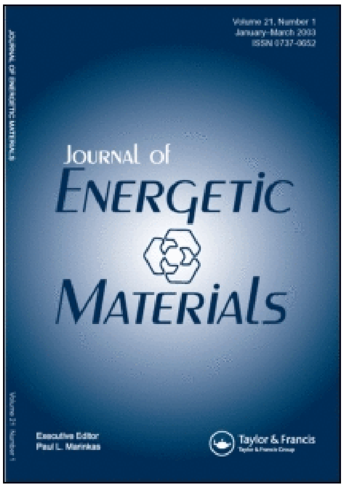

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A Simple Method to Estimate the Critical Temperature of Thermal Explosion for Energetic Materials Using Nonisothermal DSC

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A method for estimating critical temperature (T_b) *of thermal explosion for energetic materials was derived from Semenov's [9] thermal explosion theory and the non-isothermal kinetic equation* $\frac{d\alpha}{dt} = A_0 \exp(bT) f(\alpha)$ *based on Berthelot's expression using reasonable hypotheses. The final formula is* $T_b = T_{e0} + \frac{1}{b}$, *which is simple. We can easily obtain the onset temperature* (T_{ei}) *from the non-isothermal DSC curves, the value of* T_{e0} *from the equation* $T_{ei} = T_{e0} + a_1\beta_i + a_2\beta_i^2 + a_3\beta_i^3$, *the values of* b *from the equation* $\ln \beta_i = \ln\left[\frac{A_0}{bG(\alpha)}\right] + bT_i$, *and then calculate the value of* T_b. *The result obtained with this method coincides completely with the value of* T_b *obtained by Zhang et al.'s [4] method.*

Keywords: critical temperature, DSC, energetic materials, nonisothermal, thermal explosion

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Introduction

The critical temperature of thermal explosion (T_b) is a very important parameter for energetic materials (EMs). Much research based on the Arrhenius equation has been done in this area [1–8]. However, the estimation of the value of T_b based on Berthelot's equation using a nonisothermal analysis method has not been reported so far. The aim of this work is to present a new method for estimating the value of T_b of EMs. The data needed for this method can be obtained by the nonisothermal differential scanning calorimetry (DSC) measurement alone.

Theory and Method Based on Berthelot's Equation

Derivation of the Formula of Critical Temperature of Thermal Explosion (T_b)

For most energetic materials, the enthalpy of thermal decomposition reaction per unit time can be expressed by the equation

$$q_1 = Q\frac{Vd}{M}\frac{d\alpha}{dt} \tag{1}$$

where Q is the enthalpy of the thermal decomposition reaction in $J \cdot mol^{-1}$, V is the volume of explosive loaded in cm^3, d is the loading density in $g \cdot cm^{-3}$, M is the molar mass of explosive loaded in g, and $\frac{d\alpha}{dt}$ is the reaction rate, which may be expressed as

$$\frac{d\alpha}{dt} = kf(\alpha) = A_0 f(\alpha)\exp(bT) \tag{2}$$

where

$$k = A_0 \exp(bT) \tag{3}$$

Formula (3) is known as Berthelot's equation. A_0 and b are two coefficients in formula (3). Their units are s^{-1} and K^{-1}, respectively.

Substituting $\frac{d\alpha}{dt}$ in Eq. (1) with Eq. (2), the expression for q_1 becomes

$$q_1 = Q\frac{Vd}{M}A_0 f(\alpha)\exp(bT) \tag{4}$$

with a linear increase in temperature (Eq. (4)).

$$T = T_0 + \beta t \tag{5}$$

Therefore, it is apparent that q_1 is proportional to the exponent of the reaction temperature T. At the same time, the heat (q_2) lost from the reaction system in unit time may be expressed as

$$q_2 = k'S(T - T_{\mathrm{s}}) \tag{6}$$

where k' is an overall heat transfer coefficient in $\mathrm{J \cdot cm^{-2} \cdot K^{-1} \cdot s^{-1}}$, S is the external surface area of the loaded sample in $\mathrm{cm^2}$, T is the temperature of the reaction system in K, and T_{s} is the surrounding temperature in K, which is determined by the linear temperature increase in DSC analysis.

With the boundary conditions of thermal explosion, Eq. (4) becomes

$$q_1|_{T_{\mathrm{b}}} = Q\frac{Vd}{M}A_0 f(\alpha_{\mathrm{b}})\exp(bT_{\mathrm{b}}) \tag{7}$$

and Eq. (6) becomes

$$q_2|_{T_{\mathrm{b}}} = k'S(T_{\mathrm{b}} - T_{\mathrm{sb}}) \tag{8}$$

where T_{sb} is the surrounding temperature at the beginning of the thermal explosion in K.

According to Semenov's thermal explosion theory [9], the sufficient and essential conditions from thermal decomposition to thermal explosion might be expressed as

$$q_1|_{T_{\mathrm{b}}} = q_2|_{T_{\mathrm{b}}} \tag{9}$$

$$\left.\frac{\mathrm{d}q_1}{\mathrm{d}T}\right|_{T_\mathrm{b}} = \left.\frac{\mathrm{d}q_2}{\mathrm{d}T}\right|_{T_\mathrm{b}} \tag{10}$$

Differentiating Eq. (4) with respect to T, and considering Eq. (5), we can obtain

$$\begin{aligned}\left.\frac{\mathrm{d}q_1}{\mathrm{d}T}\right|_{T_\mathrm{b}} &= \frac{1}{(\mathrm{d}T/\mathrm{d}t)_{T_\mathrm{b}}}\frac{QVd}{M}A_0 f(\alpha_\mathrm{b})\exp(bT_\mathrm{b}) \\ &\quad \times \left[A_0 f'(\alpha)\exp(bT_\mathrm{b}) + b\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}}\right]\end{aligned} \tag{11}$$

Differentiating Eq. (6) with respect to T, and considering Eq. (5), we get

$$\left.\frac{\mathrm{d}q_2}{\mathrm{d}T}\right|_{T_\mathrm{b}} = \frac{1}{(\mathrm{d}T/\mathrm{d}t)_{T_\mathrm{b}}} k'S\left[\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}} - \beta\right] \tag{12}$$

Combining Eqs. (7), (8), and (9), yields Eq. (13).

$$Q\frac{Vd}{M}A_0 f(\alpha_\mathrm{b})\exp(bT_\mathrm{b}) = k'S(T_\mathrm{b} - T_\mathrm{sb}) \tag{13}$$

Combining Eqs. (10), (11) and (12), yields Eq. (14).

$$\begin{aligned}&\frac{QVd}{M}A_0 f(\alpha_\mathrm{b})\exp(bT_\mathrm{b})\left[A_0 f'(\alpha)\exp(bT_\mathrm{b}) + b\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}}\right] \\ &\quad = k'S\left[\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}} - \beta\right]\end{aligned} \tag{14}$$

Combining Eqs. (13) and (14) results in Eq. (15).

$$\left[A_0 f'(\alpha)\exp(bT_\mathrm{b}) + b\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}}\right](T_\mathrm{b} - T_\mathrm{sb}) = \left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}} - \beta \tag{15}$$

For most explosives, the differential form of the mechanism function for the thermal decomposition reaction may be expressed as $f(\alpha) = (1-\alpha)^n$ and when the transition from thermal decomposition to thermal explosion is triggered, the fraction of the material reacted α is very small; i.e., $f(\alpha) \approx 1$ and $f'(\alpha) = 0$. Equation (14) may therefore be expressed as

$$b(T_\mathrm{b} - T_\mathrm{sb}) = \frac{\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}} - \beta}{\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}}} \tag{16}$$

where $(\mathrm{d}T/\mathrm{d}t)_{T_\mathrm{b}}$ is the increasing rate of temperature in the sample when its thermal decomposition converts into thermal explosion. This is difficult to solve directly from conventional experiments.

When the transition from thermal decomposition to thermal explosion begins, the surrounding temperature is near to the onset temperature T_e of the DSC curve. Substituting T_ei of DSC curves with heating rate β_i for T_sb, when β tends to zero, we take the limitation of both sides of Eq. (16)

$$\lim_{\beta \to 0} b(T_\mathrm{b} - T_\mathrm{sb}) = \lim_{\beta \to 0} b(T_\mathrm{b} - T_\mathrm{e}) = b(T_\mathrm{b} - T_\mathrm{e0}) \tag{17}$$

$$\lim_{\beta \to 0} \frac{\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}} - \beta}{\left(\frac{\mathrm{d}T}{\mathrm{d}t}\right)_{T_\mathrm{b}}} = 1 \tag{18}$$

Therefore, Eq. (16) can be simplified into the form

$$b(T_\mathrm{b} - T_\mathrm{e0}) = 1 \tag{19}$$

It may also be expressed as

$$T_\mathrm{b} = T_\mathrm{e0} + \frac{1}{b} \tag{20}$$

Equation (20) is the relation formula for estimating the value of T_b of EMs under linear temperature increase conditions.

Substituting the measured values of T_e0 and b into Eq. (20), the value of T_b is obtained.

Calculation of the Value of T_{e0}

The value T_{e0} of the onset temperature (T_e) corresponding to $\beta \to 0$ may be obtained by using linear regression of T_{ei} and β_i as described in Eq. (21).

$$T_{ei} = a_0 + a_1\beta_i + a_2\beta_i^2 + a_3\beta_i^3 + \cdots + a_{L-2}\beta_i^{L-2} \quad i = 1, 2, \ldots, L \tag{21}$$

where a_0, a_1, $a_2, \ldots,$ a_3, and a_{L-2} are coefficients.

The value of T_{ei} is easily obtained from the DSC curve with the heating rate β, and a unique equation set can be defined using four groups or five groups of T_{ei} and β_i. When β tends to zero, the value of T_{e0} equals the value of a_0, and it is designated T_{e0}.

Calculation of the Value of b

Combining Eqs. (2) and (5), we have

$$\frac{d\alpha}{dT} = \frac{A_0}{\beta} e^{bT} f(\alpha) \tag{22}$$

Rearranging both sides of Eq. (22) and integrating yield

$$\begin{aligned} G(\alpha) &= \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A_0}{\beta}\int_0^T e^{bT} dt = \frac{A_0}{\beta}\frac{1}{b} e^{bT}\Big|_0^T \\ &= \frac{A_0}{b\beta}\left(e^{bT} - 1\right) \overset{e^{bT}\gg 1}{\approx} \frac{A_0}{b\beta} e^{bT} \end{aligned} \tag{23}$$

Taking the logarithm on both sides of Eq. (23), the integral equation (Eq. (24)) may be obtained

$$\ln \beta_i = \ln\left[\frac{A_0}{bG(\alpha)}\right] + bT_i, \quad i = 1, 2, \ldots, L \tag{24}$$

After the data (β_i, T_i, $i = 1, 2, \ldots, L$) are fitted to Eq. (24) by the linear least-squares method on the computer, the value of b may be obtained from the slope.

Table 1

Calculated values of T_{e0}, E_0, b, and T_b for the exothermic decomposition reaction of energetic materials, determined from the DSC curves at various heating rates

		Eq. (21)	Ozawa's method[a]		Eq. (24)		Zhang et al.'s method[b]	This work
β (°C · min^{-1})	T_e (°C)	T_{e0} (°C)	E_O (kJ · min^{-1})	r_o	b	r	$T_{b,ZHXL}$ (°C)	T_b (°C)
Double-base propellant								
1.053	158 [10]	151.35	135.7	0.9921	0.085735	0.9909	163.01	163.01
2.105	161							
5.333	174							
11.25	182							
22.31	192							
Triethylene glycol dinitrate								
10	189.78 [11]	180.98	109.09	0.9887	0.0612307	0.9866	197.89	197.31
20	199.91							
30	204.67							
40	212.99							

(*Continued*)

Table 1
Continued

		Eq. (21)	Ozawa's method[a]		Eq. (24)		Zhang et al.'s method[b]	This work
β (°C · min^{-1})	T_e (°C)	T_{e0} (°C)	E_O (kJ · min^{-1})	r_o	b	r	$T_{b,ZHXL}$ (°C)	T_b (°C)
3,3-Bis(azidomethyl)oxetane/tetrahydrofuran copolymer								
2.0	216.38 [12]	210.25	151.81	0.9996	0.075386	0.9989	223.78	223.52
5.0	227.54							
10.0	236.68							
20.0	246.97							
Lead salt of 2-hydroxy-3,5-dinitropyridine								
2.0	320.25 [13]	314.00	219.96	0.9968	0.075277	0.9979	327.65	327.29
5.0	334.26							
10.0	341.96							
20.0	351.06							
Lead salt of 4-hydroxy-3,5-dinitropyridine								
2.0	315.81 [13]	308.09	187.50	0.9997	0.0645296	0.9999	323.90	323.59
5.0	330.15							
10.0	340.91							
20.0	351.47							

$Pb_{0.25}Ba_{0.75}$(2,4,6-trinitro-1,3-dihydroxy-benzene) · H_2O								
2.5	294.87 [14]	288.474	161.61	0.9872	0.059981	0.9856	305.71	305.15
5.0	306.15							
10.0	313.87							
15.0	325.66							
3,4-Dinitrofurazanfuroxan (DNTF)								
2	230.63 [15]	226.0	148.05	0.9836	0.0690083	0.9798	240.82	240.48
10	248.84							
15	255.95							
20	265.07							
Highly nitrated nitrocellulose containing 14.14% of nitrogen [HNNC(14.14% N)]								
1.059	178.05 [16]	173.15	170.45	0.9991	0.0991003	0.9981	183.31	183.24
2.211	184.75							
5.202	192.55							
10.78	200.25							
21.39	208.55							
Nitrocellulose containing 12.97% of nitrogen [NC(12.97% N)]								
1.031	178.80 [17]	174.62	180.97	0.9974	0.105046	0.9961	184.23	184.14
2.146	185.75							
5.131	192.30							
10.59	199.50							
21.59	208.30							

(Continued)

Table 1

Continued

		Eq. (21)	Ozawa's method[a]		Eq. (24)		Zhang et al.'s method[b]	This work
β (°C · min^{-1})	T_e (°C)	T_{e0} (°C)	E_O (kJ · min^{-1})	r_o	b	r	$T_{b,ZHXL}$ (°C)	T_b (°C)
Pentaerythritol diazido dinitrate (PDADN)								
1	150.10 [18]	145.58	133.45	0.9932	0.0871664	0.9945	157.11	157.05
2	161.05							
5	168.26							
10	178.50							
20	184.62							
1-(2,4-Dinitrophenyl)azo-1-nitrocyclohexane								
2.5	156.05 [19]	147.72	116.77	0.9996	0.076005	0.9991	161.15	160.88
5.0	164.17							
10.0	173.81							
15.0	179.48							
Copper(II) salt of 4-hydroxy-3,5-dinitropyridine								
2.0	333.06 [20]	320.06	181.87	0.9592	0.0591899	0.9591	337.09	336.96
5.0	347.88							

10.0	365.91							
20.0	366.12							
Nitrocellulose containing 13.86% of nitrogen [NC(13.86% N)]								
1.025	177.40 [21]	172.89	163.38	0.9966	0.095133	0.9952	183.50	183.40
2.065	184.70							
5.183	192.25							
10.82	200.60							
18.02	208.25							
Lead 2,4,6-trinitroresorcinate monohydrate [Pb(TNR) $\cdot$ H_2O]								
2.5	258.20 [22]	243.53	123.07	0.9893	0.0520163	0.9916	262.94	262.75
5.0	275.38							
10.0	286.78							
15.0	292.22							
Nitrocellulose containing 13.54% of nitrogen [NC(13.54% N)]								
1.015	178.80 [23]	174.66	178.75	0.9905	0.103787	0.9885	184.29	184.29
2.044	185.75							
5.182	192.30							
11.68	199.50							
18.02	207.80							

(Continued)

Table 1
Continued

		Eq. (21)	Ozawa's method[a]		Eq. (24)		Zhang et al.'s method[b]	This work
β (°C · min^{-1})	T_e (°C)	T_{e0} (°C)	E_O (kJ · min^{-1})	r_o	b	r	$T_{b,ZHXL}$ (°C)	T_b (°C)
Barium 2,4,6-trinitroresorecinate monohydrate [Ba(TNR) · H_2O]								
2.0	299.80 [24]	292.38	166.13	0.9991	0.0600105	0.9992	309.36	309.05
5.0	315.69							
10.0	325.72							
20.0	338.59							
Nitrocellulose containing 11.92% of nitrogen [NC(11.92% N)]								
1.047	177.30 [8]	173.27	165.69	0.9975	0.0965712	0.9965	183.75	183.63
2.075	184.50							
5.378	192.05							
10.53	200.50							
19.70	208.05							
Hydroxylammonium nitrate (HAN)[c]								
1.007	106.45 [25]	98.72	78.95	0.9990	0.0631292	0.9993	114.55	114.57
2.056	116.65							

5.100	132.45							
10.95	143.65							
Triethanolammonium nitrate (TEAN)[c]								
1.989	116.65 [25]	106.74	55.35	0.9917	0.040284	0.9959	131.31	131.56
5.174	145.45							
11.10	160.25							
22.07	176.85							
HAN-based liquid propellant, LP								
1.919	130.85 [25]	120.40	78.36	0.9821	0.0551728	0.9804	138.37	138.52
5.185	140.85							
10.84	161.65							
21.30	170.65							
TB propellant M32 (45/23/30/5-NC/NG/DMATZ/additive)								
0.5260	157.85 [25]	151.12	161.94	0.9919	0.101782	0.9899	160.78	160.94
1.100	161.85							
2.000	169.85							
5.332	177.85							
10.63	182.85							
19.86	193.85							

(*Continued*)

Table 1
Continued

		Eq. (21)	Ozawa's method[a]		Eq. (24)		Zhang et al.'s method[b]	This work
β (°C · min^{-1})	T_e (°C)	T_{e0} (°C)	E_O (kJ · min^{-1})	r_o	b	r	$T_{b,ZHXL}$ (°C)	T_b (°C)
TB propellant SD (32/23/40/5-NC/NG/NGU/additive)								
1.111	164.85 [25]	158.54	186.64	0.9951	0.116692	0.9951	167.17	167.10
2.000	167.85							
5.455	178.85							
11.00	182.85							
22.31	189.85							
CL-20								
2	218.40 [25]	212.65	166.43	0.9990	0.082402	0.9981	225.05	224.79
5	228.00							
10	236.90							
20	246.20							

PBX-JH-94 (94/3/2/1-RDX/TNETB/PVAC/SA)								
2.12	183.85 [25]	175.96	112.53	0.9995	0.0631124	0.9998	191.94	191.80
5.26	197.85							
10.74	209.85							
22.22	220.85							
PBX-JO-96 (96.5/2/1.5-HMX/binder/plasticizer)								
2.11	270.85 [25]	267.87	404.87	0.9804	0.168827	0.9796	274.01	273.79
5.62	273.85							
10.00	278.85							
21.88	283.85							

[a] E, apparent activation energy; r, linear correlation coefficient; subscript O: data obtained by Ozawa's method.

[b] Subscript ZHXL: data obtained by Zhang et al.'s method.

[c] The DSC data for HAN and TEAN were obtained with a stainless steel sealed cell (diameter $5\,\text{mm} \times 2.85\,\text{mm}$). The other DSC data were obtained with an aluminum cell (diameter $5\,\text{mm} \times 3\,\text{mm}$) whose side was rolled up.

Comparison of the Calculated Values T_b in this Work with Literature Ones $T_{b,ZHXL}$

To verify the reliability of Eq. (20), the literature values of β_i and T_i ($i = 1, 2, \ldots, 5$), the calculated values of E_O by Ozawa's method, the obtained value of T_{e0} when β tends to zero, the value of b obtained by Eq. (24), together with the reasonable values of $T_{b,ZHXL}$ obtained by Zhang et al.'s method [4] and the values of T_b obtained by substituting the above-mentioned values of T_{e0} and b into Eq. (20), are shown in Table 1.

It can be seen that the calculated values of T_b obtained by the two different formulae agree well to each other, clearly demonstrating that Eq. (20) and Zhang et al.'s formula are suitable for estimating the values of T_b for EMs. The results obtained by the two methods are all the same.

Conclusion

The T_b results of 25 energetic materials estimated using this method agree with the data available in the literature. Therefore, it can be concluded that this method may be used for estimating the critical temperature of thermal explosion for energetic materials.

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