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Test Method

Determination of sample size for step-wise transient thermal tests

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ABSTRACT

The step-wise transient method is a dynamic thermal testing method with the advantages of high speed and multi-parameter measurement. However, one area causing a potential error in the measurement is the limitation of finite sample size, leading to test results inconsistent with the theoretical predictions. A detailed analysis of the problem has been conducted in this paper through a numerical method along with experimental verification. The results show that this size effect involves several factors, each playing a different role. The sample size can be optimized based on our analysis so as to improve the measurements.

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1. Introduction

Thermal properties are undoubtedly important physical parameters of materials. In many applications, such as microelectronic, composite materials and aerospace, thermal parameters have become the top critical factors in product design, manufacture and usage. Meanwhile, the thermal parameters are useful indicators for the shifting of other material properties – a new way of thermal parameter application [1–3].

For experimental study, thermal testing methods have been developed gradually from steady-state mode to transient (or dynamic) ones. Many transient approaches have been proposed and studied in recent decades [4–16]. For high speed and multi-parameter testing, transient methods meet the requirements of advancing science and technology and have, hence, been successfully applied to solid material testing. Relatively recent trends have shown that many researchers begin to apply these methods to more complex media, such as composite materials [13,17–21], films [22], liquids [23–26], porous materials [27–31], biomaterials [32], circuit boards [33,34], etc., and results have been highly promising.

The fundamental principle of the transient method is to generate a small thermal disturbance on the tested sample with an otherwise uniform initial temperature, and then record the sample temperature disturbance due to the external thermal excitation. The thermo-physical properties of the sample can be estimated by analyzing the recorded data and using some optimization methods. As there are multiple ways of generating such thermal excitation, several transient thermal testing devices have been developed to date, and these devices can be divided into groups according to the heat source type, such as heat line, hot zone, plane heater and hot plate [5,28,35–39].

This paper is focused on one such method termed the step-wise transient method, which has been investigated more extensively for its several advantages: in theory at least, the step-wise transient method can measure the thermal conductivity and thermal diffusivity of materials simultaneously. Owing to its heating mode, the step-wise transient method also brings much less impact on the sample properties compared to other methods.

The main problem in the step-wise transient method lies in the differences between experiment result and theoretical prediction regarding the sample size. The sample size is assumed infinite in the theoretical model, but this cannot be the case in an actual experiment, leading to the problem of multi-dimensional heat flow in the sample, instead of just the one dimension case dealt with in

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the theory. Since the measurement principle and calculation of results of the experimental device are based on the theoretical model, such difference in sample size is the major source for errors in experimental results.

In order to solve this problem, previous researches attempted to develop more suitable theories to eliminate the influence [40,41]. However, the new theories so far can only be applied to materials with simple geometry and internal structure, otherwise the problem becomes intractable.

Another way to solve this problem is to find an appropriate sample size so that the error caused by the limited sample size can be restricted to an acceptable level. This paper explores the ways to estimate the acceptable sample dimensions.

2. Measurement theory

In Fig. 1, sketch (a) represents a case where the sample size is infinite in all three dimensions, and (b) the actual experiment device. Heat is supposed to be transferred only by thermal conduction. For the sample with infinite size, assume the sample is at a uniform initial temperature T_0 before heating. Once a constant heat flux starts at $x = 0$ and $t > 0$, the temperature distribution at any point in the sample will depend only on the distance between the point and the heat source x and the time t, and can thus be treated as a one-dimensional problem with the solution as:

$$T(x,t) = T_0 + \frac{q}{k}\left\{\left(\frac{at}{\pi}\right)^{1/2}\exp\left(-\frac{x^2}{4at}\right) - \frac{x}{2}\,\mathrm{erfc}\left(\frac{x}{\sqrt{4at}}\right)\right\} \quad (1)$$

$$\lambda \left.\frac{\partial T}{\partial x}\right|_{x=0} = \begin{cases} q & t > 0 \\ 0 & t \le 0 \end{cases} \quad (2)$$

where $T(x, t)$ is the temperature at position x and time t, q is the flux of heat source; k is the thermal conductivity and a is the thermal diffusivity.

The temperature T (x, t) in Eq. (1) is a time-domain function, which can be measured by a thermometer and recorded by a computer. The thermal conductivity k and the thermal diffusivity a of the sample can be found by superimposing Eq. (1) on the recorded temperature-time curve using appropriate fitting techniques.

3. The difference between the experiment and theory

However, there is one factor, sample size, affecting the accuracy of the step-wise transient method. The sample size in establishing Eq. (1) is assumed to be infinite but, obviously, this assumption cannot be realized in experiment. Comparing with the ideal model, the actual sample with limited size has sample edges or boundaries. For easier treatment without losing generality, we assume (YZ plane) the actual square sample of equal sides W with sensor located at the center, and (XZ plane) the thickness L with sensor at distance H from the heat source as shown in Fig. 2. Because of the planer symmetry, we define all the edges in the YZ plane as the *Boundary I* type and in the thickness direction the *Boundary II* type.

The influences of the two types of boundaries can be analyzed as follows:

(A) On *Boundary I* (because of symmetry, we only analyze one side), the heat flows out of this side into the environment so the heat leaking coefficient $\beta_I > 0$, and $\beta_I = 0$ if an infinite sample size were used so no heat loss occurs.

(B) On *Boundary II*, the heat flows diffusely through this side, i.e., $\beta_{II} > 0$. The heat flux Q at this section of $x = L$ would be given by Eq. (3) in the infinite sample case, but will be influenced by β_{II} as the sample size is now finite.

$$Q = -k\left.\frac{\partial T}{\partial x}\right|_{x=L} = -\frac{q}{2}\,\mathrm{erfc}\left(\frac{L}{\sqrt{4at}}\right) \quad (3)$$

In other words, the temperature distribution in a sample with limited size cannot be described by Eq. (1). However, it is easily conceivable that when heating begins it will take some time t_m before the heat can reach the boundaries for the influence due to the limited sample size to take effect. That is to say, if we take the measurement within the period $0 < t < t_m$, the difference between samples of

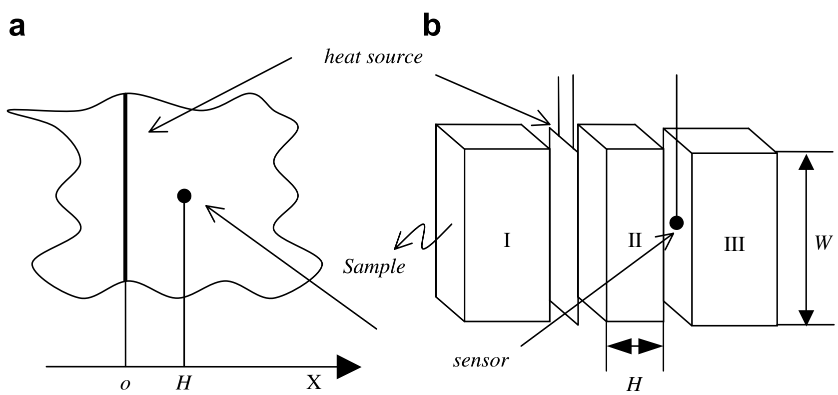

Fig. 1. The step-wise transient method: (a) infinite sample size; (b) actual experiment.

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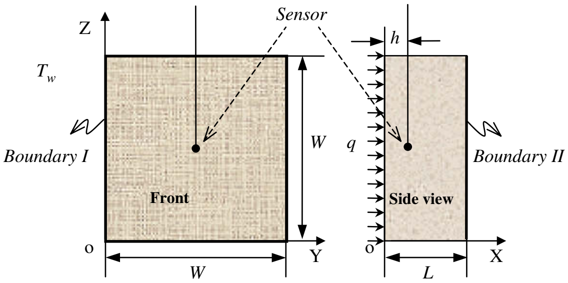

Fig. 2. The sample in experiment.

infinite and finite sizes becomes negligible and Eq. (1) will be valid to use in estimating the sample thermal properties k and a. Furthermore, this maximum allowable measuring time t_m is longer for a larger sample size, at given sample type, test method and conditions. Therefore, one solution to obtaining acceptable k and a by this step-wise transient method is to determine a minimum sample size whose corresponding t_m is such so we can complete the test within it to eliminate the boundary influence on the test results.

4. The numerical error between experiments and the theory

Since this method is based on estimation, we need to define an acceptable error between the experimental and theoretical results, Δ_{fa}, hereby to derive t_m.

4.1. Determination of Δ_f

In general, the error Δ_f between the experiment and theory is a function of many variables related to sample type, size and time and location as in Eq. (4)

$$\Delta_f = (T_E - T_T)/T_T = f(L, W, H, k, a, q, \beta_I, \beta_{II}, t \ldots) \quad (4)$$

where T_E is the actual temperature in the experiment, and T_T is the corresponding temperature in theory, i.e., Eq. (1), at given (x, t). We know the general trend that as $W, L, H \to +\infty$, $\Delta_f \to 0$. Among the factors that affect Δ_f, sample dimensions W, L, H can be treated as the known parameters, k, a the properties to be experimentally determined, and β_I, β_{II} are unknown parameters. Therefore, $|\Delta_{fm}| < \Delta_{fa}$ is a logical criterion to be used for the sample size determination, where $|\Delta_{fm}|$ is the maximum value of $|\Delta_f|$, and Δ_{fa} is the allowable testing error. However, without knowing its relationship, accurate computation of Δ_f as a function of time t is not easy, even if possible.

We can start by first examining the influences of each unknown parameter involved with Δ_{fm} so as to determine the importance of the individual factors.

4.2. The values of heat leaking coefficients β_I and β_{II}

The two heat leaking coefficients β_I and β_{II} will impact Δ_{fm} directly. We will explore the maximum possible values for the two coefficients based on which to calculate the max $|\Delta_{fm}|$. Since both values of β_I and β_{II} can be reduced by increasing the sample size, we can find out the sample size range to satisfy the criterion $|\Delta_{fm}| < \Delta_{fa}$.

As discussed before, $\beta_I = 0$ in the original theory Eq. (1) but $\beta_I > 0$ in practice. Thus a smaller β_I represents less heat loss at *Boundary I*, thus leading to a smaller $|\Delta_{fm}|$. So we can choose its upper limit as the base. According to Refs. [41,42], β_I ranges from 1 wm^{-2} °C^{-1} to 10 wm^{-2} °C^{-1} under free convection conditions.

The effect of β_{II} to Δ_{fm} does not, however, maintain such monotonicity. Nonetheless, when $\beta_{II} = 0$, there is no heat flow through *Boundary II* and the heat will be completely absorbed by the sample; while $\beta_{II} \to +\infty$ indicates that the temperature at *Boundary II* equals the ambient temperature T_w, so that the heat flux through *Boundary II* reaches the maximum. Obviously, under other boundary conditions, $0 < \beta_{II} < +\infty$, and the actual heat flux at *Boundary II* is within these two extreme cases.

4.3. Calculation of Δ_{fm}

In calculating Δ_{fm}, T_T and T_E must be obtained first. T_T is the temperature response in the ideal model and it can be calculated from Eq. (1). T_E is the temperature response in the actual sample, and will be estimated here by numerical approaches. As the heat transfer in the actual sample is in 3D mode, the temperature field will be governed by Eq. (5),

Table 1
Models in computation.

Model	M–ID (T_T)[a]	M–L1 (T_{E1})	M–L2 (T_{E2})	M–W (T_{E3})
Boundary conditions	$\frac{\partial T}{\partial x}\vert_{x=0} = q$	$\frac{\partial T}{\partial x}\vert_{x=0} = q$ $\frac{\partial T}{\partial x}\vert_{x=L} = 0$ $\frac{\partial T}{\partial y}\vert_{y=0,\,W} = 0$ $\frac{\partial T}{\partial z}\vert_{z=0,\,W} = 0$	$\frac{\partial T}{\partial x}\vert_{x=0} = q$ $T(x,\tau)\vert_{x=L} = T_w$ $\frac{\partial T}{\partial y}\vert_{y=0,\,W} = 0$ $\frac{\partial T}{\partial z}\vert_{z=0,\,W} = 0$	$\frac{\partial T}{\partial x}\vert_{x=0} = q$ $T(x,\tau)\vert_{x=L} = T_w$ $k\frac{\partial T}{\partial y}\vert_{y=0,\,W} = \beta_I(T - T_w)$ $k\frac{\partial T}{\partial z}\vert_{z=0,\,W} = \beta_I(T - T_w)$
Initial conditions	$T(x, 0) = T_w$[b]	$T(x, y, z, 0) = T_w$	$T(x, y, z, 0) = T_w$	$T(x, y, z, 0) = T_w$

We use the finite element software ANSYS to analyze Δ_{fm} by setting the element type as *SOLID90*, the gird type as hexahedron, and the internal for every sub-step as 0.01 s.
[a] The ideal model only has one boundary on the surface $x = 0$.
[b] For simplicity, $T_w = T_0 = 0$.

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Table 2
Parameters range in computation.

Parameter	k (wm^{-1} k^{-1})	a (m^2 s^{-1})	q (wm^{-2})	β_I (wm^{-2} °C^{-1})	L (cm)	H (cm)	$W/2$ (cm)
Range	0.03 ~ 0.3	$5 * 10^{-7} \sim 5 * 10^{-6}$	30 ~ 90	1 ~ 15	4 ~ 6	0.5 ~ 1.5	4 ~ 12
Default value	0.15	$2.5 * 10^{-6}$	60	10	5	1	5

which can be solved under certain boundary conditions using the finite difference method (FDM) or the finite element method (FEM). Then, Δ_{fm} can be calculated once the temperature field is solved.

$$\frac{\partial T}{\partial t} = \frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \tag{5}$$

Table 1 listed some possible boundary conditions in solving Eq. (5). M–$L1$ and M–$L2$ correspond to the cases where $\beta_I = 0$, so there is no heat leaking at *Boundary I*, similar to the ideal model Eq. (1), also termed as Model *M–ID* in the table. We can first focus only on the influence of β_{II} to determine the sample length L. Conversely, if we assume L to be adequately large, we only have to consider the influence on W due to β_I. In other words, we can resolve the problem into two cases to first eliminate the coupling effects between the non-zero coefficients β_I and β_{II}. Then, by selecting a sufficiently large value for one sample size L or W, we will be able to optimize the other size so as to make the problem tractable.

Furthermore, according to the thermal physical properties of normal insulation materials, we selected in our computation the ranges of the related parameters as in Table 2. When dealing with the influence of one factor to Δ_{fm}, others will always be given the *default values*.

5. Results and discussion

5.1. Analysis of Δ_{fm} using models M–L1 and M–L2

According to Ref. [43], the analytic solution for $T_E = T_{E1}$ using model M–$L1$ can be expressed as Eq. (6)

$$T(x,\tau) = T_0 + \frac{qL}{2k}\left[\frac{a\tau}{L^2} - \frac{L^2 - 3x^2}{6L} + L\sum_{n=1}^{\infty}(-1)^{n+1}\frac{2}{n^2\pi^2}\cos\frac{n\pi x}{L}e^{-(n^2\pi^2 a\tau/L^2)}\right] = T_{E1} \tag{6}$$

From Eqs. (1) and (6), we obtain

$$\Delta^1_{fm} = \frac{T_{E1} - T_T}{T_T} \tag{7}$$

By bringing the condition $T_o = T_w = 0$, Δ^1_{fm} value in this case turns out to have nothing to do with q and k. The conclusion still holds when using the numerical solutions for model M–

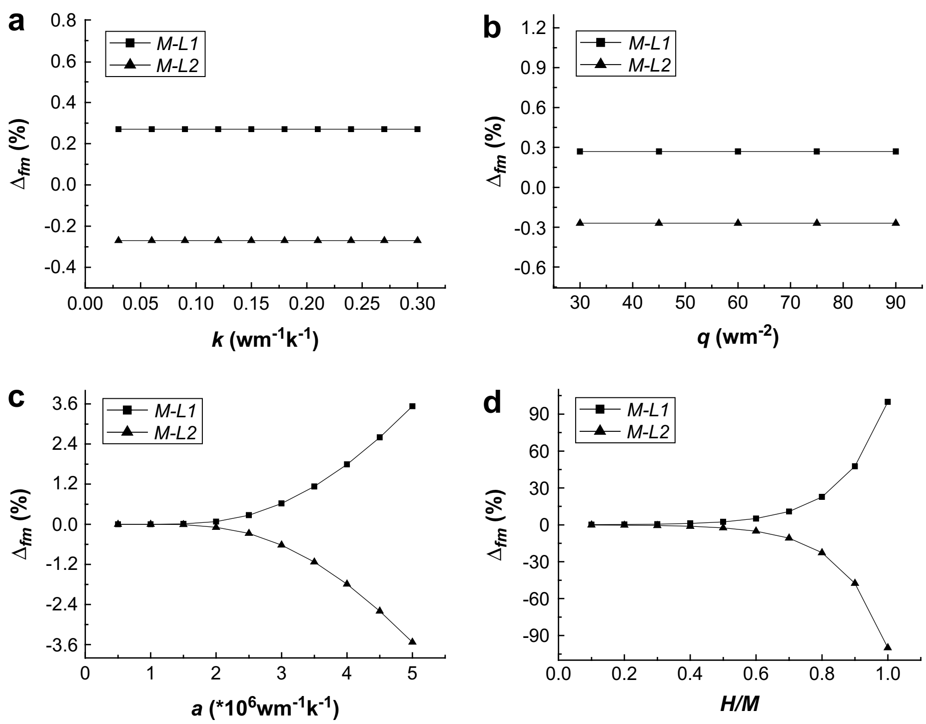

Fig. 3. Factor analysis based on models M–$L1$ and M–$L2$.

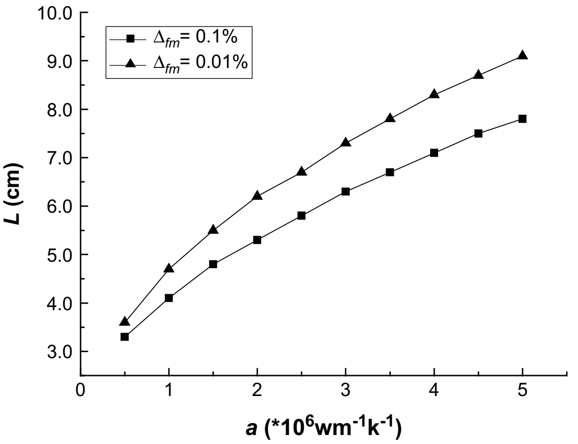

Fig. 4. The thickness L vs. the thermal diffusivity.

$L2$ as shown in Figs. 3a,b when $T_E = T_{E2}$. That is, both models M–$L1$ and M–$L2$ are equivalent in our effort to calculate Δ^1_{fm}.

The position of temperature measuring point H will also affect the deviation, Δ_{fm}, and this effect will intensify as the ratio of H/L increases. In order to eradicate the effect caused by sample thickness, it is required in Ref. [35] that the sample dimension ratio $H/L < 1/2.4 \approx 0.417$; however this requirement does not take into account the role of the thermal diffusivity a. We can see from Fig. 3c that the thermal diffusivity a will also affect Δ_{fm}. In order to optimize L, we need to fix other related parameters which can be determined independently. For instance, the value for H is constrained by the tester itself, so we can fix H first and obtain a suitable L at given allowable time t_m, i.e., the acceptable error Δ_{fa}. If we decide $H = 1.5$ cm, $t_m = 200$ s, $\Delta_{fa} = 0.1\%$ and 0.01%, respectively, then the thickness L vs. the thermal diffusivity a can be calculated in Fig. 4.

From Fig. 4, we can conclude that L will become larger for the sample with higher thermal diffusivity for a given value Δ_{fa}. Also, by estimating from Fig. 4, we can see that for a given $a = 5 * 10^{-7}$ m^2 s^{-1}, the $|\Delta_{fm}|$ can be decreased by nearly one order of magnitude via increasing L by only 16.67%. In other words, the error caused due to finite sample size W can be compensated by adding suitable sample thickness L.

5.2. *The thickness selection in M–W model*

In this article, the sample thickness can be determined from Fig. 4 as $L = 9$ cm, which makes the deviation caused by finite thickness less than 0.01% at $t_m = 200$ s. What follows is a discussion on the deviation caused by the heat loss when using the M–W model.

5.3. *Factor analysis of Δ_{fm} using model M–W*

After fixing the thickness and removing the effect of deviation caused by the finite thickness, the solution of Model M–W in Table 1 becomes very straightforward, from which parametric studies of single factors can be obtained as shown in Fig. 5. When other parameters are

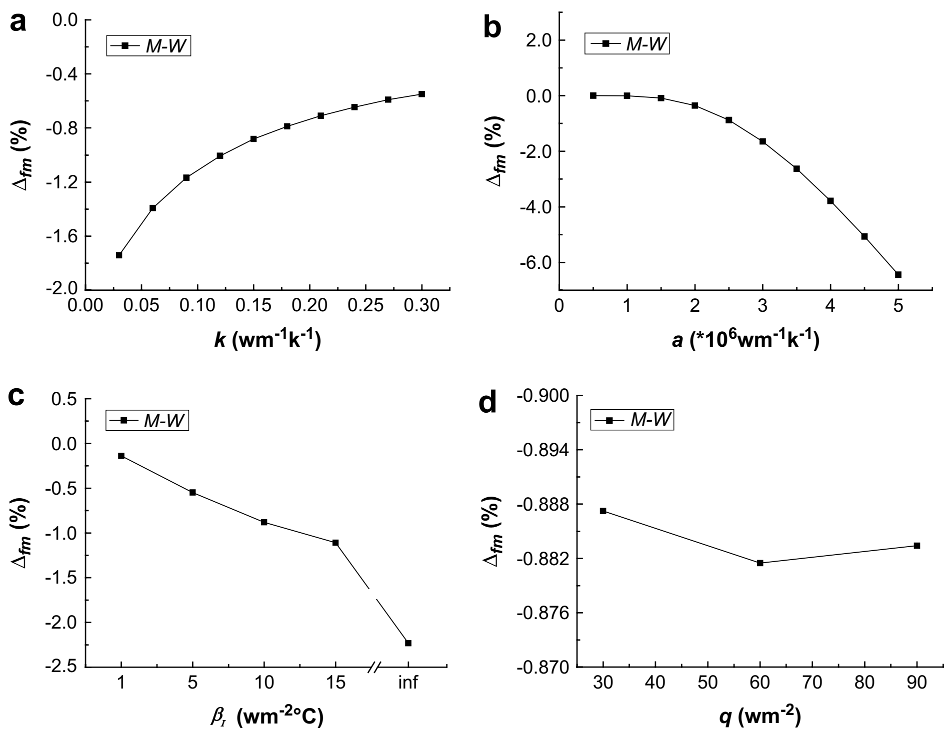

Fig. 5. Single factor analysis for M–W Model.

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fixed, $|\Delta_{fm}|$ will get smaller by increasing the thermal conductivity k (note the negative sign of Δ_{fm} in the figures). On the contrary, the thermal diffusivity a has an opposite trend.

The direct cause of heat loss is from thermal transfer coefficient $\beta_I > 0$. This influence will grow with β_I. But the influence of thermal transfer coefficient β_I to deviation is finite, as shown in Fig. 5c, when $\beta_I \to +\infty$, Δ_{fm} approaches a constant calculated from Table 2 as $|\Delta_{fm}| < 2.5\%$. This suggests that the accuracy of step-wise transient method will not be as highly sensitive to the sample size once the size exceeds certain limits.

The influence of heat flux q on Δ_{fm} as seen in Fig. 5d is not significant and can hence be neglected in further analysis.

5.4. The compound effect of β_I and k

So far, we see the deviation caused by the heat loss is related to the thermal coefficient k, thermal diffusivity a, heat converting β_I and sample length W, for the thickness of the sample L has been determined. Next, we need to find out the relationship between W and L so as to estimate W using given L, once we can get a sense of the influence of β_I and k on Δ_{fm} in this Model M–W by plotting Δ_{fm} against k at two different levels of β_I/k in Fig. 6.

From Fig. 6, we can see there's no connection between k and Δ_{fm}, provided that β_I/k remains a constant. A larger value of β_I/k, indicating either a bigger β_I or a small k, will yield a larger Δ_{fm}. For most materials, $k > 0.024$ (the thermal coefficient for silica aerogel under 32 °C). So, based on the range for β_I from [41,42], $\beta_I/k < 1000$ for common materials.

5.5. The combined action of L and W to Δ_{fm}

From Fig. 7, we can see when $W/2L = 1.2$, $\Delta_{fm} \approx 0.9900$ and $W/2L = 1.7$, $\Delta_{fm} \approx 0.9998$, i.e., change of $W/2L$ by nearly 50% causes only less than 0.01% to Δ_{fm}. Furthermore, $a = 2.5 * 10^{-6}$ and $5.0 * 10^{-6}$ will make little difference

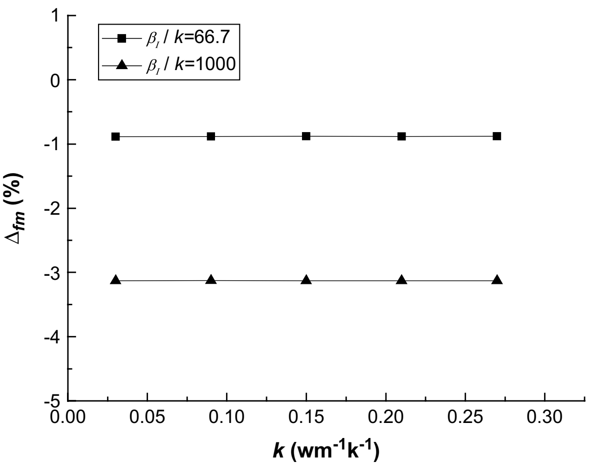

Fig. 6. The influence of k on Δ_{fm} with constant ratio of β_I/k.

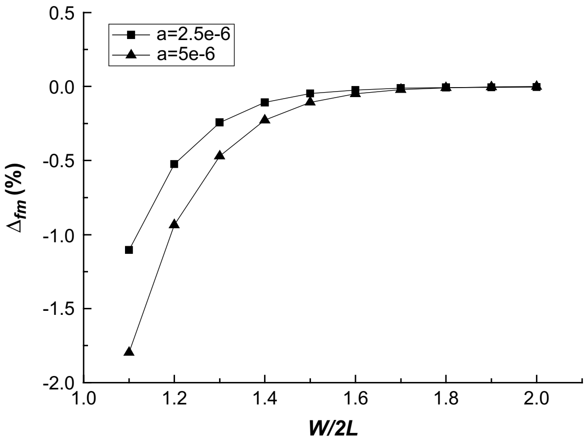

Fig. 7. The influence of ratio W/L to Δ_{fm} ($\beta_I/k = 1000$).

once $W/2L \geq 1.7$. We choose W ranging between $2.4L \sim 3L$, that is, 21.6 cm ∼ 27 cm.

6. The comparison between the test results and theoretical predictions

In order to examine the accuracy of the above analysis, we used YG6020 silicone grease manufactured by GE-TOSHIBA as our testing material, with thermal conductivity when solidified $k = 0.84\ \mathrm{wm^{-1}\ k^{-1}}$. A box is filled with silicone grease. In Table 3, the values of L and W are calculated based on the analysis in the present work under special restriction conditions chosen according to the silicone grease's thermal properties and our test environment. So, the box size was selected as $6.0 \times 6.4 \times 6.4\ \mathrm{cm^3}$.

A heat source was inserted in parallel into the box at a distance of $H = 3.0$ cm from the bottom, which makes it divide the sample thickness into two equal and symmetrical parts. The thickness of the heat source is 0.02 mm, so its influence can be ignored.

The point for measuring temperature was located in the axes of the box, with a distance of 5.6 mm from the heat source. The flux of the heat source in the test was 1610.8 W/m^2. Total heating time period was 490 s.

There are several parameter fitting methods for a temperature-time curve and we used the one mentioned in Ref. [41]. Generally, this fitting method divides the total heating time period into small intervals, and within each interval the fitting procedure was applied based on the theoretical response from Eq. (1). Each interval was 2 s.

Table 3
Calculation results and restriction conditions for the silicone grease.

Calculation results			
L		=6.0 cm	
$W/2$		=3.2 cm	
Restriction conditions			
Δ_{fa}	=0.1%	k	$=0.84\ \mathrm{wm^{-1}\ k^{-1}}$
β_I	$=20\ \mathrm{wm^{-2}\ ^\circ C^{-1}}$	A	$=6 * 10^{-7}\ \mathrm{m^2\ s^{-1}}$

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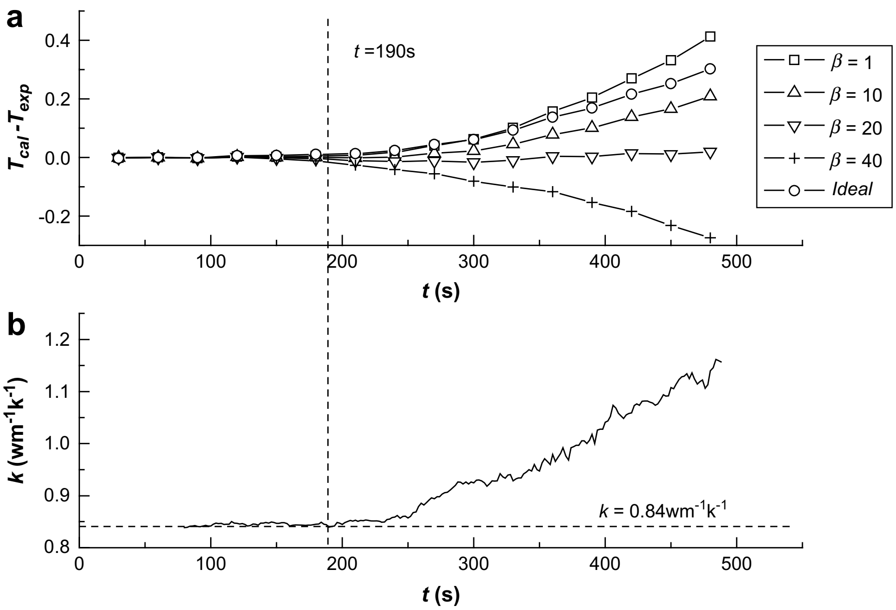

Fig. 8. The comparison between experiment and finite element analysis; (a) the temperature error with time at different values of β; (b) the change of thermal k conductivity with time.

Also, since all the surfaces of the box contact with the ambient air, the two heat leaking coefficients β_{I} and β_{II} have the same value, so we set $\beta_{\mathrm{I}} = \beta_{\mathrm{I}} = \beta$.

Fig. 8a shows the change of the temperature difference $T_{cal} - T_{exp}$ with time under different β values; where T_{cal} is calculated from Eq. (1) using the data in Table 3, and T_{exp} is measured on the solidified silicone grease sample. For the first 190 s, all the curves coincide; but then the deviation between these curves will grow. Fig. 8b illustrates the thermal conductivity calculated from the recorded temperature curve in the experiment. It can be found that the fitting results for those time periods before 190 s agree well with the actual material k value, but the deviation then grows with time, indicating the divergence of the results between T_{cal} and T_{exp} seen in Fig. 8a due mainly to the heat leaking associated with the finite sample size. Nonetheless, the test results confirm that at the chosen L and W, reliable measurement of the sample properties are obtainable as long as it is done before the time limit $t_m < 190$ s.

A check on the influence of β value on the temperature difference depicted in Fig. 8a could be attempted. However, such influence does not exist as long as $t_m < 190$ s. From the figure, it shows that as β increases from 1 wm^{-2} °C^{-1} to 40 wm^{-2} °C^{-1}, the temperature difference $T_{cal} - T_{exp}$ swings from positive maximum to negative maximum. If both T_{cal} and T_{exp} predicted were reliable even beyond $t_m = 190$ s, $\beta = 0$ would lead to $T_{cal} - T_{exp} \to 0$. Actually, in Fig. 8a, $\beta = 0$ (*ideal*) curve still shows some positive temperature difference, i.e., $T_{cal} > T_{exp}$. This reveals that at $t_m > 190$ s, since Eq. (1) is still valid, the experimentally measured T_{exp} deviates from the theory because of the finite sample size, highlighting the importance of the criterion $t_m < 190$ s for conducting the experiment. This deviation however can be off-set by choosing $\beta \approx 20$ wm^{-2} °C^{-1}, for in Fig. 8a, $\beta = 20$ wm^{-2} °C^{-1} leads to $T_{cal} \approx T_{exp}$, thus determining the heat leaking coefficient for the sample.

7. Conclusions

The step-wise transient method offer a simple, fast procedure for thermal properties with less impact on tested material. However, as the sample under test cannot meet the infinite assumptions in the theory adopted, the potential error and the measures to take to assure the reliability of the testing results are discussed in this study.

First, because of the complexity in analyzing actual samples with limited sample size, we employed a couple of numerical approaches to estimate the problem. It is then concluded from our analysis that, using this approach, for given testing condition and sample, we can determine a maximum time t_m. As long as $t < t_m$, the error caused by the finite sample size is negligible. Alternatively, we can also provide a heat leaking coefficient β to account for the heat loss from the boundary of the actual sample so that the original theory can become applicable in the case of limited sample size by including this β into the analysis.

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