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ORIGINAL ARTICLE

Analytical Expressions for the Mixed-Order Kinetics Parameters of TL Glow Peaks Based on the two Heating Rates Method

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Abstract

The two heating rates method (originally developed for first-order glow peaks) was used for the first time to evaluate the activation energy (E) from glow peaks obeying mixed-order (MO) kinetics. The derived expression for E has an insignificant additional term (on the scale of a few meV) when compared with the first-order case. Hence, the original expression for E using the two heating rates method can be used with excellent accuracy in the case of MO glow peaks. In addition, we derived a simple analytical expression for the MO parameter. The present procedure has the advantage that the MO parameter can now be evaluated using analytical expression instead of using the graphical representation between the geometrical factor and the MO parameter as given by the existing peak shape methods. The applicability of the derived expressions for real samples was demonstrated for the glow curve of $Li_2B_4O_7$:Mn single crystal. The obtained parameters compare very well with those obtained by glow curve fitting and with the available published data.

Keywords Mixed-order kinetics · Kinetics parameters · $Li_2B_4O_7$:Mn single crystal · Two heating rates method

Introduction

Kinetic analysis provides valuable information about the trapping levels within the crystal. Each trapping level is characterized by a set of trapping parameters depending on the model used for analysis. In the case of general-order (GO) kinetics, these parameters include the activation energy, the frequency factor and the order of kinetics. In this regard, several methods have been proposed to evaluate these parameters based on the GO kinetics expression [1–4]. However, the empirical nature of the GO expression has motivated the development of the more physical mixed-order (MO) model. Chen et al. [5] suggested the use of the MO model to describe the cases of intermediate kinetic order. Sunta et al. [6] have applied the two expressions to synthetic TL glow peaks derived from three physical models and concluded that the MO expression is a better alternative to the GO model in the description of experimental TL glow peaks. Despite of this, the use of the MO expression to evaluate the trapping parameters is rather limited, possibly because the MO expression is more complicated when compared with the GO expression. This complexity reflects itself in the limited number of methods that are based on the MO expression to extract the trapping parameters from experimental glow peaks. These methods are limited to peak shape methods and curve fitting [5, 7–12]. Therefore, the need for new methods to evaluate the trapping parameters from glow peaks obeying MO kinetics is of utmost important. This is in one hand allows inter-comparison between different methods and gives credibility for the obtained values of the trapping parameters. On the other hand, deriving analytical expression for the trapping parameters based on the original MO equation should be a better alternative to the methods that use semi-empirical expressions for the activation energy and a graphical plot for the mixed order parameter α (e.g. peak shape methods). In the present work we proposed that the two heating rates method originally developed for first-order glow peaks [13, 14] can be safely used to determine the activation energy from glow peaks obeying MO kinetics. The advantage of this method is that it only uses the temperature of peak maximum, which can be easily determined from the experimental peak with good accuracy. In addition, the method is simple and has proven excellent accuracy in the case of first- and general order glow peaks. The validity of the method was proved by analyzing numerically computed MO peaks. In

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addition, the mixed-order parameter α was calculated, for the first time, using analytical expression instead of the graphical representation between the geometry factor μ_g and α as given by the peak shape methods. The advantage of such expression is obvious since the μ_g versus α graph was constructed by taking the average of many combinations of the trapping parameters. The relative concentration of electrons at deep traps and the pre-exponential factor were also calculated. The derived expressions were applied to the experimental TL glow curve of $Li_2B_4O_7$:Mn single crystal. The obtained parameters show good agreement with those obtained by curve fitting and the available published data on this material.

The two Heating Rates Method

Randall and Wilkins suggested the following equation to describe the TL intensity [15]:

$$I = -\frac{dn}{dt} = ns\,\exp(-E/kT) \tag{1}$$

where E is the activation energy, s is the frequency factor, n is the concentration of trapped electrons, $k = 8.617 \times 10^{-5}$eV/K is the Boltzmann constant and T is the absolute temperature.

The solution of the above equation for a constant heating rate β is given by:

$$I = sn_o\exp(-E/kT)\exp\left[(-s/\beta)\int_{T_o}^{T}\exp\left(-E/kT'\right)dT'\right] \tag{2}$$

The condition of maximum intensity is obtained by differentiating Eq. (2) and equating the derivative to zero. From this condition one gets:

$$\beta E/kT_m^2 = s\,\exp(-E/kT_m) \tag{3}$$

where T_m is the temperature of the maximum intensity.

The two heating rates method makes use of Eq. (3) by heating the sample at two heating rates and noting the shift in the temperature of peak maximum. When Eq. (3) is written twice for β_1 and β_2 and dividing the two equations one by the other one gets the following expression for E [13, 14]:

$$E = [(kT_{m1}T_{m2})/(T_{m1}-T_{m2})]1n\left[(\beta_1/\beta_2)(T_{m2}/T_{m1})^2\right] \tag{4}$$

where T_{m1} and T_{m2} are the temperatures of peak maximum at β_1 and β_2 respectively.

Since this method was strictly proved for the first-order glow peaks, we will call the activation energy obtained by this method E_{FO}.

The Mixed-Order Kinetics Expression

The phenomenological model from where the MO kinetics expression was derived assumes one type of active traps, one type of recombination centers and thermally disconnected deep traps (TDDT) assumed to be at one level. The model further assumes that the concentration of electrons in the TDDT traps remains constant during the TL emission. The TL intensity is given by [16, 17]:

$$I = ns'(n+h)\exp(-E/kT) \tag{5}$$

where s' is the pre-exponential factor and h is the concentration of trapped electrons at the deep traps.

The solution of Eq. (5) for a constant heating rate (β) is given by [16, 17]:

$$I(T) = \frac{s'h^2\alpha\exp\left[\left(hs'/\beta\right)\int_{T_0}^{T}\exp\left(-E/kT'\right)dT'\right]\exp(-E/kT)}{\left[\exp\left\{(hs'/\beta)\int_{T_0}^{T}\exp(-E/kT')dT'\right\}-\alpha\right]^2} \tag{6}$$

where $\alpha = [n_o/(n_o + h)]$ and n_o is the initial concentration of trapped electrons at the active traps.

Expressions for the Trapping Parameters

The Activation Energy E

The condition of maximum intensity is obtained by equating the derivative of the TL intensity in Eq. (6) to zero. From this condition one gets [7, 10]:

$$E/kT_m^2 = \left(s'h/\beta\right)Z_m\exp(-E/kT_m) \tag{7}$$

where Z_m is given by:

$$Z_m = \frac{\exp\left[\left(hs'/\beta\right)\int_{T_0}^{T_m}\exp\left(-E/kT'\right)dT'\right]+\alpha}{\exp\left[(hs'/\beta)\int_{T_0}^{T_m}\exp(-E/kT')dT'\right]-\alpha} \tag{8}$$

When Eq. (7) is written twice for β_1 and β_2 and dividing the two equations one by the other we get the following expression for the activation energy:

$$E = [(kT_{m1}T_{m2})]/(T_{m1}-T_{m2})\Big]\,1\mathrm{n}\left[(Z_{m2}/Z_{m1})(\beta_1/\beta_2)(T_{m2}/T_{m1})^2\right] \tag{9}$$

where Z_{m1} and Z_{m2} are given by Eq. (8) with T_m is replaced by T_{m1} and T_{m2} respectively; T_{m1} and T_{m2} are the temperatures of peak maximum when β_1 and β_2 are used.

The activation energy in Eq. (9) is based on the MO expression, hence we will call the activation energy obtained by this method E_{MO}.

After rearrangement Eq. (9) can be written as:

$$E_{MO} = E_{FO} = \Delta E \tag{10}$$

where

$$\Delta E = [(kT_{m1}T_{m2})/(T_{m1}-T_{m2})]1n(Z_{m2}/Z_{m1}) \tag{11}$$

Equation (10) clearly shows that the activation energy obtained by the two heating rates method from glow peaks following first-order or mixed-order kinetics differ by ΔE. The difference in the activation energy given by Eq. (11) will be evaluated by analyzing numerically computed MO glow peaks covering a wide range of the trapping parameters (see below).

The Mixed-Order Parameter α

The condition of maximum intensity given by Eq. (7) is not helpful in determining any of the trapping parameters since Eq. (7) contains all the parameters that cover the TL intensity of MO glow peaks. Instead, the condition of maximum intensity is obtained by finding (dI/dT) or more easily ($d1n(I)/dT$) directly from Eq. (5) to get:

$$\frac{\mathrm{d}1\mathrm{n}(I)}{dT} = \frac{dn/\mathrm{d}T}{n} + \frac{\mathrm{d}n/\mathrm{d}T}{n+h} + \frac{E}{kT^2} \tag{12}$$

Substitute $\mathrm{d}n/\mathrm{d}T = \mathrm{d}(n/\mathrm{d}t)(\mathrm{d}t/\mathrm{d}T) = -I/\beta$ in the above equation to get:

$$\frac{\mathrm{d}1\mathrm{n}(I)}{dT} = \frac{-I}{\beta n} - \frac{I}{\beta(n+h)} + \frac{E}{kT^2} \tag{13}$$

Equate the derivative to zero at peak maximum (at$T = T_m$) and make use of the relation $\alpha = [n_o/(n_o+h)]$ to get after a little algebra:

$$\alpha^{-1} = 1 + \frac{2n_m I_m kT_m^2 - n_m^2 \beta E}{n_o\left(n_m \beta E - I_m kT_m^2\right)} \tag{14}$$

where I_m is the maximum intensity at $T = T_m$ and $n_m = n(T = T_m)$ is related to the area of the high temperature half of the peak (a_m) through the relation:

$$n_m = \frac{1}{\beta}\int_{T_m}^{\infty} I\, dT = \frac{a_m}{\beta} \tag{15}$$

In terms of the areas a_o (total area) and a_m, Eq. (14) reads:

$$a^{-1} = 1 + \frac{2a_m I_m kT_m^2 - a_m^2 E}{a_o\left(a_m E - I_m kT_m^2\right)} \tag{16}$$

Once E is determined using the two heating rates method, the MO parameter can be determined using Eq. (16) since the quantities I_m, T_m, a_m and a_o can be easily determined from the experimental glow peak with good accuracy.

The Relative Concentration of Electrons at Deep Traps *h*/n$_o$

The relative concentration of trapped electrons at the deep traps, h/n_o, may be not important by itself but it is crucial for determining the value of the pre-exponential factor. The obtained value of α is used to determine the ratio h/n_o through the relation $h/n_o = [(1 - a)/a]$. Note that the ratio h/n_o is solely depends on the MO parameter.

The Pre-Exponential Factor S′

With E and h/n_o are known, the pre-exponential factor, s', can be determined when Eq. (5) is evaluated at $T = T_m$ and make use of Eq. (15). The s' is given by:

$$S' = \frac{\beta^2 I_m \exp(E/kT_m)}{a_o a_m[(a_m/a_o) + (h/n_o)]} \tag{17}$$

Results and Discussion

Generating the Synthetic Mixed-Order Glow Peaks

Numerically computed MO glow peaks based on the MO expression (Eq. (6)) were produced using different combinations of the trapping parameters. All calculations were performed using the commercial program *Mathematica*. The synthetic peaks were generated with a small temperature increment of 0.05 K to accurately determine the temperature of peak maximum. The trapping parameters were varied to obtain peaks of different shapes and peak maxima within the temperature range 200–700 K. The input parameters that are used to generate the MO peaks are listed in Table 1. In addition, Table 1 shows the temperatures of peak maximum at the heating rates of 1 and 5 K/s.

Evaluating the Ratio Z$_{m2}$/Z$_{m1}$ and ΔE

The purpose of generating the MO glow peaks is to evaluate the ratio Z_{m2}/Z_{m1} and hence the difference in the activation energy ΔE. The integral that appears in Eq. (8) was calculated numerically using the commercial program *Mathematica*.

Table 1 shows the calculated values for the ratio Z_{m2}/Z_{m1} and for ΔE. It is obvious that the ratio is very close to unity and ΔE is on the scale of a few meV. This small value is below the accuracy of the methods devised to evaluate the activation

Table 1 The input parameters used to generate the synthetic mixed-order glow peaks together with the temperatures of peak maximum at the heating rates of 1 and 5 K/s. The calculated ratio Z_{m2}/Z_{m1}, ΔE and the determined activation energy using Eq. (4) appear in the last three columns

No.	Input parameters				T_{m1} (K)	T_{m2} (K)	Z_{m2}/Z_{m1}	ΔE(eV)	E(eV)
	E(eV)	α	s' ($cm^3\ s^{-1}$)	h					
1	0.6	0.7	5×10^6	5×10^2	276.35	293.65	1.0033	−0.0013	0.601
2	0.8	0.1	1×10^8	1×10^5	288.25	302.45	1.0007	−0.0004	0.801
3	0.9	0.5	3×10^4	2×10^5	403.40	428.05	1.0013	−0.0008	0.900
4	1.0	0.6	1×10^7	1×10^6	350.20	366.95	1.0023	−0.0015	1.002
5	1.1	0.2	1×10^8	2×10^6	360.30	376.45	1.0006	−0.0004	1.101
6	1.2	0.99	5×10^8	8×10^3	386.40	403.45	1.0011	−0.0009	1.200
7	1.3	0.8	1×10^{10}	6×10^5	375.85	390.75	1.0013	−0.0011	1.301
8	1.4	0.3	1×10^6	4×10^4	585.20	618.55	1.0011	−0.0010	1.402
9	1.6	0.03	1×10^5	5×10^6	619.15	651.90	1.0001	−0.0001	1.600
10	1.8	0.4	5×10^{11}	1×10^5	504.65	524.10	1.0000	0.0000	1.797

energy from TL glow peaks. Therefore, for all practical purposes the value of the activation energy of MO glow peaks can be safely obtained using Eq. (4). The calculated values of the activation energy (last column of Table 1) using Eq. (4) are in excellent agreement with the input values. The percentage error in *E* for the analyzed samples when Eq. (4) is used is less than 0.2%.

Note that the two heating rates method was originally developed to determine the activation energy from first-order TL glow peaks [13, 14]. Gartia et al. [18] and Rasheedy and Abd-Elmageed [19] showed that the two heating rates method can be used to obtain the activation energy from GO glow peaks with errors similar to that encountered experimentally when analyzing TL peaks. In the present work, we showed that the method can safely be used in the case of MO glow peaks with errors below the accuracy of the methods of TL analysis. It seems that the two heating rates is quite general and can be used to determine the activation energy regardless of the physical model from where the TL equation was obtained.

Applying the Method to the Glow Curve of $Li_2B_4O_7$:Mn

The trapping parameters of $Li_2B_4O_7$:Mn single crystal were evaluated using the expressions derived above. The TL glow curves were collected at the heating rates of 1, 2, 5 and 10 K/s [20]. The experimental details regarding samples preparation, measurements and analysis can be found in [20]. The $Li_2B_4O_7$:Mn single crystal shows a simple glow curve consisting of apparently two well isolated glow peaks as illustrated in Fig. 1. To avoid large errors in the determined values of the trapping parameters one must correct for the temperature lag between the heating element and the emitting sample especially when high heating rates are involved. In this regard, Kitis et al. [21] showed that for heating rates up to 2 K/s the temperature lag is negligible, and for some samples the temperature lag exceeds 90 K at a heating rate of 20 K/s. Therefore, it becomes necessary to correct for the temperature lag before any attempt to evaluate the trapping parameters. Kitis et al. [22] derived the following equation to correct for the temperature lag:

$$T_m^j = T_m^i - c\ln\left(\frac{\beta_i}{\beta_j}\right) \qquad (18)$$

where T_m^i and T_m^i are the maximum temperatures of a glow peak with heating rates β_j and β_i respectively and c is a constant. Eq. (18) takes into account only the peak maximum which is sufficient in our case because this is the only temperature required to apply Eq. (4). The constant c is determined using two low heating rates with negligible temperature lag (i.e. the experimental T_m values are assumed to represent the actual ones). The value of c is then inserted in Eq. (18) to determine the temperature of peak maximum at high heating rates relative to the lowest heating rate. In our case, the heating

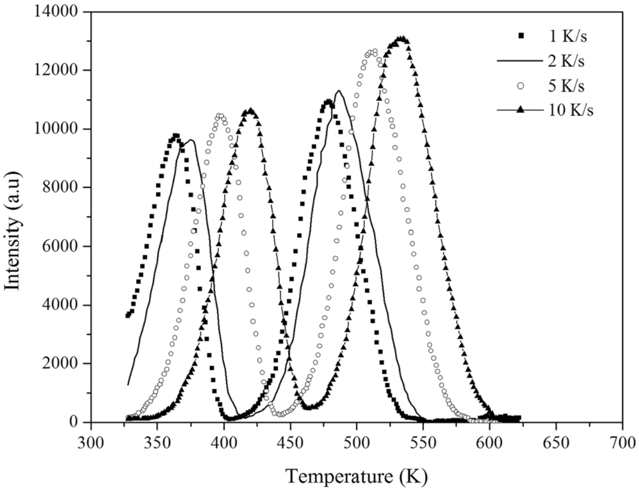

Fig. 1 TL glow curves of $Li_2B_4O_7$:Mn single crystal at different heating rates. The curves are not corrected to the temperature lag

Table 2 The determined values of the activation energy for peak I and peak II using all combinations of the heating rates. The average value of E for each peak is also given together with the standard deviation (±sd). The temperatures of peak maximum are corrected to the temperature lag according to Eq. (18)

		Peak I			Peak II		
β_1(K/s)	β_2(K/s)	T_{m1}(K)	T_{m2} (K)	E(eV)	T_{m1}(K)	T_{m2} (K)	E(eV)
1	2	363	375	0.61	478	488	1.31
1	5	363	391	0.64	478	501	1.36
1	10	363	403	0.66	478	511	1.38
2	5	375	391	0.66	488	501	1.40
2	10	375	403	0.68	488	511	1.42
5	10	391	403	0.72	501	511	1.44
				E_{av}±sd = 0.66±0.04			E_{av}±sd = 1.39±0.05

rates of 1 and 2 K/s were used (negligible temperature lag) to determine the value of the constant c. The constant c was calculated for peak I and peak II and equals to 17.31 and 14.43 respectively. The determined value of the constant c was used to estimate the temperature of peak maximum at the heating rates 5 and 10 K/s relative to the maximum temperature at 1 K/s. At 5 K/s peak I was shifted from 398 to 391 K while peak II was shifted from 514 to 501 K. At 10 K/s peak I was shifted from 420 to 403 K while peak II was shifted from 535 to 511 K. This procedure significantly reduces the error in the determined value for E as a result of the temperature lag, but does not totally eliminate its effect. After the correction for the temperature lag was taken into account, the trapping parameters were determined in sequence. The activation energy was determined using Eq. (4). Note that, the activation energy was determined for all combinations of the heating rates as seen in Table 2. The obtained values for E show a gradual increase with the heating rate (about 18% for peak I and 10% for peak II). This artifact is due to the small error involved in locating the correct position of the peak maximum when Eq. (18) is used. At a heating rate of 10 K/s, Eq. (18) predicts the correct value of T_m within 2 degrees which amounts to about 10% error in E when the two heating rates method is used. Therefore, the most trusted values of the activation energy are those when pairs of two low heating rates are employed.

The rest of the parameters were determined for the glow curve at 2 K/s. However, it is better to evaluate these parameters at the heating rate of 1 K/s, but the missing initial part of the first peak preclude us from evaluating the total area under this peak. The average value of E was then inserted in Eq. (16) to determine α. The relative concentration of trapped electrons at the deep traps h/n_o was determined through the relation $h/n_o = [(1-\alpha)/\alpha]$. Finally, the pre-exponential factor s' was evaluated using Eq. (17).

The obtained values of the trapping parameters for $Li_2B_4O_7$:Mn single crystal are summarized in Table 3. The extracted parameters using the present procedure with β =2 K/s were used to generate the theoretical peaks according to Eq. (6). Figure 2a shows the experimental glow curve and the generated curve using the calculated parameters. A good match between the first peak of the experimental curve and the generated peak was obtained. For the high temperature peak/complex there is a mismatch between the two peaks especially at the high temperature side which indicates that this complex may contain more than one peak. To check this possibility, a glow curve fitting was applied to fit the

Table 3 The calculated values of α, h/n_o and s' for the glow curve $Li_2B_4O_7$:Mn single crystal with β = 2 K/s. The quantities used in the calculations are: $a_o = 4.4 \times 10^5$, $a_m = 1.91 \times 10^5$, $I_m = 9.63 \times 10^3$, $T_m =$ 375 K for peak I and $a_o = 6.32 \times 10^5$, $a_m = 3.21 \times 10^5$, $I_m = 1.13 \times 10^4$, $T_m = 488$ K for peak II. The curve fitting values are also given when the glow curve was fitted with two or three peaks

	Present procedure				Fitting results using two peaks			
Peak no.	E(eV)	α	h/n_o	s'	E(eV)	α	h/n_o	s'
1	0.66	0.17	4.9	63	0.63	0.11	8.05	25
2	1.39	0.96	0.04	9.2×10^7	1.28	0.87	0.14	5.7×10^6
					Fitting results using three peaks			
1					0.63	0.11	8.05	25
2					1.41	0.94	0.05	2.2×10^8
3					1.44	0.19	4.1	8.9×10^7

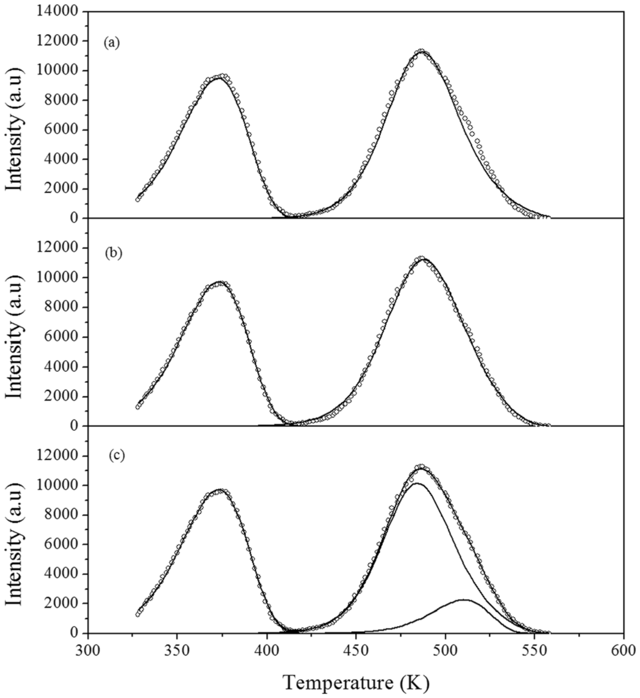

Fig. 2 TL glow curves of $Li_2B_4O_7$:Mn single crystal at 2 K/s (open circles) fitted with (**a**) the calculated parameters using the present procedure, (**b**) curve fitting using two peaks and (**c**) curve fitting using three peaks. The fitting parameters are listed in Table 3

experimental curve. In the first step, the high temperature complex was fitted with one peak (fig. 2b). Although the fitting was improved, a mismatch at the initial raise of the peak and at the peak maximum is visible. In the next step, the high temperature complex was fitted with two peaks (fig. 2c) where a very good match was obtained. The fitting parameters are shown in Table 3. For peak I, the obtained parameters using the derived expressions compare very well with the fitting results. For the high temperature complex, the obtained parameters are in very good agreement with the main peak of the complex when the high temperature complex was fitted with two peaks.

The MO parameter for the high temperature complex has a value of 0.96 which corresponds to order of kinetics b ~1.9 in the case of GO kinetics [8]. Ekdal et al. [20] evaluated the trapping parameters of the high temperature complex (assumed single peak) using several methods. They found that the activation energy falls in the range 1.18–1.24 eV depending on the method applied for analysis, and an order of kinetics equal to1.8 using the isothermal decay and 2 using the peak shape methods. Their values for E are lower than the values obtained by the present method possibly because they treated the high temperature complex as a single peak. On the other hand, their results are in good agreement with the fitting results when the high temperature complex was fitted with one peak.

Conclusions

In the present work we showed that the two heating rates method can be applied to glow peaks obeying MO kinetics to evaluate the activation energy. In addition, the MO parameter was calculated for the first time via an analytical expression which uses quantities that can be determined from the experimental curve with good accuracy. In order to apply this method, we suggest using low heating rates (less than 2 K/s) to avoid the temperature lag. The advantage of the present method is that it only uses the value of maximum temperature in evaluating the activation energy. The present work may encourage researchers to use the physically meaningful MO kinetics expression instead of the purely empirical GO kinetics expression in evaluating the trapping parameters.

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