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Measuring solid material's thermo-physical properties wtih non-stationary hot wired method

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Based on the in-depth analysis of the basic principle of non-stationary parallel hot wire method and existing measuring standards, with the foundation of heat transfer differential mathematical model of hot wire in cylindrical coordinates, research is made on the measurement method of solid materials' thermal conductivity and thermal diffusivity. The temperature measuring points, which are originally arranged in parallel with hot wire, are changed to specimen thickness direction. Combined with computer programming, Numerical algorithms are used to directly measure the materials' thermal conductivity and thermal diffusivity without looking up the table. It analyzes in detail the effect of heat penetration on measurement results, explores treatment measures, and then develops a device for thermo-physical properties measurement. Measuring experiments are conducted on thermo-physical properties of glass, asbestos, diatomite bricks and others, and the experimental results are in close compliance with individual thermo-physical properties tester and literature values, with less than 5% of relative error.

Key words: Parallel hot wired method, Solid materials, Thermal conductivity, Thermal diffusivity, Heat penetration effect.

Introduction

Thermal conductivity λ, thermal diffusivity α and specific heat capacity C_p are importantly basic parameters [1-2] for materials. While, with the rapid development of materials science and continuous enhancement of energy saving requirement of buildings, fast and reliable obtaining of solid materials' thermal-physical properties with high accuracy becomes one of developing trends. As a classic non-stationary measuring method, hot wire method has advantages of short measuring time, high measuring precision and etc., and has been successfully applied in the thermal conductivity [6] measurements of plate molding materials [3-4], fluid [5-6] and others materials [7-10]. National standard of thermal conductivity measurement of the fireproofing (GB/T 5009-2006) [11] and the measuring method of thermal conductivity of non-metallic solid materials (GB 10297-98) [12] both take hot wire method as measuring means. J. DeBoer and others firstly proposed parallel hot wire method in 1980 to simultaneously measure the materials' thermal conductivity and thermal diffusivity [13]. However, as lots of simplification was made to the model, it got large errors in thermal diffusivity measuring [18]. Chen Qinghua conducted appropriate improvements to the theoretical model of hot wire method, successfully applied it to the simultaneous determinations of thermal conductivity and thermal diffusivity of bulk coal, and designed a measurement device [15] which was only available for bulk materials. Based on the principle of hot wire method, Luo Xiaoqin proposed a method of simultaneous determinations of thermal conductivity and thermal diffusivity of plate materials [16]. However, in order to get the slope and intercept, it removed the infinite series in the impulsive solutions of one-dimensional cylindrical heat transfer differential equations, which was approximate linear equation, and thus measurement accuracy could not be guaranteed. Based on the basic principle of non-stationary parallel hot wire method and without many simplifications to mathematical model, this paper conducts simultaneous measurements of solid materials' thermal conductivity and thermal diffusivity by means of numerical algorithms. The hot wire collocation mode, which is traditionally double-teamed at top and bottom by specimen blocks and filled with specimen powder in the gap at the parallel direction of hot wire, is changed to all around teamed hot wire by specimen blocks and just fill specimen powder in vertical gap. By such way, it reduces the experimental operation difficulty. As for device structures, the

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temperature measuring points are arranged at the top of (specimen thickness direction) instead of originally in parallel with hot wire. Finally, the developed device is used to conduct measuring experiments of thermo-physical parameters on doors and glass, asbestos, diatomite bricks, silica bricks, etc. to explore the heating power of hot wire and the influence of thermal breakdown effect on the measurement results.

Theoretical Model

Assuming a slender linear heat wire source with constant power is placed in a homogeneously isotropic solid medium and thermal conductivity λ and thermal diffusivity α of the dielectric material are constant, then the temperature variation in the medium would be represented by the thermal differential equation in cylindrical coordinate and its corresponding initial boundary conditions as follows [17]:

$$\frac{\partial\theta(\tau)}{\partial\tau}=\left(\frac{\partial^2\theta(\tau)}{\partial r^2}+\frac{1}{r}\frac{\partial\theta(\tau)}{\partial r}\right) \tag{1}$$

$$\tau=0, \theta(\tau)=0 \tag{2}$$

$$\tau=r_0, q=-2\pi\lambda r_0\frac{\partial\theta(\tau)}{\partial\tau}=const \tag{3}$$

By adopting separation of variables, the analytical solution of formula (1) in initial conditions (2) and boundary conditions (3) is:

$$\theta(\tau)=\frac{q}{4\pi\lambda}\square -Ei\left(\frac{-r^2}{4\alpha\tau}\right) \tag{4}$$

In which, θ (τ) is the excessive temperature at the time of *ô* with distance to the hot wire source *r,* K; λ is the thermal conductivity of solid medium, *w/m.k*; α is the thermal diffusivity of the solid medium, m^2/s; r_0 is the radius of the linear heat source, m; *q* is the power of linear heat source with unit length, *w/m*, while *Ei* is the exponential integral.

Making $p=\frac{r}{2\sqrt{\alpha\tau}}$, then the formula (4) can be rewritten as:

$$\theta(\tau)=\frac{q}{2\pi\lambda}\int_p^\infty\frac{1}{u}\square e^{-u}du=\frac{q}{2\pi\lambda}\Omega(p) \tag{5}$$

In the formula $u=\frac{r^2}{4\alpha\tau}$, then it has

$$f(p)=\frac{\theta(\tau)}{\theta(2\tau)}=\frac{-E_i\left(\frac{-r^2}{4\alpha\tau}\right)}{-E_i\left(\frac{-r^2}{8\alpha\tau}\right)}=\frac{\Omega(p)}{\Omega\left(\frac{\sqrt{2}}{2}p\right)} \tag{6}$$

Where Ω (P) can be expanded [14] into the following forms:

$$\Omega(p)=-\frac{1}{2}E(-p^2)=\frac{1}{2}\left(-0.57721-\ln p^2-\sum_{n=1}^{+\infty}\frac{(-1)^n p^{2n}}{n\square n!}\right) \tag{7}$$

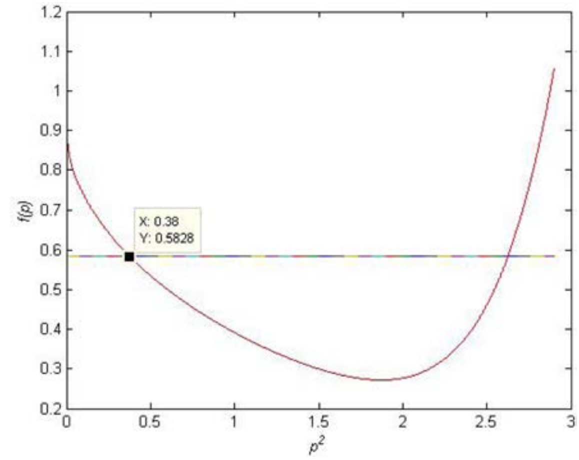


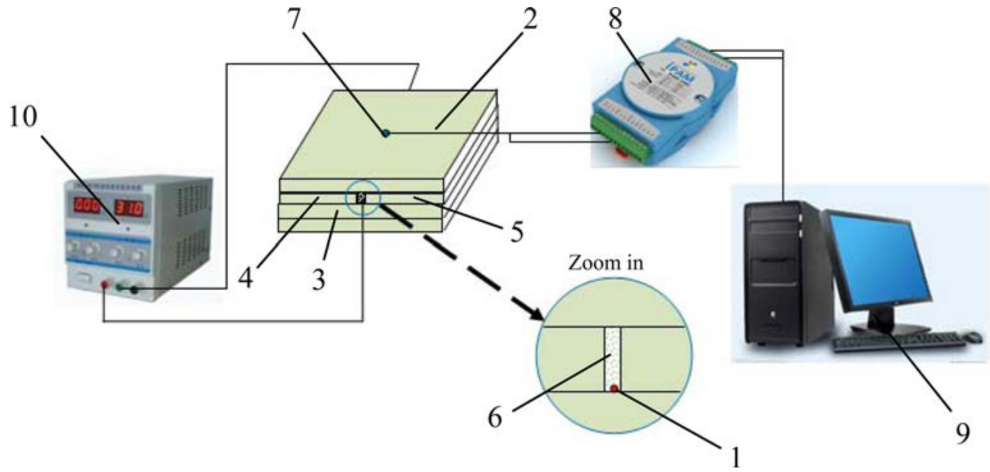
Fig. 1. Relationship between p^2 and *f* (*p*).

Formula (7) contains infinite series items, but after the trail calculation, the first six items taken are sufficiently to meet the accuracy requirements. As a transcendental equation, the formula cannot be solved directly but by numerical algorithms combined with computer programming. It is obvious that after measuring θ (τ), the thermal conductivity λ can be obtained by using formula (5), and θ can be further measured. The thermal diffusivity α can be calculated by formula (6) and further specific heat capacity C_p. Figure 1 shows the relationship between *p2* and *f (p)* with abscissa p^2 value range [0.001, 2.900] and *(p)* [0.2720, ∞]. However, it is not just the monotonic variation relationship of increasing or decreasing between *f (p)* and p^2, but at first monotonically decreasing and then increasing, i.e., there are two corresponding *p* for specific *f (p)*. Furthermore, it is necessary to make judgment and remove an invalid *p* value.

Measuring Device

According to measuring principle, the measuring device of solid materials' thermo-physical properties is designed and fabricated. The theoretical model is as shown in Figure 2.

Hot wire made by nichrome resistance with a diameter of 0.1 mm, is caught in the middle of specimen I, specimen II, specimen III and specimen IV with the same thickness, while the gap of hot wire caught by specimen III and specimen IV is filled in by finely grinding specimen powder to minimize the impact caused by the thermal contact resistance. Being different from the arrangement of traditionally parallel hot wire method, both sides of the hot wire are double-teamed by the same kind of specimen, not all wrapped with specimen powder any more so as to reduce the experimental operation difficulty. Temperature is measured by thermocouple, and the measuring points' location is above the hot wire. The temperature signal



1-hot wire; 2-specimen I; 3-specimen II; 4- specimen III; 5- specimen IV; 6- specimen powder; 7-temperature measuring point; 8- data acquisition module; 9- microcomputer; 10-DC regulated power supply
Fig. 2. Theoretical model of measuring system.

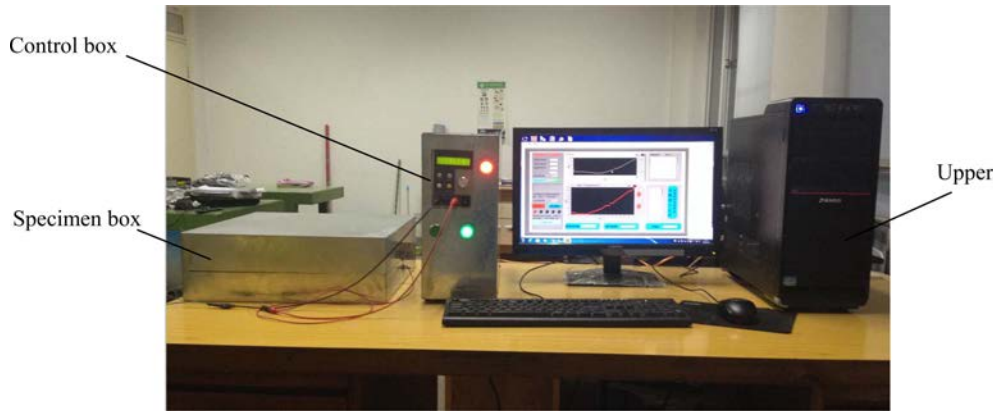


Fig. 3. Photo of measuring system.

is collected by temperature acquisition module and transferred into the microcomputer. Assuming the specimen thickness is δ, as the hot wire is relatively smaller compared with specimen size, thus it can be considered that hot wire is heated at the bottom of the specimen, and then the distance between temperature measuring point and hot wire would be denoted as 2δ,. Hot wire is supplied by DC regulated power, with adjustable output voltage of 0-220 V.

Photo of experimental device is shown in Figure 3, which is mainly composed of specimen box, control box and host computer. The specimen box's enclosure is made of stainless steel and lined with insulating materials to avoid the impact of environmental and temperature fluctuations on the specimen during the measuring. The regulated power supply and data acquisition modules are centrally located in the control box. Based on the collected temperature signal, the host computer calculates the thermal conductivity and thermal diffusivity of the specimen by combining the procedural formula.

Experimental Measuring and Analysis

Measuring analysis of glass thermo-physical properties

A particular type of float glass with thickness of 3.5 mm is selected to conduct an experiment with the density of 2530 kg/m^3 and ambient temperature of 300K. Firstly, it is processed into a number of specimen blocks with the size of 300 × 300 mm. Placed on two stacked specimens, the hot wire is sandwiched with two specimens on the left and right sides, and the gap is filled with finely grounded glass powder, then the hot wire is covered with another specimen block. Temperature thermocouple is arranged on the top surface of the specimen block above the hot wire with heating power as 48.67 W/m. Temperature sampling period is 6S. According to the collected temperature data, formula (6) and formula (7) are firstly used to calculate and obtain the thermal diffusivity á, and then formula (5) to obtain the thermal conductivity λ. Specific results are described in Table 1. Based on p_1^2 and p_2^2, the corresponding thermo-physical parameter values can be obtained respectively. However, apparently, the value calculated according to p_2^2 is not

Table 1. Calculation results of thermal conductivity and thermal diffusivity.

Time /S	Thermal rise /K	$f(p)$	P_1^2	Thermal diffusivity α /($10^{-7}m^2/s$)	Thermal conductivity λ / w/m · k	P_2^2	Thermal diffusivity α /(10^{-7} m^2/s)	Thermal conductivity λ / w/m · k
24	0.6	0.316	1.414	3.6	0.711	2.201	2.3	0.231
30	0.9	0.360	1.163	3.5	0.718	2.335	1.7	0.123
36	1.3	0.419	0.903	3.7	0.759	2.430	1.4	0.089
48	1.9	0.452	0.774	3.3	0.662	2.499	1.0	0.052
60	2.5	0.462	0.712	2.9	0.574	2.500	0.8	0.043
72	3.1	0.484	0.643	2.6	0.523	2.520	0.7	0.021
96	4.2	0.483	0.643	2.0	0.389	2.520	0.5	0.021
120	5.4	–	–	–	–	–	–	–
144	6.4	–		–	–	–	–	–
192	8.7	–		–	–		–	–

accord with the facts, therefore, only the calculation results of the thermal conductivity and thermal diffusivity based on p_1^2 can be taken. After heating the hot wire, the variations of temperature rise are detected from 18S, and the data after 24S are selected to conduct the parameter calculation of thermo-physical properties. What can be seen from the data of Table 1 is that the calculation results of the thermal conductivity λ and thermal diffusivity α before 96S are relatively stable, and then gradually decrease. Therefore, only the averaged calculation data of the first three are taken as the final measuring results, and then the thermal conductivity λ and thermal diffusivity α of the measured specimen are respectively obtained as 0.729 w/m · k and 3.6e-7 m^2/s. Multiple thermo-physical parameter values can be calculated at various times, and the proximity of these values to each other may reflect the stability of the measurement results [17], which has nothing to do with the true value and is measured by deviation. Deviation (d) means the difference between individual measured value X and arithmetically averaged X of n-times measured values. Deviation is divided into absolute deviation and relative deviation, and relative deviation is adopted here.

$$\text{relative deviation} = \Delta d = d / \overline{X} \times 100\% \quad (8)$$

By formula (8), it can separately calculate and obtain the thermal conductivity λ and the thermal diffusivity α, with the maximum relative error of the calculated values separately as 4.1% and 2.85%, which fully meets the accuracy requirements.

In order to verify the accuracy of the measured parameters, the calculated thermal conductivity ë and thermal diffusivity α are taken into formula (5) to calculate the temperature rise at the measuring point. At the same time, the simulation model in Fluent is established. According to the measurement results, the thermo-physical parameters of the material is set for the simulating calculation of the temperature variations at the temperature measuring point during 200S when hot wire is heated. The comparisons with the measured temperature variations are shown in Figure 4.

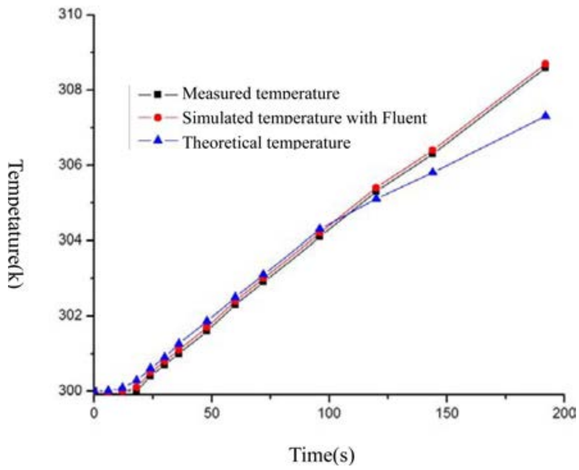


Fig. 4. Contrast between measurement, theoretical and simulating temperature rise.

It can be seen from the figure that the measured temperature rise is more consistent with Fluent simulation calculation result, indicating the accuracy of calculation result of the thermo-physical parameters. However, starting from the 96S, the gradually increasing difference between the theoretical temperature rise and the masured one reflects that, the specimen surface insulation leads to the actual situations that the untimely diffusion of heating transferred by hot wire, and then the accumulation of more and more heat have gradually increasing impact on the temperature rise of the measuring point. Before the 96S, as the impact brought about by the heat accumulation effect is not yet obvious, and the error between the measured temperature rise and the theoretical one is relatively small, thus it can be used to calculate the thermo-physical properties, being called effective measuring time.

Thermal conductivity tachometer of Shotherm QTM-D2 type is used to measure the thermal conductivity of the same kind of glass specimens, and differential

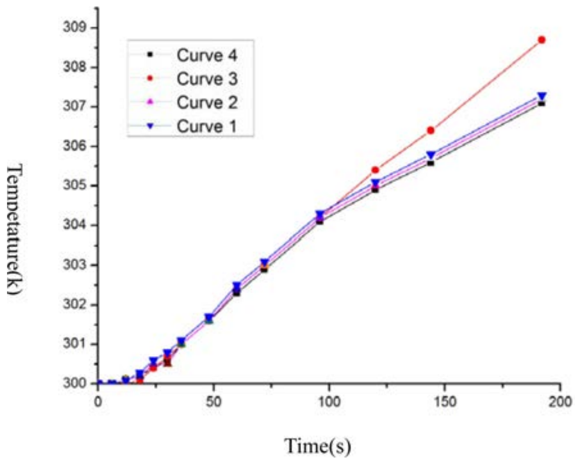


Fig. 5. Temperature rise of the measuring point in various specimen condition.

Table 2. Measurement results of thermo-physical in different specimen condition.

Experimental conditions	Thermal diffusivity α /(10^{-7} m^2/s)	Thermal conductivity λ / w/m · k
Cover insulation	3.60	0.729
Melt specimen	3.53	0.726
Not melt specimen	3.56	0.727

thermal analysis instrument is adopted to measure the specific heat capacity, with measurement results respectively as 0.721 w/m · k and 800 J/kg · K. Then through conversion, the heat diffusion rate is 3.56e-7 m^2/s, which is basically consistent with the measured result in this paper.

What can be shown from the experimental results above is that it will cause impact on the temperature rise of the measuring point when the heat penetrates the specimen surface. In order to further verify whether too big error of parameter calculation will be caused, by means of melting the glass specimen used in the previous experiment is manufactured into some glass specimen blocks with thickness as 14 mm and size as 300 mm × 300 mm respectively. The specimen surface is directly grooved so that the hot wire and thermocouples can be embedded. Still, without melting, four glasses with thickness of 3.5 mm are stacked directly and put on the temperature measurement thermocouples. Both sides of the thermocouple are filled with amount of glass powder to conduct thermo-physical measurement experiment, keeping constant heating power of the hot wire. Figure 5 presents the comparative temperature curve of the measuring points in the experiment.

As can be seen from the figure, without thickening glass by melting, within the range of the first 96S it is nearly the same between the temperature measuring values of thermocouples covered by insulating material (Curve 3), and the temperature experiment measuring of the specimen coverage of the temperature measurement

Table 3. Measurement results of thermo-physical parameters of various specimens with tacheometer and differential thermal analysis instrument.

Materials	Thermal capacity C_p /J/kg·K	Thermal conductivity λ / w/m · k	Thermal diffusivity α /(10^{-7} m^2/s)
diatomite bricks	882	0.211	3.40
asbestos	800	0.113	1.56
silica bricks	850	1.012	6.25

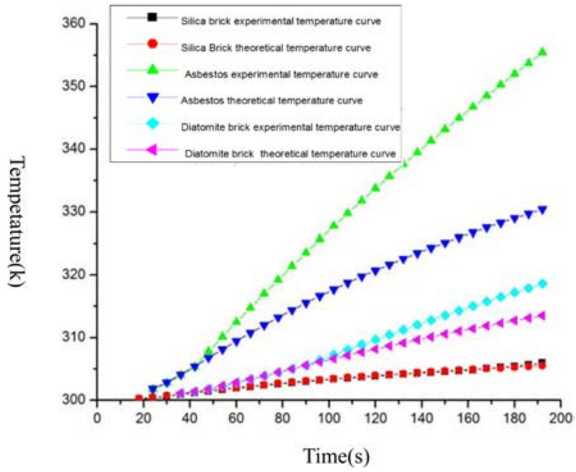


Fig. 6. Experimental and theoretical temperature rise of various specimens When the power of hot wire is 48.67 W/m.

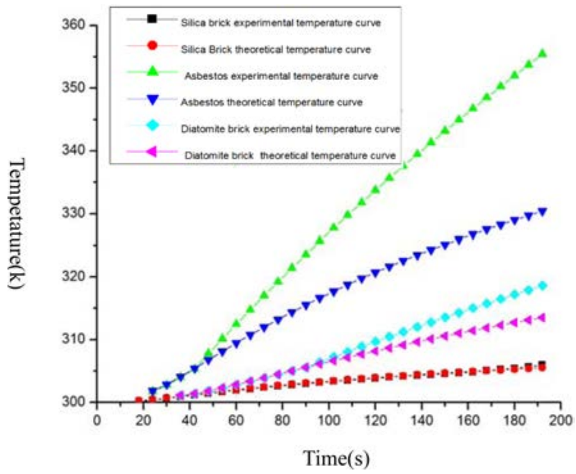


Fig. 7. Experimental and theoretical temperature rise of asbestos With different hot wire heating power.

thermocouples processed by melting (Curve 4). While, when the specimen is directly covered on the thermocouples by means of superposition method (the surface is processed to ensure good contact) instead of melting, the temperature errors between the experimental values (Curve 2) and the theoretical values (Curve 1) as well as Curve 4 are less than 0.2 K. If these three cases all take the temperature data between 24S and 96S to make parameter calculation, the final results would be as shown in Table 2. As what can be seen is that it can also

Table 4. Comprehensive analysis of experimental results of four materials.

Materials	Thermal diffusivity α /m^2/s	Thermal conductivity λ / w/m·k	Testing results of λ with tacheometer /w/m·k	Relative error /%	Measuring results of ë in literature [13][15] / w/m·k	Relative error /%
diatomite firebrick	3.26e-7	0.220	0.211	4.09	0.227	3.18
asbestos	1.58 e-7	0.116	0.113	2.59	0.10-0.14	–
silica brick	6.32e-7	1.035	1.012	2.22	1.051	1.55

ensure adequate measurement accuracy of parameters when the measuring time is properly controlled, the data during the effective test period is used to conduct calculation, and heat penetration effect is avoided without thickening the specimen through melting.

Comprehensive analysis of measurement of materials' thermo-physical properties

An experimental analysis of measurement of thermo-physical properties of asbestos, diatomite bricks and silica bricks is conducted respectively. The specifications of asbestos plate are 6 mm thickness, with length and width 200 mm × 200 mm separately and density 880 kg/m^3. The length and width of the diatomite bricks are 230 mm × 114 mm with thickness 10 mm and density 700 kg/m^3. The length and width of the silica bricks are 230mm × 173 mm with thickness 10 mm and density 1900 kg/m^3. All specimens are finely grinded into a number of powder to fill the gap, and then the thermo-physical measurement is proceeded as above mentioned experimental steps. Firstly, tacheometer and differential thermal analysis instrument are used to measure the thermal conductivity and specific heat capacity of each specimen, and the results are shown in Table 3.

What is presented in Figure 6 is the measured temperature rise curve of various specimens when the power of hot wire is 48.67 W/m. It can be seen that due to the comparatively small thermal conductivity and thin thickness, the temperature rise of the measuring point of the asbestos is relatively rapid resulting in a shorter effective measuring time, i.e., starting from the 30S, the difference between the experimental and theoretical temperature rise gradually become larger. Thus, no enough data is available to calculate the thermo-physical parameters. While, for the diatomite bricks and silica bricks with comparatively greater thermal conductivity, the temperature rise are relatively slower and the effective measurement time periods are 96S and 192S separately, which can meet the measurement requirements.

Figure 7 is the temperature rise conditions when the heating powers of the asbestos plate are 30 W/m and 10 W/m respectively. It can be observed that when the heating power is decreased to 30 W/m, the temperature rise of the asbestos plate is relatively slowed, correspondingly, the effective measurement time increases (to around 54S). However, it still cannot get enough temperature data. While, when the heating power is 10 W/m, the temperature rise is further gentle and the effective measurement time increases to 144s, reaching the requirements of measurement time. Thus, in the actual measurement, the heating power should be adjusted appropriately according to the approximate range of the thermal conductivity of the measured materials to ensure the measurement accuracy.

On the basis of the above analysis, different heating power conditions are adopted to separately carry out thermo-physical measuring experiments on asbestos (10 W/m), diatomite firebrick (30 W/m) and silica brick (48.67 W/m). The measuring results comparison with the literature [17], [19] and the tacheometer are presented in Table 4.

Conclusions

(1) Based on the heat transfer differential mathematical model of hot wire in cylindrical coordinates, without model simplification, by means of numerical algorithms, the simultaneous measurement of the thermal conductivity and thermal diffusivity of the solid materials can be realized.

(2) The temperature measuring points are arranged in the direction of the specimen thickness, and the hot wire is teamed all around by specimens and it is only need to fill specimen powder in vertical gap, which reduce the experimental operation difficult. When the specimen with thinner thickness and smaller thermal conductivity is measured, the effects of heat penetration and heat accumulation on the adiabatic surface will cause the temperature rise of the measuring point higher than the actual one, thus brings error to the measurement results of thermo-physical properties. However, in the initial period, the impact is relatively small and the error can be neglected, which is called effective measurement time. It is proved by the experiments that by adjusting the heating power of the hot wire and controlling the measurement time, the influence of heat penetration effect can be eliminated as much as possible. Thereby, without taking the method of melting specimens to increase thickness, it can also ensure the accuracy of parametric measurement and make the specimen manufacture easier.

(3) For the materials with lower thermal conductivity, the heating power of hot wire should be decreased

properly to assure adequate and effective measurement time. While for measuring the materials with higher thermal conductivity, the heating power should be increased to make the specimens have obvious temperature rise. By using the designed measuring device, the measuring experiments of the thermo-physical properties are conducted on glass, diatomite firebrick, asbestos, silica bricks and other materials. The measurement results of the thermal conductivity and thermal diffusivity are relatively consistent with the values of similar device and in related literatures, with the relative error less than 5%, revealing better measurement accuracy.

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The Conflict of Interests Declaration

We declare that we do not have any commercial or associative interest that represents a conflict of interest in connection with the work submitted.