

ON THE DETERMINATION OF KINETIC PARAMETERS THROUGH THE PEAK TEMPERATURE METHOD FOR DIFFERENTIAL SCANNING CALORIMETRY

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(Received August 12, 1983; in revised form May 8, 1984)

A critical assessment of the so-called "peak temperature methods" (originally proposed by Kissinger) is presented. The two ingredients of peak temperature methods, namely, Kissinger's assumption and transformation equations, are considered. First it is argued that Kissinger's assumption although not being valid for DTA holds for DSC. Then it is shown that the only way to use kinetic parameters obtained from non-isothermal experimental data to describe both iso- and anisothermal kinetics is to take the same reaction rate equation for the two kinetics, as previously done by Henderson.

Non-isothermal calorimetric techniques (DTA and DSC) are rapidly becoming a valuable tool for obtaining kinetic data in a large variety of phase transformations [1–5]. An important shortcoming of non-isothermal techniques is that it is rather usual to have several reactions occurring in a given temperature range. When this happens it hinders the obtaining of kinetic data from a single scan. Kissinger [1] was first to suggest a method suitable for these cases. He showed that, assuming the maximum reaction rate coincides with the peak of the DTA curve, it was straightforward to obtain kinetic data from several scans performed at different heating rates (multiple scan techniques [2]). Methods based upon Kissinger's assumption are commonly called "peak temperature methods", and, although this assumption is not well founded for DTA [4], they have been widely used for obtaining kinetic data [2–4].

The purpose of this paper is to present a critical assessment of "peak temperature methods". The reason for the non-validity of Kissinger's assumption for DTA will be first reviewed [4], and it will be shown that instead it holds for DSC [6]. Then the question of transformation equations will be addressed. Concerning this point it should be noted that although the literature on thermal analysis shows a general consensus on how to analyze experimental data obtained from isothermal techniques [7, 8], several and quite different methods have been suggested for the study of aniso-

thermal kinetics [2–5, 7, 9, 10]. The fundamental difference between these methods does not lie in the various analytical expressions assumed for the fraction reacted, but rather it concerns what iso- and anisothermal kinetics have in common. Namely, whereas some methods assume that the same reaction rate equation holds for the two kinetics (method I) [2, 5], others take the same fraction reacted (method II) [3] and more recently it has even been assumed that neither the fraction reacted nor the reaction rate follows the same equation for iso- and anisothermal kinetics [9]. The question is whether these methods lead or not to similar results, and, in particular, whether a particular method obeys or not what we shall hereafter call the transferability principle, that is, kinetic parameters obtained from anisothermal experimental data should also be valid to describe isothermal kinetics (the opposite should also hold). The discussion will be illustrated by analyzing DSC curves for recrystallization processes in cold rolled AA1145 and AA8011 aluminium alloys [11], by means of methods I and II; in both cases the Johnson–Mehl–Avrami equations will be used [7]. The kinetic parameters obtained by means of the two methods will be used to describe both iso- and anisothermal kinetics. It will be shown that whereas method I describes correctly both iso- and anisothermal kinetics, method II fails in the isothermal case. It will be argued that the reason for this failure is that method II uses different rate equations for iso- and anisothermal kinetics. This provides a general conclusion applicable to all methods used for the analysis of anisothermal data (peak temperature methods included), namely, in order to fulfil the transferability principle *the same rate equation has to be used for iso- and anisothermal kinetics*.

Critical assessment of peak temperature methods

This section will be devoted to criticize and compare the most commonly used peak temperature methods. First the common base for all peak temperature methods will be discussed: Kissinger's assumption [1, 2]. As regards transformation equations, the use of two methods of types I and II (mentioned in the previous section) will be revised; in both cases the Johnson–Mehl–Avrami equations [7] will be used.

Kissinger's assumption

Without any theoretical basis and mostly based upon some experimental evidence Kissinger assumed some years ago that the peak of the DTA curve coincided with the maximum reaction rate [1]. This assumption has been strongly criticized [4] and argued that even in cases where it could be approximately valid it might lead to large errors in the kinetic parameters.

Kissinger's assumption does not hold for DTA (DSC will be considered later) if one assumes Borchardt and Daniels' analysis of the DTA techniques [4] to be valid.

$$-\frac{d\alpha}{dt} = \frac{1}{KA} \left(hC_p \frac{d\Delta T}{dT} + K\Delta T \right) \quad (1)$$

This Eq. holds for constant heating rate h , namely $T = T_0 + ht$, T_0 is the starting temperature. In Eq. (1) α is the fraction of the transformation completed at time t , C_p the heat capacity of the cell (assumed to be independent of t), K the heat transfer coefficient of the cell and A the total area under the DTA curve. ΔT is actually the data obtained in a DTA experiment, the outcome of the experiment being a graph of ΔT versus time (or temperature).

At the peak (p) of the DTA curve the following relations hold:

$$\left. \frac{d\Delta T}{dT} \right|_{T=T_p} = 0 \quad \text{and} \quad - \left. \frac{d\alpha}{dt} = \frac{1}{A} \Delta T \right|_{T=T_p} \quad (2)$$

then at T_p , the second derivative of α with respect to t can be written as

$$\left. \frac{d^2\alpha}{dt^2} \right|_{T=T_p} = \frac{h^2 C_p}{KA} \left. \frac{d^2\Delta T}{dT^2} \right|_{T=T_p} \neq 0 \quad (3)$$

Therefore Kissinger's assumption is invalidated. This result is a consequence of the reaction rate not being directly proportional to ΔT but rather being given by Eq. (1). It should be here remarked that although Kissinger's assumption is strictly invalid, it has been argued that, in some cases, it approximately holds, and the resulting kinetic data are very similar to those obtained by means of DTA single scan techniques [3]. This might actually be the case whenever the DTA curve is rather flat around T_p , more specifically, when

$$\frac{d\Delta T}{dT} \ll \frac{K}{hC_p} \Delta T \quad \text{around } T_p.$$

Note that this condition also depends on the characteristics of the DTA cell through C_p and K .

Once proved that Kissinger's assumption is strictly invalid for DTA, we turn to show that it holds for DSC [6]. Differential scanning calorimetry directly gives the heat evolved during the reaction, instead of ΔT . This fact allows to obtain $\alpha(t)$ directly from the DSC curve [12, 13], namely

$$\alpha(t) = \frac{A(t)}{A} \quad (4)$$

where $A(t)$ is the area under the DSC curve from the time (or temperature) at which the reaction is started (t_0) up to time (or temperature) t , namely

$$A(t) = \int_{t_0}^t dt \Delta C_p(t) \quad (5)$$

$\Delta C_p(t)$, the output of the DSC experiment, is the change in specific heat along the linear heating. Now if one differentiates Eq. (4) with respect to t , the following expression is obtained

$$\frac{d\alpha(t)}{dt} = \frac{1}{A} \Delta C_p(t) \quad (6)$$

Note that now, in contrast with Eq. (2), the reaction rate is directly proportional to the outcome of the experiment.

At the peak of the DSC curve it is evident that

$$\frac{d\Delta C_p(t)}{dt} = 0 \quad \text{and therefore} \quad \frac{d^2\alpha(t)}{dt^2} = 0 \quad (7)$$

This proves Kissinger's assumption for DSC, and therefore kinetic parameters obtained by making that assumption (peak temperature methods) are as trustable as those obtained from any other method. This makes the conclusions achieved in this paper concerning transformation equations (see next subsection) applicable to all methods used to analyze anisothermal experimental data (peak temperature methods being a particular case).

Transformation equations

It has to be remarked that as the two methods here considered have been thoroughly described elsewhere [2, 3, 5] we shall only outline briefly the points pertinent to the present discussion.

Method I assumes that the reaction rate follows the same equation for iso- and anisothermal kinetics, and that it can be factorized into two ordinary functions of temperature and the fraction reacted α , namely

$$\frac{d\alpha}{dt} = f(\alpha)g(T) \quad (8)$$

The rate function $g(T)$ is usually taken equal to the Arrhenius relation, that is,

$$g(T) = \nu \exp\left(-\frac{\Delta E}{RT}\right) \equiv g_0(T) \quad (9)$$

where ν is the frequency factor, ΔE the activation energy, and R the gas constant.

On the other hand, depending upon the actual reaction, the function $f(\alpha)$ takes one form or another, here we shall only discuss the so-called Johnson-Mehl-Avrami equation [2, 7]

$$\frac{d\alpha}{dt} = n(1-\alpha)(-\ln(1-\alpha))^{\frac{n-1}{n}} g_0(T) \quad (10)$$

This is exactly the equation obtained for isothermal kinetics starting from the Avrami Eq. (7) for the fraction reacted that is,

$$\alpha = 1 - \exp(-(g_0(T)t)^n) \quad (11)$$

where n is an exponent accounting for the nucleation rate and for growth morphology.

In obtaining α for non-isothermal kinetics one integrates Eq. (10) considering that, now, $g_0(T)$ is a function of time through T , the final result is [2]

$$\alpha = 1 - \exp\left\{-\left(\frac{\nu\Delta E}{Rh} p(x)\right)^n\right\} \quad (12)$$

where

$$x = \frac{\Delta E}{RT}, \quad p(x) = \int_x^{\infty} \frac{e^{-x}}{x^2} dx \quad (13)$$

Summarizing, whereas the reaction rate is described in this method for both iso- and anisothermal kinetics by means of the same Eq. (10) [2], the fraction reacted is not, being given by Eqs (11) and (12) respectively. It is also worth remarking that Meisel and Cote [5] have recently argued that in the anisothermal case the Arrhenius rate function in Eq. (11), should be replaced by an integral from $t = 0$ up to time t , of the isothermal function rate (Eq. (9)). Then Eq. (12) is obtained, and therefore justified from an alternative point of view.

The transferability of kinetic parameters obtained with the method described above has been proved in different cases [2, 5] and it will be also checked in the present paper.

The use of Kissinger's assumption leads to the following expression [2],

$$\ln \frac{h}{T_p^2} = -\frac{\Delta E}{RT_p} + \ln \frac{\nu R}{\Delta E} \quad (14)$$

T_p is the peak temperature. This equation has been obtained by taking

$$\frac{\Delta E}{RT_p} \gg 1 \quad (15)$$

this is usually valid for most solid-state reactions.

Equation (14) allows, through a plot of $\ln h/T_p^2$ versus $1/RT_p$, to obtain ΔE and ν . Values for T_p are obtained from several scans at different heating rates (h). It should be remarked that although the method gives ΔE and ν , it does not allow to calculate n . The exponent n has to be obtained from the full DSC curve or assumed to be equal to that of a similar isolated reaction. For example, recrystallization may in some cases overlap with precipitation reactions. The latter can be eliminated by means of an intermediate annealing [11], allowing the determination of n . It can then be assumed that the same n is valid for the two cases.

Equation (14) was first derived by Kissinger [1, 2] although taking $n = 1$ in Eq. (10), therefore, we shall hereafter refer to Eq. (14) as Kissinger's equation.

Method II has been recently suggested by Augis and Bennett [3]. These authors assumed that the fraction reacted for both iso- and anisothermal kinetics was given by Eq. (11), whereas, on the other hand, the isothermal reaction rate followed Eq. (10) and in the anisothermal case it was obtained by differentiating Eq. (11) with respect to time. The result is [3, 10],

$$\frac{d\alpha}{dt} = n(1-\alpha) [-\ln(1-\alpha)]^{\frac{n-1}{n}} g_1(T) \quad (16)$$

where $g_1(T)$ is given by

$$g_1(T) = \left[1 + \frac{\Delta E(T - T_0)}{RT^2} \right] g_0(T) \quad (17)$$

T_0 is the temperature at which the DSC run is started.

Notice that, as first remarked by Boswell [10] the difference between this method and method I lies in the rate function $g(T)$, in fact Eq. (16) is identical to Eq. (10) but with $g_1(T)$ instead of $g_0(T)$.

Kissinger's assumption leads now to the following equation

$$\ln \left(\frac{h}{T_p - T_0} \right) = -\frac{\Delta E}{RT_p} + \ln \nu \quad (18)$$

This equation has been obtained by assuming that

$$\frac{\Delta E}{R} \frac{T_p - T_0}{T_p^2} \gg 1 \quad (19)$$

this is essentially equivalent to Eq (15) as in most cases T_0 is much smaller than T_p .

Equation (18) can be used in a similar way as Eq. (14) to obtain ΔE and ν .

Comparison between the two methods

The equation from which the kinetic parameters are obtained in the two methods (Eqs (14) and (18)) are completely different. The analysis of the experimental data will show that,

$$\Delta E(K) \cong \Delta E(AB) = \Delta E \quad \text{and} \quad \nu(K) \neq \nu(AB) \quad (20)$$

where K stands for Kissinger, the first to obtain Eq. (14), and AB for Augis and Bennett.

On the other hand it will be also shown that both procedures lead to similar non-isothermal behaviours. This can be understood as follows. First a relation between the parameters of Eq. (20) can be obtained from Eqs (14) and (18), that is,

$$\frac{\nu(K)}{\nu(AB)} = \frac{\Delta E}{R} \frac{T_p - T_0}{T_p^2} \quad (21)$$

Then to obtain similar non-isothermal behaviours, the exponents of Eq. (11) with ΔE and $\nu(AB)$ and that of Eq. (12) with ΔE and $\nu(K)$, should be similar, that is

$$\frac{T - T_0}{h} \nu(AB) \exp \left(-\frac{\Delta E}{RT} \right) \cong \frac{\nu(K) \Delta E}{Rh} \rho(x) \quad (22)$$

and using Eq. (21)

$$(T - T_0) \exp \left(-\frac{\Delta E}{RT} \right) \approx \frac{\Delta E^2}{R} \frac{T_p - T_0}{T_p^2} \rho(x) \quad (23)$$

This approximate equation holds if the reaction occurs in a narrow temperature range, for in this case only exponential functions vary strongly in the temperature range and it can be taken,

$$T - T_0 - T_p - T_0 \text{ and } \beta(x) \approx \left(\frac{RT_p}{\Delta E} \right)^2 \exp \left(-\frac{\Delta E}{RT} \right) \quad (24)$$

Therefore for many solid state reactions, Eq. (23) might be approximately valid and the non-isothermal behaviours predicted by the two methods will be very similar.

Now the controversy raises: if for both isothermal kinetics, Eq. (11) is used with parameters given in Eq. (20), the isothermal behaviours will be noticeably different, whereas, as discussed above, the anisothermal ones are not. As stated earlier in this paper, it will be shown that method II do in fact fail in describing isothermal kinetics. The reason for this failure will be proved to be the use of different rate equations for iso- and anisothermal kinetics. In fact this controversy would be settled had the reaction rates for iso- and anisothermal kinetics been given by the same Eq. (16) and therefore the fraction reacted by Eq. (11) for the anisothermal case and by the same Eq. (11), with $g_1(T)$ instead of $g_0(T)$, for the isothermal case. But this turns out to be a method of type I although with an equation for the reaction rate different from the usual Johnson-Mehl-Avrami equation (Eq. (10)).

Experimental procedures

Material

As mentioned in the introduction the experimental data analyzed in this work are on recrystallization processes in commercial aluminium alloys. Two alloys were used, namely, 1145 and 8011, both continuously cast (compositions in Table 1). The alloys were supplied by the Rolling Division of the "Empresa Nacional del Aluminio, S. A.", the as-cast thickness being 8.0 mm. The two alloys were 60% cold rolled in a laboratory rolling mill, and submitted to an intermediate anneal at 673 K for 2 h, to avoid interference with precipitation reactions [11]. The samples were 85% cold rolled after intermediate annealing.

Method

The DSC measurements were performed using a Perkin-Elmer DSC-2C apparatus controlled through a mini-computer. The runs were carried out at heating rates of 5, 10, 20, 40, 60 and 80 deg/min from 300 K to 800 K, and under dynamic nitrogen atmosphere (1 l/h). High purity aluminium was used as reference. Samples for DSC measurements were discs 5 mm in diameter punched from the rolled sheet. It has to be remarked that as the total energy released in recrystallization processes is very small, the highest sensitivity of the DSC apparatus is required. This increases the noise (Fig. 1), leading to an error in the determination of the peak temperature of ± 2 degree.

Table 1 Chemical compositions (as obtained from atomic absorption) in wt% for the two alloys used in this work

	Fe	Si	Mg	Mn	Zn	Ti	Cu
AA1145	0.31	0.11	0.01	0.05	0.02	0.015	0.015
AA8011	0.655	0.59	0.007	0.03	0.016	0.017	0.012

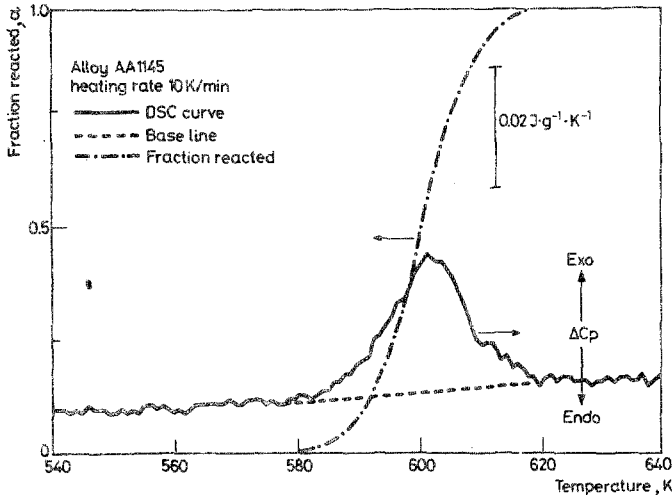


Fig. 1 DSC curve and fraction reacted (as obtained from Eq. (3)) for alloy AA1145 85% cold-rolled after an intermediate anneal at 400 °C for 2 h. The exothermic reaction is mainly associated to recrystallization processes (see ref. [11])

As mentioned above isothermal experiments were also carried out in the same DSC apparatus, at 580 K and 590 K. Two are the main difficulties of isothermal experiments, i) the loss of stored energy during the initial period in which the sample is heated up to the desired temperature, and ii) time needed for sample equilibration. To reduce the effects of these difficulties to a minimum, a medium heating rate (80 deg/min) was used up to 500 K, and then a higher one (320 deg/min) up to the actual temperature. The two chosen temperatures were found to be the most adequate for alloy AA1145. Despite these precautions only data for the time at which the reaction rate is maximum, were considered as reliable.

Results and discussion

As pointed out elsewhere [11] samples subjected to an intermediate anneal at 400° for 2 h, have a DSC curve with a single exothermic reaction (Fig. 1) mainly related to recrystallization process. At higher temperatures an endothermic reaction

Table 2 DSC peak temperatures (K) for the exothermic reaction (recrystallization) in alloys AA1145 and AA8011 (see Fig. 1), as a function of heating rate h (deg/min)

h	5	10	20	40	60	80
AA1145	592	601	612	622	627	632
AA8011	559	565	572	580	583	587

Table 3 Kinetic data (activation energy ΔE (kJ/mol) and frequency factor ν (min^{-1})) for recrystallization reactions in alloys AA1145 and AA8011 as obtained from two peak temperature methods (see text). Peak temperatures given in Table 2

Data	Kissinger		Augis and Bennett		
	ΔE	ν	ΔE	ν	
this work	1145	204.3	$3.60 \cdot 10^{17}$	203.7	$1.73 \cdot 10^{16}$
	8011	259.7	$9.58 \cdot 10^{23}$	258.5	$3.03 \cdot 10^{22}$

appears, associated with the redissolution of phases formed during casting and intermediate annealing [11]. In Table 2 the peak temperatures of the DSC curves for the two alloys and different heating rates are reported. From these data the kinetic parameters (activation energy and frequency factor) were obtained by means of either the Kissinger or Augis and Bennett equations (14) and (18) respectively; the results are given in Table 3.

It should be noticed that the activation energies obtained from the two methods are very similar whereas the frequency factors differ by a factor of 20–30.

Once obtained the activation energy ΔE and frequency factor ν , the fraction reacted α might be evaluated for different values of n by means of either Eq. (12) with data named Kissinger in Table 3 or Eq. (11) (with $g_1(T)$ given by Eq. (17)) and data named Augis and Bennett in Table 3. The results for α can be compared with those obtained from the full DSC curve. Figure 1 shows the DSC curve for alloy 1145 and the fraction reacted $\alpha(t)$ as obtained from that curve. The value for the exponent n which best fitted the DSC results was found to be 2.5 for the two alloys [11]. The DSC results and those obtained from the two peak temperature methods for alloy AA8011 are shown in Fig. 2. Similar results were obtained for alloy AA1145.

It is striking that the two peak temperature methods lead to very similar anisothermal behaviours having very different kinetic parameters. Although the reasons for this result were discussed in the previous section, it will be considered here once more although from a slightly different point of view. As noted above, the two peak temperature methods use equations for the reaction rate which only differ in the rate

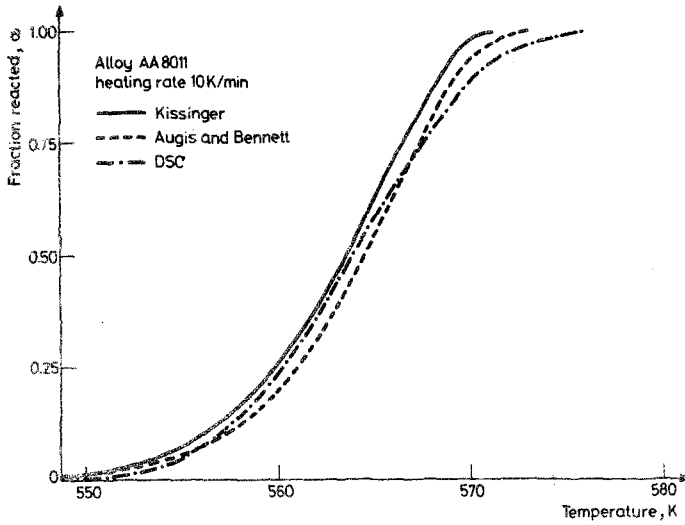


Fig. 2 Fraction reacted as obtained from the DSC curve (Fig. 1) and from the two peak temperature methods discussed in the text, for alloy AA8011. Kinetic data given in Table 3. The exponent n (see text) has been taken equal to 2.5

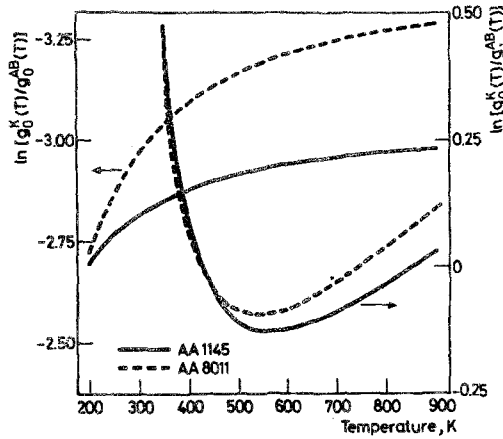


Fig. 3 Plots of Eqs (25) and (26) for recrystallization processes in alloys AA1145 and AA8011. Kinetic parameters given in Table 3

function $g(T)$. Figure 3 shows the logarithm of the ratio between the two rate functions, that is,

$$\ln \frac{g_0^K(T)}{g_1^AB(T)} \tag{25}$$

where the superscripts K and AB stand for Kissinger and Augis and Bennett parameters (Table 3) and the functions g_0 and g_1 are given in Eqs (9) and (17) respectively. It is noted that in the region where the reactions occurred, i.e. 500–800 K the two $g(T)$ functions differ by less than 10%. The behaviour close to 300 K is due to the difference $T - T_0$ in Eq. (17).

These results are in line with the comments of the previous section and give the reasons for the similarity between the non-isothermal behaviours predicted by the two peak temperature methods.

We turn now to compare the isothermal behaviours predicted by the two peak temperature methods. They are completely different as the fraction reacted is given in both cases by Eq. (11). In fact if we plot

$$\ln \frac{g_0^K(T)}{g_0^{AB}(T)} \quad (26)$$

marked differences are found (Fig. 3). To see which, of the two methods gives the correct results for isothermal kinetics, we compare the predicted values for the time at which the reaction rate is maximum with those obtained from the isothermal experiments. The experimental results are 1.9 min and 4.7 min for 590 K and 580 K respectively, whereas the predicted values are 2.7 and 5.4 min for method I, and 5.2 and 107.0 min for method II. This supports furtherly the reliability of the method suggested by Henderson [2], and proves the failure of method II in describing isothermal kinetics.

The isothermal results obtained with method II would have been much closer to the experimental ones and to those of method I had we use $g_1^{AB}(T)$ in Eq. (11) instead of $g_0^{AB}(T)$. But this would have amounted to use the same rate equation for iso- and anisothermal kinetics (Eq. (16)) and different expressions for the fraction reacted (Eq. (11)) with $g_1(T)$ and $g_0(T)$ respectively).

Concluding remarks

The following conclusions arise from the discussion of the previous sections:

- i) Kissinger's assumption, although not being valid for DTA, holds for DSC.
- ii) The only way to consistently apply a given method to the analysis of anisothermal experimental data, is to describe the reaction rate for both iso- and anisothermal kinetics by the same equation (it does not matter whether this equation coincides or not with Eq. (16). This procedure guarantees that, the transferability principle, that is, kinetic parameters obtained from anisothermal experiments should be also valid to describe isothermal kinetics and vice versa, is not violated.

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Zusammenfassung — Die ursprünglich von Kissinger vorgeschlagene sog. "Peak temperatur-Methode" wird einer kritischen Überprüfung unterzogen. Die zwei Bestandteile dieser Methode, nämlich Kissinger's Annahme und die Transformationsgleichungen werden erörtert. Zunächst wird bewiesen, daß Kissinger's Annahme, obzwar sie nicht für die DTA gültig ist, für die DSC zutrifft. Danach wird gezeigt, daß die einzige Möglichkeit, von Daten nicht-isothermer Experimente kinetische Parameter zur Beschreibung sowohl einer isothermen als auch einer nicht-isothermen Kinetik zu erhalten, darin besteht, für beide kinetische Fälle die gleiche Reaktionsgeschwindigkeitsgleichung anzunehmen, wie schon früher Henderson verfahren ist.

Резюме — Представлена критическая оценка так называемых "методов температурных пиков", впервые предложенных Киссинджером. Рассмотрены две составные части методов температурных пиков по Киссинджеру: уравнения допущения и преобразования. Сначала доказано, что уравнение допущения Киссинджера не применимо к методу ДТА, но справедливо для метода ДСК. Затем показано, что только использования кинетических параметров, полученных из неизотермических экспериментальных данных для описания обоих изо- и неизотермических кинетик, учитывает тоже самое уравнение скорости реакции для двух кинетик, как это ранее было сделано Хендерсоном.