{O}_7\text{:Dy,Li}$ (MBO) and $\text{CaSO}_4\text{:Ce,Tb}$ (CSO), and tested in laboratory conditions, i.e., slow temperature heating in oven (2 h duration) or relatively fast temperature heating by quick passage through a chamber (approximately 1 s duration) [32]. The results were consistent with thermocouple measurements, except in cases in which particle aggregation was observed. Although the study provided further demonstration of the concept of TL particle thermometry, the conditions were still far from the intended application in detonation events.

The objective of this study was to test the performance of different thermoluminescence (TL) materials as particle temperature sensors using a closed detonation chamber at the Naval Surface Warfare Center Indian Head Explosive Ordnance Disposal Technology Division (NSWC IHEODTD). Particles of $\text{Li}_2\text{B}_4\text{O}_7\text{:Cu,Ag}$ (LBO), $\text{MgB}_4\text{O}_7\text{:Dy,Li}$ (MBO), and $\text{CaSO}_4\text{:Ce,Tb}$ (CSO), or a mixture of them, were tested in 12 detonations (“shots”) using two different explosives, a high temperature plastic bonded explosive (HPBX) and a low temperature plastic bonded explosive (LPBX), in duplicate conditions when appropriate.

2 EXPERIMENTAL DETAILS

2.1 Materials

$\text{Li}_2\text{B}_4\text{O}_7\text{:Cu}_{0.4\%}\text{,Ag}_{0.1\%}$ (LBO), $\text{MgB}_4\text{O}_7\text{:Dy}_{0.1\%}\text{,Li}_{1\%}$ (MBO) and $\text{CaSO}_4\text{:Ce}_{0.2\%}\text{,Tb}_{0.2\%}$ (CSO) samples were prepared as described earlier [31]. LBO samples were sieved into two groups, one with $<38\text{ }\mu\text{m}$ mesh size and the other between 53 and 75 μm mesh sizes. MBO and CSO were

sieved with $< 125 \mu\text{m}$ mesh size. These materials consist of aggregate particles of tens of micrometers in diameter. The samples were irradiated with $\sim 500 \text{ Gy}$ using a Gamma Cell (Co-60 gamma radiation), placed in centrifuge vials, 2 g of material per vial, and mailed to NSWC IHEODTD in individually labeled black plastic bags, each containing one vial, to protect them from light. Irradiated particles were also kept in laboratory for control.

2.2 Closed-chamber tests

The closed chamber used for the tests consist of a 243-liter cylindrical steel chamber with an inner diameter of 23 inches and fitted with three thermocouples (Type R, model P13R-005 Omega Engineering Inc.) in different positions (Figure 1). The charges labeled LPBX or HPBX, chosen to achieve low and high temperatures, were placed at the center of the chamber. The particles ($\sim 2 \text{ g}$ per shot) were placed 6 inches from the charges. Additional measurements using three-color optical pyrometry were also performed. See further description of the closed chamber in Daniels et al. [6]. One of the shots (shot 7) was performed with $1/3$ of the LPBX charge [6].

2.3 TL measurements

The TL from the particles was measured at 1 K/s using a Risø TL reader (model TL/OSL-DA-15, Risø National Laboratory, Denmark) in the presence of N_2 gas. The TL was detected using a photomultiplier tube (model 9235QB, Electron Tubes, Inc.) and optical filters optimized for each material: 5 mm Hoya U-330 (Hoya Corporation) for LBO, 6 mm Schott BG-39 (Schott AG) for MBO, and 7.5 mm Hoya U-340 (Hoya Corporation) for CSO.

The samples (typically $< 1 \text{ mg}$) were deposited in stainless steel sample cups for the readouts. After the first readout, the samples were re-irradiated with a beta dose of $\sim 500 \text{ Gy}$ from

a $^{90}\text{Y}/^{90}\text{Sr}$ and the TL was read again to obtain the “regenerated” TL. Finally, the samples were heated again, this time without irradiation, to record the background due to blackbody radiation.

Control samples, which were irradiated but not exposed to the detonation, were also read and used for comparison with the samples exposed to the detonation.

2.4 TL analysis

Data analysis followed a procedure similar to that described in laboratory tests of LBO, MBO and CSO [32].

First, the TL curves from the control samples were used to obtain an estimate of the distribution of activation energies responsible for the TL signal in each material assuming a single frequency factor $s = 10^{14} \text{ s}^{-1}$. Then, this model was used to calculate the expected TL curves for samples heated to different temperatures Θ and durations τ . The simulated TL curves were compared to the experimental curves recorded for the samples exposed to different explosives using the parameter $\eta(\Theta, \tau)$ given by:

$$\eta(\Theta, \tau) = \frac{n}{\sqrt{\sum_i (y_i(\Theta, \tau) - y_{i,\text{exp}})^2}}, \quad (1)$$

where $y_i(\Theta, \tau)$ is the intensity of the TL curve predicted by the model for a sample heated to temperature Θ for a duration τ (at readout temperature T_i), $y_{i,\text{exp}}$ is the experimental intensity of the TL curve for the same readout temperature, and n is the number of data points. $\eta(\Theta, \tau)$ is calculated after normalization of both TL curves (simulated and experimental) to the maximum intensity. To account for possible temperature shifts in the TL curve due to different thermal contact, the minimum value of η when shifting the TL curves by $\pm 20 \text{ K}$ was taken as the result. The parameter $\eta(\Theta, \tau)$, as defined in Eq. (1), is large when there is good agreement between the

simulated and the experimental TL curves, indicating the temperature Θ and duration τ that would best explain the observed results.

As discussed earlier [32], it is not possible to make an independent estimation of the time and temperature to which the particles were exposed without a more sophisticated TL model for the materials. In this work, therefore, we assumed a constant heating profile of duration 0.1 s based on thermocouple data [6] and investigated the sensitivity of our solutions to this assumption (e.g. if the duration is varied or if an exponential decay is assumed for the temperature profile during the detonation).

3 RESULTS

3.1 TL curves

Figure 2 shows examples of TL curves obtained using the readout protocol. The as-received curve (black) corresponds to the TL curve of the sample under analysis (e.g., control or heated by detonation inside the chamber). For samples heated inside the detonation chamber, this is the curve used for the temperature determination. The “regenerated” TL (red curve) represents the TL curve after the same sample was re-irradiated in laboratory and indicates the amount of material contained in each sample cup. This information helps identify cases in which no TL is observed because of absence of TL material in the cup, as opposed to cases in which the material was heated to a high temperature. The background curve (green curve) is the TL with no irradiation, indicating the contribution from blackbody radiation and other possible spurious sources of light. This background was subtracted from the other curves.

Experimental TL curves for different shots are presented in Figure 3. Each graph shows the TL curve for the control sample as well as the TL curve for each shot. Since different

amounts of material are present in each cup, the TL curves were made comparable by normalizing them to the maximum intensity of the “regenerated” TL curve. This correction is not perfect because of factors such as non-uniform dose rate distributions and sample position, resulting in some variability in intensity.

Based on this data, several qualitative observations can be made. LPBX heated all material to temperatures within the range of applicability: the corresponding TL curves are visible and clearly different from the control samples. On the other hand, HPBX heated LBO and MBO to a temperature beyond their range of applicability, essentially erasing all TL. It is also interesting to note that in shot 7 (Figure 3a), performed with a 1/3 of the LPBX quantity of explosive, the temperature was in fact low, since the TL curve of LBO is similar to the control curve and very different from the other LPBX shots (Figure 3a). In shot 10, MBO was heated to a temperature lower than in shot 11, as evident in the TL curves (Figure 3b). CSO was suitable for temperature measurements for both LPBX and HPBX charges due to the wider temperature range of TL peaks in this material.

It should be pointed out that some samples showed still some emission from low-temperature peaks, even though the temperature experienced by the particles was sufficiently high to decrease the intensity of high temperature peaks. This can be evidence of non-uniform heating of the particle distribution (some particles were not heated to a temperature high enough), as will be discussed later (Section 3.3). Nevertheless, in these experiments we cannot exclude the possibility of contamination from previous shots.

The possibility of contamination with samples from previous shots is evidenced by the data in Figure 4. This figure shows an as-received curve typical for LBO (see Figure 3a), but the “regenerated curve” shows a combination of peaks from LBO and CSO, indicating residual

contamination from CSO used in shot 6. The samples from shot 6 also showed a large amount of residual contamination from LiF used in shot 5 for another study (data not shown). The possibility of contamination was also confirmed through discussions with the NSW IHEODTD group.

3.2 TL model

Based on the analysis of the TL curves for the control samples, we determined the distribution of activation energies of each sample [32]. The results are essentially identical to those found in Ref. [32], except for normal variations from batch to batch, and will not be repeated here.

The distribution of activation energies for each material allows us to calculate the expected TL curves following different heating temperatures Θ and durations τ . As an example, Figure 5 shows the simulated TL curves for LBO as a result of heating for 0.1 s at various constant temperatures.

Based on such TL curves, it is also possible to calculate the residual TL after heating to different temperatures. This is shown in Figure 6 for the three different materials and assuming a heating duration of 0.1 s. This data gives an idea of the range of applicability of these materials as temperature sensors for heating durations of this order of magnitude.

3.3 Temperature determination

To determine the temperature to which the particles were exposed, we compared the experimental TL curves with a large set of simulated TL curves similar to those shown in Figure 5, calculated for different temperatures Θ and durations τ , using the parameter η expressed in Eq. (1). The results are contour plots of η as a function of the heating temperature Θ and duration τ ,

as illustrated in Figure 7 for shot 12 (CSO, LPBX). In this figure it is possible to see that, assuming a heating duration of 0.1 s, the heating temperature that best explains the experimental TL curves is ~ 670 K.

The heating temperature can be seen more clearly if we plot only the profile corresponding to 0.1 s heating duration, as shown in Figure 8. This figure shows that the best agreement between simulated and experimental TL curves is obtained for 677 K heating (assuming 0.1 s heating duration). This figure is also an example of a well-behaved case, in which there is a clear agreement between the simulated and experimental TL curves.

If this analysis is applied to all samples and all shots, we obtain the temperatures shown in the fourth column in Table 1. For the mixed samples (MBO mixed with CSO), the TL measurements were carried out using both Schott BG-39 filters, which detects emission from both MBO and CSO, and Hoya U-340 filters, which detects emission from only CSO. We found the results obtained using the Schott BG-39 filters to be unreliable, due to the varying composition of MBO and CSO in each sample used for readout. For this reason, the results presented in Table 1 include results obtained using Hoya U-340 filters, which monitors only CSO samples.

Some shots presented in Table 1 were duplicated. Particles with grain size $< 38 \mu\text{m}$ and between $63\text{--}75 \mu\text{m}$ of LBO were tested to evaluate the influence of the grain size in the results, but the results were identical within the experimental uncertainties. Some samples were also analyzed after sieving ($125 \mu\text{m}$ mesh size) to remove debris. Again, the results were similar within experimental uncertainties.

Figure 9 shows examples of the experimental TL curves and the simulated TL curves for the best fit and for ± 10 K for each single shot. A temperature estimation can be obtained even

when the agreement between the experimental and simulated TL curves are not exact, as in shot 3 (Figure 9a) and shot 4 (Figure 9b). This is probably because each single TL peak is very sensitive to temperature in their range of applicability, changing very rapidly with small variations in temperature.

We noticed in some samples evidence of partial (non-uniform) heating. This is evident in shot 8 (Figure 9e), shot 9 (Figure 9f), and shot 11 (Figure 9h). In these cases the TL curves cannot be explained by particles heated to a single temperature. The profile of η versus the temperature Θ has a more complex structure, as shown in Figure 10. Some of these results may also be explained by sample contamination from different tests, as previously discussed in Figure 4.

Daniels et al. [6] compared the temperatures obtained in this study with thermocouple data and modeling. Figure 11 shows the temperatures estimated using TL *versus* the maximum thermocouple measurements. The TL temperatures are higher than the thermocouples data from NSWC IHEODTD due to the faster response time and proximity of the particles with the charge, but there is a clear correlation between the TL and thermocouple temperatures. Because of a lack of techniques for comparison, the validity of the temperatures obtained using TL must rely on indirect observations, such as the reproducibility of the results (Table 1), consistency with experimental conditions (Figure 11), and agreement with thermocouple data in laboratory conditions [32].

3.4 Sensitivity to analysis parameters

We evaluated the sensitivity of the results shown in Table 1 to various analysis parameters.

CSO samples corresponding to shot 4 were analyzed using distribution of activation energies obtained from different samples. The temperatures obtained using the different distributions varied only by 1%, indicating that the control samples resulted in a TL model with sufficient reproducibility.

TL curves from different samples were sufficiently reproducible. As shown in Table 1, the maximum experimental standard deviation of the data was 3.6%.

The TL curves are allowed to be shifted up to ± 20 K to account for possible systematic errors during the readout process (e.g., caused by bad contact between the sample cup and the heater in the Risø Reader). We repeated the analysis for CSO shot 4 reducing this degree of freedom and the variation was less than 1%.

One source of uncertainty in the analysis is the assumption on the heating duration. We analyzed CSO shot 4 using different assumptions for the heating duration τ . Figure 12 shows the best temperature estimation for various heating durations τ . The variation in temperature is only 5% when the time-scale is five times faster or five times slower than the time-scale used. Therefore, the data indicates that the time-scale does not need to be known with high precision.

We also analyzed the influence of replacing the constant temperature model (heating to a constant temperature Θ for duration τ) with an exponential decay model (instantaneous heating to temperature Θ_m , followed by exponential decay to room temperature with time constant τ), as represented by the following equation:

$$\Theta = \Theta_0 + (\Theta_m - \Theta_0)e^{-t/\tau}, \quad (2)$$

where Θ_0 is the room temperature. We calculated for shot 4 (CSO, HPBX) the maximum temperature Θ_m that would result in the best agreement between simulated and experimental TL curves for various values of τ .

Figure 13 shows the maximum temperature Θ_m estimated for various exponential decay time constants τ . The data show that, for a decay constant of $\tau \sim 2$ s, the exponential decay model agrees with the constant temperature model with a 0.1 s heating duration, as presented in Table 1. If the heating occurred faster (i.e., for lower τ), the temperature estimates can increase by as much as 10%. These results indicate that reasonable temperature estimations can be obtained without a highly precise estimate of the time constant τ . It also provides an equivalency between a pulse constant temperature heating and an exponential decay heating.

4 CONCLUSIONS

The tests of LBO, MBO and CSO in closed chamber detonations at NSWC IHEODTD demonstrated that the particles survived the detonation event and the luminescence from the collected particles was sufficient for temperature determination. The temperatures obtained were reproducible and consistent with the experimental conditions, typically higher than the thermocouple temperatures, as expected. The LPBX charges produced temperatures experienced by the TL particles to be between $\sim 550 - 670$ K, depending on the shot (this excludes shot 7, which used a smaller charge and resulted in heating only to ~ 470 K). The HPBX charges produced temperatures determined to be between $\sim 700 - 780$ K, again depending on the shot. Variations seem to be caused by real differences in heating inside the chamber from shot to shot, since the TL curves for the different shots are clearly different in shape.

In spite of cross-contamination observed, the analysis procedure was relatively robust, not strongly influenced by variations in the TL model, sample, shift of the TL curve in temperature, and timescale of the heating event. This indicates that TL may represent a reliable

way of estimating the temperature experienced by the particles inside an opaque post-detonation fireball.

The results described here provide further demonstration of the potential of the TL technique for particle temperature sensing in detonations, filling an important gap in diagnostic techniques for this type of study. The next step would be to correlate TL temperatures with bioagent inactivation.

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TABLE AND FIGURE CAPTIONS

Table 1. Results from TL analysis assuming uniform heating by a pulse of 0.1 s of duration. The uncertainties are the experimental standard deviation of the data based on five TL curves obtained for each shot, therefore representing only the sample to sample variation.

Figure 1. Diagram of closed detonation chamber used in this study, showing the placement of the charge (C), the samples (S), the thermocouples (TC1, TC2 and TC3) as well as the optical window for other diagnostics and pressure gauges (P1 and P2).

Figure 2. Examples of TL curves obtained as part of the readout protocol: first the sample is read to obtain the “as-received signal”, then it is re-irradiated with ~ 500 Gy to obtain the “regenerated signal”, and finally it is read again to measure the background (BG). This particular sample is a control of LBO.

Figure 3. TL curves obtained for the different shots. The curves are the average of five samples after normalization to the maximum TL of the regenerated curve. Shot 7 was carried out with 1/3 of the amount of explosive in shots 8 and 9.

Figure 4. Example of TL curves for LBO shot 7, indicating contamination of the material in the regenerated signal.

Figure 5. TL curves for LBO for different heating temperatures (assuming a heating duration of 0.1 s), calculated based on the distribution of activation energy obtained for each material.

Figure 6. Residual TL according to model for 0.1 s heating duration.

Figure 7. Example of contour plot of the parameter η as a function of the heating temperature Θ and duration τ (in logarithm scale) for one CSO sample from shot 12. The light yellow region separating the light and dark blue regions indicate the maxima of η , which represents the best agreement between the simulated and experimental TL curves.

Figure 8. Value of η as a function of heating temperature Θ for a constant heating duration of 0.1 s (CSO, shot 12). The best solution in this case is 677 K.

Figure 9. Comparison between the experimental TL and the simulated TL for the best temperature (T) estimate and for the same temperature ± 10 K, shown for examples of each shot.

Figure 10. Example of “unusual” profile of η versus heating temperature Θ . The data is for a sample of shot 8 (LBO, LPBX).

Figure 11. Correlation between temperatures obtained using TL and the maximum thermocouple (TC) temperatures measured by the NSW IHEODTD group [6].

Figure 12. Variation in the temperature Θ as a function of heating duration, obtained based on the TL curves from four samples of CSO shot 4.

Figure 13. Maximum temperature Θ_m as a function of time constant τ for particles subjected to an exponential decay temperature profile, based on data from four samples of CSO shot 4. The dashed line shows the temperature obtained assuming heating to a constant temperature for a 0.1 s duration.

Table 1

Shot	Charge	Material	T^(a) (K)	Observations
1	HPBX	LBO	NA	Beyond material range
2	HPBX	MBO	NA	Beyond material range
3	HPBX	CSO	723 ± 12	
3	HPBX	CSO sieved	732 ± 27	
4	HPBX	CSO	703 ± 13	
4	HPBX	CSO sieved	717 ± 21	
6	HPBX	Mixed (CSO) ^(b)	782 ± 31	Contamination from LiF
7	LPBX*	LBO	468 ± 2	Contamination
8	LPBX	LBO (53 – 75 µm)	618 ± 5	Evidence of partial heating
9	LPBX	LBO (<38 µm)	626 ± 1	Evidence of partial heating
10	LPBX	MBO	548 ± 4	Evidence of partial heating
11	LPBX	MBO	607 ± 4	Evidence of partial heating
12	LPBX	CSO	667 ± 8	
13	LPBX	Mixed (CSO) ^(b)	663 ± 5	

*lower explosive mass

^(a)Temperatures estimated using TL. The uncertainty refers only to the variation obtained for different aliquots of the same sample.

^(b)Read using Hoya U-340 filter to select CSO emission.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Figure 12

Figure 13