Measurement of thermal conductivity of materials using single-side TPS technique

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Abstract: Based on the traditional measurement theory of transient plane source (TPS) technique, single-side TPS method is proposed for measuring the thermal conductivity of single specimen. The problem of transient heat conduction in a semi-infinite boundary condition is studied and the theoretical formula of single-side TPS method is deduced. During the measurement, the influence of the probe heat capacity on the results is analyzed and the corresponding mathematical compensation model is established, and a series of experiments on different materials are conducted by hot disk probe at normal temperature and pressure. The results show that the relative error with the single-side TPS method is less than 5% and the relative standard deviation is no greater than 3%. This method has high accuracy and good reproducibility, which provides a feasible measuring method for single material that does not meet the requirements of the standard TPS theory.

Key words: single-side transient plane source (TPS) method; thermal conductivity; single material; probe heat capacity

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0 Introduction

Thermal conductivity is one of the most important thermal properties of materials, which is closely related to their structure, composition and density. Researching the accurate thermal conductivity has great significance for all walks of life. For example, the study on thermal conductivity of agricultural products can provide effective guidance for food storage[1]; the measurements of thermal conductivity of building materials are related to their energy saving evaluation[2-3]; and the tests of thermal conductivity of semiconductor materials in integrated circuits help to assess the stability of the components. Therefore, it is necessary to obtain accurate and reliable thermal conductivity of materials.

The methods of measuring the thermal conductivity can be divided into theoretical methods and experimental methods. The application of the theoretical methods is restricted because they need to obtain the microstructure of the material. Therefore, the experimental methods are more widely applied in practical engineering. These experimental methods can be divided into two categories, steady methods and transient methods[4-5]. The steady methods are widely used to measure the materials with low thermal conductivity due to their high accuracy and easy operation. However, the steady methods suffer from some drawbacks. They require a long time to complete the test and the measurement range is narrow[6]. In contrast, the transient methods have the advantages of short measuring time, high efficiency, wide range and non-destructivity[7]. As one of the most potential techniques among the transient methods, transient hot disk technique is proposed by Professor Gustafsson in 1991[8]. It is developed from the transient hot wire method. By bending the line heat source into the plane heat source with a double-spiral structure, the contact area between the probe and the sample is largely increased, which makes the heat transfer more efficient and the measurement accuracy is significantly improved. At present, the transient plane source (TPS) method has developed the measurement modules for bulk samples, slab samples, thin film samples and anisotropic samples[9-10]. The corresponding theoretical formulas and mathematical models have been established and the performance of this technique has been deeply studied.

The TPS technique is widely used to gain the

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thermal conductivity of most materials. During the measurement, it requires the probe to be sandwiched between two identical samples. As for some special materials, such as wall, equipment components and other single materials, this method is not satisfactory because there is only one piece of the measuring material and it cannot be covered on both sides of the probe. In order to enrich the measurement theory of TPS method so that it can measure the thermal conductivity of single specimen, single-side TPS method is proposed on the basis of standard TPS theory. The influencing factor of probe heat capacity is discussed through a correction formula. And the analysis on the accuracy and reproducibility of the single-side TPS method are conducted using the experimental materials of black rubber board, polymethyl methacrylate (PMMA), marble, soap stone, stainless steel, lead and Q235-A. F steel. In the following, the standard TPS technique is briefly introduced and the theoretical formula of single-side TPS is presented. Then the probe heat capacity is corrected. The experimental materials and apparatus are prepared and the tests are conducted. Finally, some useful conclusions are given through the discussion of the results.

1 Theory base

1.1 Theory of TPS technique

The TPS technique has become a standard method for measuring the thermal properties of materials and it can obtain the thermal conductivity, thermal diffusivity and specific heat capacity in one measurement. The core sensor of this method is a hot disk probe, as shown in Fig. 1. This probe is made of nickel because it has a large temperature coefficient of resistance in a wide temperature range. When conducting the experiment, the current can be led from one end to the other since the nickel is made into a shape of double spiral[11]. There are two insulating layers of Kapton covered on both sides of the sensor and this design can effectively improve the mechanical strength and service life of the probe.

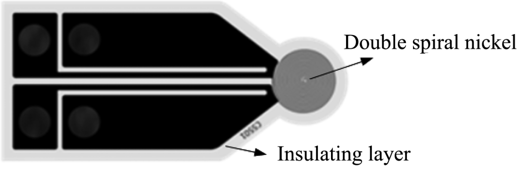

Fig. 1 Hot disk probe

The probe acts as both a heat source and a temperature sensor. Normally, it is placed between two pieces of identical samples with smooth surface. When a constant power is applied to the probe, the temperature of the probe increases because of the heat effect of the nickel and the heat is transmitted to the samples on each side of probe. The increasing temperature of the sample is reflected by the change of the probe resistance. This relationship can be expressed as

$$R(t)=R_0[1+\alpha\Delta T(t)], \qquad (1)$$

where R_0 is the resistance of the sensor before it is being heated; $R(t)$ is the resistance of the sensor at time t; α is the temperature coefficient of resistance of the sensor; ΔT is the mean temperature increase of the sensor.

The change of probe resistance value can be measured in real time using the hot disk hardware measurement system. The increase of the temperature mainly results from two parts: One part is caused by the heat flow passing through the probe insulating layer, the other part represents the temperature increase of the specimen surface during the measurement. This relationship can be given by

$$\Delta T(t)=\Delta T_{\mathrm{i}}+\Delta T_{\mathrm{s}}(t), \qquad (2)$$

where ΔT_{i} is the temperature difference across the probe insulating layer; $\Delta T_{\mathrm{s}}(t)$ is the temperature increase of the specimen surface. It is important to note that ΔT_{i} becomes constant after a short time because the insulating layer is thin and the power output is constant.

In order to obtain the accurate temperature rise in the sample surface, it is necessary to study the transient heat conduction problem in a semi-infinite boundary condition. Assuming that the sample around the probe is isotropic material, when the probe is placed between two samples, the probe can be regard as the heat source and the heat conduction differential equation in the sample is expressed as

$$\kappa\nabla^2 T+\frac{Q}{\rho c}=\frac{\partial T}{\partial t}, \qquad (3)$$

where κ is the thermal diffusivity of the sample; Q is the heat provided by the probe; ρc is the volumetric specific heat capacity of the sample.

The hot disk probe with a double-spiral structure can be considered as a number of concentric and equally spaced ring sources. According to the Fourier law and the first law of thermodynamics, the solution of the average temperature rise in Eq. (3) can be given by

$$\Delta T_s(\tau) = \frac{P_0}{\pi^{3/2} r\lambda} D(\tau), \tag{4}$$

where P_0 is the heating power output of the probe; λ is the thermal conductivity of the sample; r is the radius of the outermost ring of the probe; τ is the dimensionless time parameter and it can be defined as

$$\tau = \frac{\sqrt{\kappa t}}{r}, \tag{5}$$

$D(\tau)$ is the dimensionless function and defined as

$$D(\tau) = \frac{1}{m^2(m+1)^2} \times \int_0^\tau \left[\sum_{k=1}^{m} k \sum_{l=1}^{m} l \mathrm{e}^{-\left(\frac{k^2+l^2}{4m^2\sigma^2}\right)} I_0\left(\frac{kl}{2m^2\sigma^2}\right)\right] \frac{\mathrm{d}\sigma}{\sigma^2}, \tag{6}$$

where m is the number of the concentric ring sources; σ is the variable of integration; I_0 is a modified Bessel function.

According to Eq. (6), we can see that $D(\tau)$ is a function determined by the dimensionless time parameter τ since the probe circle m is known. We can get the relationship between t and τ from Eq. (5). Therefore, it is obvious that the average temperature increase $\Delta T_s(t)$ versus the function $D(\tau)$ is a straight line and the slope of the straight line is $P_0/(\pi^{3/2} r\lambda)$. The thermal conductivity of the measured sample can be calculated from the slope. However, the thermal diffusivity κ is unknown before the experiment. In order to accurately obtain the thermal conductivity of the sample, the iterative method is usually used to solve it. A linear relationship between $\Delta T_s(\tau)$ and $D(\tau)$ is established by a least-squares fitting procedure. The optimum fitting is the thermal diffusivity of the sample, and the thermal conductivity of the sample can be obtained from the slope of best fitting line. Those calculations can be completed by the hot disk software.

1.2 Theoretical formula of single-side TPS technique

The single-side TPS method is different from the standard TPS method since it requires only one piece of sample. In the process of measurement, one side of the probe is the testing sample and the other side is the background material with known thermal conductivity and thermal diffusivity. A schematic diagram of the measurement structure is depicted in Fig. 2.

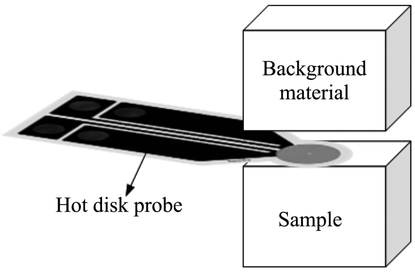

Fig. 2 Measurement structure of single-side TPS

When a constant power P_0 is applied to the probe, it begins to heat the samples on both sides. For the standard TPS method, the probe is placed on the center of the sample surface and both samples are identical, and the heating power applied to each specimen can be considered as $P_0/2$, as show in Fig. 3. However, for the single-side TPS method, the heat powers of the probe is not distributed equally to the materials on both sides of the sensor due to the difference of the thermal properties between the background material and the measured sample. Fig. 4 shows the different power flowing into the different materials. It is assumed that the heating power applied to the sample is P_p, And $P_0 - P_p$ is the power applied to the background material.

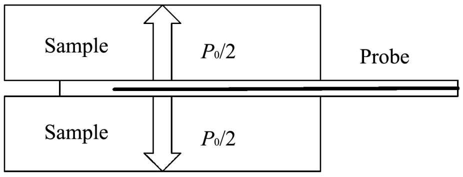

Fig. 3 Power distribution of identical samples

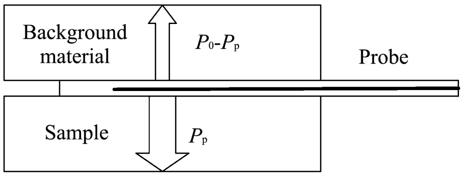

Fig. 4 Power distribution of different materials

Gustafsson has studied the relationship between the total power and the heating power applied to measured sample when a hot-strip probe is in contact with two different materials[12]. And this expression can be represented as

$$P_p = \frac{P_0}{1 + \frac{\lambda_p}{\lambda}\left(\frac{\kappa}{\kappa_p}\right)^{1/2}}, \tag{7}$$

where λ_p is the thermal conductivity of the background material; K_p is the thermal diffusivity of the background material.

The above expression is established based on the assumption that the experimental situation can be described by the plane-source solution. Therefore, it is applicable to the single-side TPS condition[13]. And Eq. (7) shows that the power applied to the background material is only related to its thermal conductivity and thermal diffusivity. Supposing that heat transfer ratio will not affected by the shape of the heat source between the testing sample and the background material, when the probe is in contact with two different materials, the heating process can still be regarded as the transient heat conduction problem in a semi-infinite boundary condition as long as the sizes of the two different materials are large enough to meet the requirements of measurement. According to Eq. (4), it is possible to give the average temperature rise in a single-side measurement with the following expression as

$$\Delta T_{\mathrm{s}}(\tau)=\frac{2P_{\mathrm{p}}}{\pi^{3/2}r\lambda}D(\tau). \tag{8}$$

From Eqs. (7) and (8), we can obtain the solution of thermal conductivity by

$$\lambda=\frac{2P_0}{\pi^{3/2}\left(1+\frac{\lambda_{\mathrm{p}}}{\lambda}\left(\frac{\kappa}{\kappa_{\mathrm{p}}}\right)^{1/2}\right)}\frac{D(\tau)}{\Delta T_{\mathrm{s}}(\tau)}. \tag{9}$$

The thermal conductivity λ_{p} and thermal diffusivity κ_{p} of the background material are known before the measurement. Similar to the solution process of standard TPS method, we can see that $\Delta T_{\mathrm{s}}(\tau)$ is proportional to $D(\tau)$. And we can get the value of $\Delta T_{\mathrm{s}}(\tau)$ from the hot disk data acquisition system. By an iterative procedure with thermal diffusivity κ, the optimum fitting is the thermal diffusivity of the sample and the thermal conductivity of the sample can be calculated by Eq. (9).

2 Mathematical compensation model of probe heat capacity

In the process of deriving the probe temperature increase model of single-side TPS measurement, all the equations are proposed based on the assumption that the output power of the probe element is constant in the whole process of measurement and all the heat generated by the output power should be transferred to the sample and background material. However, the heat capacity of the probe cannot be ignored in the actual measurements. A part of the heat consumed by the probe insulating layer, which leads to the actual total heating power being less than the set output power of the probe. During the measurement. The expression of the power applied to increasing the temperature of the probe itself can be given as

$$\Delta P_{\mathrm{probe}}(t)=\mathrm{d}\pi r^2(\rho c)_{\mathrm{probe}}\frac{\Delta\overline{T}(t)}{t}, \tag{10}$$

where $\Delta P_{\mathrm{probe}}$ is the power consumed by the probe itself; d is the total thickness of the probe; $(\rho c)_{\mathrm{probe}}$ is the volumetric specific heat capacity of the probe; $\Delta\overline{T}(t)$ is the probe temperature increase at time t.

Supposing that the input power of the probe is constant, the actual heating power of the probe at time t can be given as

$$P(t)=P_0-\Delta P_{\mathrm{probe}}(t)=P_0-\mathrm{d}\pi r^2(\rho c)_{\mathrm{probe}}\frac{\Delta\overline{T}(t)}{t}. \tag{11}$$

From Eq. (11), we can see that power loss is caused by the existence of the heat capacity of the probe, which leads to the measurement errors of the results. By replacing P_0 in Eq. (9) with in Eq. (11), the thermal conductivity of measured sample in single-side TPS measurement can be rewritten as

$$\lambda=\frac{2\left(P_0-\mathrm{d}\pi r^2(\rho c)_{\mathrm{probe}}\frac{\Delta\overline{T}(t)}{t}\right)}{\pi^{3/2}r\left(1+\frac{\lambda_{\mathrm{p}}}{\lambda}\left(\frac{\kappa}{\kappa_{\mathrm{p}}}\right)^{1/2}\right)}\frac{D(\tau)}{\Delta T_{\mathrm{s}}(\tau)}. \tag{12}$$

3 Measurement preparations

3.1 Experimental samples and background material

It should be noted that the single-side technique is only applicable to isotropic bulk materials. In order to evaluate the accuracy and reproducibility of this method, experiments have been carried out on seven different single specimens, including black rubber board, PMMA, marble ,soap stone, stainless steel, lead and Q235-A. F steel. And we choose extruded polystyrene (XPS) as the background material since it has low heat transfer ability.

Based on the principle of sample preparation, the surface of the sample should be smooth enough and it helps to avoid the contact thermal resistance during the experiment. In addition, the sample size is also need to be large enough to satisfy the assumption that the sensor is placed in an semi-infinite medium. This assumption requires that the distance of heat transferred inside the sample must be smaller than the outer boundaries of the sample. It depends on the thermal diffusivity and measuring time of the testing sample[13]. The probing depth is expressed as

$$\Delta_p = 2\sqrt{\kappa t_{\max}}\,, \tag{13}$$

where Δ_p is the distance of the heat flow into the specimen; $t_{\max}$ is the total time of experiment.

In order to making a good measurement, the longitudinal size of the sample should be greater than the probing depth and the transverse dimension should be larger than the sum of the probe diameter and two times of the probing depth. Therefore, the actual whole sizes of these samples are black rubber board (100 mm × 100 mm × 30 mm), PMMA (50 mm× 50 mm × 30 mm), marble (60 mm × 60 mm×50 mm), soap stone (60 mm × 60 mm × 50 mm), stainless steel (50 mm×50 mm×50 mm), lead (50 mm×50 mm×50 mm) and Q235-A. F steel (90 mm×90 mm×50 mm).

During the measurement, the probe is placed between the measured sample and the background material. In order to avoid the heat from the probe spreading through the boundary of the background material to the environment, the size of the background material in this experiment is 100 mm× 100 mm×50 mm.

3.2 Apparatus

Based on the theory of single-side TPS technique, when the probe is placed between the background material and the specimen, a heat pulse in the form of a stepwise function is produced by an electrical current through the probe to generate a dynamic temperature field within the specimen. The probe acts as both a heat source to increase the sample temperature and a component that records the change of resistance during the measurement. When constant heating power applied to the probe, the probe resistance increases with the increase of the probe surface temperature, which makes the imbalance of the bridge test system produce the potential change.

Fig. 5 shows the measurement apparatus of the hot disk system. The probe and samples are placed in a thermostat chamber. The heating power is provided by the power meter and the voltage value of bridge system is measured by the digital voltmeter. The software is installed on the computer and the whole measurement process is operated on the computer. In order to keep the output power is constant, the value of series resistance in bridge system should be close to the resistance of the probe and its lead. During the measurement. The interval time should not be less than 15 min because it is necessary to make the sample and probe return to the thermal condition before the measurement. The function of temperature increment with time can be obtained through the measurement of electrical parameters.

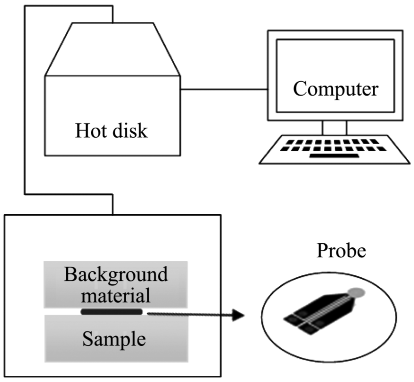

Fig. 5 Apparatus of single-side TPS measurement

4 Results and discussion

The hot disk thermal analyzer is used to measure the thermal conductivity and thermal diffusivity of the seven samples based on the standard TPS technique. The results are shown in Table 1. These measured values are taken as the standard values of these samples. In order to evaluate the performance of the single-side TPS technique, a series of validation tests are carried out on the seven samples. During the measurement, the hot disk probe is placed between the sample and the background material, as shown in Fig. 2. The background material XPS is an insulation material and its thermal conductivity and thermal diffusivity are 0.032 W/(m · K) and 0.60 $\mathrm{mm^2/s}$, respectively. The heat capacity of the hot disk probe used in these experiments is 9.84×10^{-3} J/K and the radius and thickness are 6.403 mm and 60 m, respectively.

The temperature rise response on the probe is collected by the hot disk data acquisition system during the single-side experiments. Firstly, the reproducibility tests of thermal conductivity have been conducted at room temperature and the initial humidity of the environment is about 40% RH. The experimental material is PMMA since it is often used as a standard sample. On the premise of choosing the appropriate measurement parameters, the thermal conductivity of PMMA is calculated according to the

Eq. (9). Measurement results are shown in Fig. 6.

Table 1　Measurement results of samples by using a Hot disk thermal analyzer

Sample material	Heating power (W)	Measuring time (S)	Thermal conductivity (W/(m·K))	Thermal diffusivity (mm^2/S)
Black rubber board	0.05	200	0.037 10	0.455 0
PMMA	0.08	160	0.200 3	0.130 7
Marble	0.1	40	0.445 7	0.377 7
Soap stone	0.5	10	1.822	0.861 6
Stainless steel	1	10	14.58	3.558
Lead	2	3	34.83	26.67
Q235-A. F steel	3	1	74.61	21.04

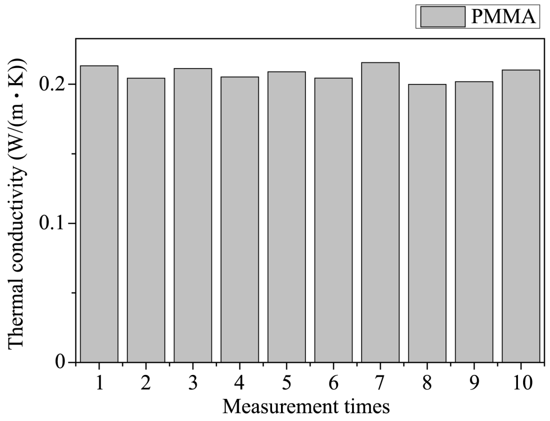

Fig. 6　Measurement results of PMMA

The above data are measured in the same environment using single-side TPS method. And the reproducibility of this technique is evaluated by the parameter of relative standard deviation (RSD). The RSD is 2.3% and it shows that single-side TPS method has a good reproducibility.

Meanwhile, the verification experiments about the accuracy of the single-side TPS method have been performed. The measurement results of the seven experimental materials are shown in Table 2. In standard TPS measurements, the power is equably applied to each side of the probe. However, during the single-side TPS measurements, the thermal properties of the background material are different from that of those measured samples. In order to ensure that the power divided into the sample in single-side experiments is the same as that in standard measurements. The total output power of the sensor can be calculated by Eq. (7) because the power is proportionally distributed to each side of the sensor and the distribution ratio is only related to the thermal properties of the tested materials. The following results with or without compensation of heat capacity are calculated by Eqs. (9) and (12).

Table 2　Measurement results of single-side TPS with or without compensation of heat capacity

Sample material	Heating power (W)	Without compensation		With compensation	
		Thermal conductivity (W/(m·K))	Relative error (%)	Thermal conductivity (W/(m·K))	Relative error (%)
Black rubber board	0.043 8	0.038 62	4.10	0.038 13	2.77
PMMA	0.043 0	0.207 5	3.59	0.207 0	3.36
Marble	0.051 1	1.891	3.78	1.889	3.68
Soap stone	0.253	6.637	3.48	6.632	3.40
Stainless steel	0.503	14.95	2.52	14.94	2.45
Lead	1.006	35.91	3.10	35.89	3.04
Q235-A. F steel	1.504	76.77	2.89	76.71	2.82

From the measurement results of Table 2, it can be seen that the relative errors of the thermal conductivity are less than 5%, which indicates that the single-side TPS method proposed in this paper has a good measurement accuracy in a wide range (0.03—70 W/(m·K)). Compared with the standard TPS method, the single-sided TPS method only needs single specimen, which provides support for the measurement of single materials.

As shown in Fig. 7, the existence of the probe heat capacity makes the measurement results larger than the actual values. With the compensation of the probe heat capacity, the measurement result is more accurate. Among the seven samples, the relative errors of stainless steel (14.58 W/(m·K)) is less than that of the other materials and it makes the curve show a sudden downtrend. This is because the surface of stainless steel could be very smooth during the preparation process. In addition, the influence of probe heat capacity on the measurement results decreases with the increase of thermal conductivity. For materials with high thermal conductivity, the heating power is relatively large and the corresponding temperature difference is relatively small. According to Eq. (10), the proportion of the power consumed by the probe heat capacity can be neglected. Therefore, it can be found that the relative errors with or without compensation are almost the same when the materials have relatively large thermal conductivity.

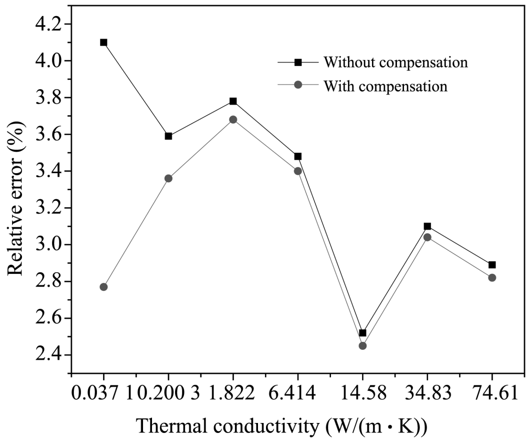

Fig. 7 Relative errors with or without compensation of heat capacity

5 Conclusion

In this paper, single-side TPS method for measuring the thermal conductivity of single material is presented. The theoretical formula is given and the heat capacity of the probe is compensated. A series of test experiments have been carried out to study the measurement performance of this method. The results indicate that the single-side TPS method has high measurement accuracy and good reproducibility. For the samples with thermal conductivity of 0. 03—70 W/(m · K), the accuracy of the method can reach 5% and the RSD is less than 3%. For the compensation of the probe heat capacity, it can effectively improve the accuracy of materials with lower thermal conductivity. This method can be used for transient measurements of thermal conductivity of single material.

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TPS单面法测量材料导热系数

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摘　要： 在传统瞬态平面热源(Transient plane source，TPS)法测量理论的基础上，提出了一种可用于测量单试件材料导热系数的TPS单面法。通过研究半无限边界条件下瞬态热传导问题，推导了TPS单面法的理论公式，分析了测量过程中探头热容对测量结果的影响，建立了相应的数学补偿模型，并在常温常压下利用hot disk探头对不同材料开展了一系列实验。结果表明，TPS单面法测量材料导热系数的相对误差小于5%，相对标准偏差低于3%。该方法具有良好的准确度和重复性，为不满足标准TPS法测试要求的单试件材料提供了可行的测量手段。

关键词： TPS单面法；导热系数；单试件材料；探头热容

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