RESEARCH ON EXPERIMENTAL PARAMETER SELECTION FOR MEASURING THERMAL DIFFUSIVITY BY LASER FLASH METHOD AT ROOM TEMPERATURE

by

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Based on the laser flash method, the selection of experimental parameters on the accuracy of measurement results for measuring thermal diffusivity is investigated in this paper. High purity graphite is employed as the experimental material. Three experimental parameters are taken into consideration, including specimen thickness, laser pulse power, and laser pulse width. Firstly, the principle of the laser flash method is introduced. Then, a numerical simulation model is established and independence tests are performed. In order to investigate the impact of different experimental parameters to the precision of the measurement results using the laser flash method, six thicknesses of the specimens, five laser pulse powers, and five laser pulse widths are selected for numerical simulation and LFA 427 measurement experiments. Finally, an orthogonal design method with three-factor and three-level is constructed to investigate the influence degree of these three factors on the measurement results. It is found that the laser pulse width has the most significant influence, while the laser pulse power has the least impact. For high purity graphite specimens, it is required to choose a thicker specimen, a lower laser pulse power, and a smaller laser pulse width to ensure better measurement accuracy.

Key words*: thermal diffusivity, laser flash method, experimental parameters, influence degree analysis*

Introduction

Thermal diffusivity is a crucial thermophysical property in the thermal design of materials. It presents the ability of materials to reach uniform temperatures during heating or cooling. In other words, it reflects the physical quantity of the temperature change rate in the heat conduction process. The measurement methods of thermal diffusivity include the laser flash method [1, 2], the AC calorimetric method [3], the transient plane source method [4, 5], and the photoacoustic method [6, 7]. Since the laser flash method is the standard method for the measurement of thermal diffusivity, it has been widely used in commercial apparatuses, such as NETZSCH, LINSEIS, and TA Instruments [8]. Besides, the laser flash method has the advantages of high measurement speed, small specimen size, and Wide range of temperature measurement.

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In recent years, based on the principle of laser flash method presented by Parker *et al.* [9], scholars have carried out various researches to improve the accuracy of measurement. In terms of measurement method improvement, Carr *et al.* [10, 11] developed a new formula to calculate thermal diffusivity under adiabatic conditions, which can reach a better accuracy. Pavlov *et al.* [12] developed a new inverse method to evaluate thermophysical properties. Based on the comparison of experimental results and literature data, it can be concluded that accurate results can be obtained by using this method. Ferrarini *et al.* [13] investigated a new control and data acquisition system, which can give multiple shots averaging results based on the laser flash method to improve the signal-to-noise ratio of the measurement. In the research field of influencing factors of laser flash method, Philipp *et al.* [14] proposed a numerical model of laser flash method to quantify the influence of experimental conditions on the measurement accuracy, which confirmed the advantages of numerical simulation in the research of influencing factors of laser flash method. Akoshima *et al.* [15] mentioned that the experimental conditions of laser flash method, such as the pulse energy, would affect the measurement results of thermal diffusivity. At the same time, they verified that the extrapolation method is effective for measuring thermal diffusivity of specimens with different thicknesses.

The current research shows that the experimental parameters of laser flash method have a significant impact on the accuracy of the measurement results. However, there is still a lack of research in this area. Moreover, compared with the actual experiment, the numerical simulation experiment can set the experimental parameters more conveniently, thus enabling the study of the influence of the experimental parameters on the measurement results. Therefore, in this paper, the influence of different experimental parameters on the accuracy of the flash method measurement results is studied in order to optimize the selection of the measurement experimental parameters. The experimental parameters include the specimen thickness, the laser pulse intensity and the laser pulse width. The influence degree of each factor is evaluated by orthogonal design method, which provides guidance for the selection of test parameters.

Experimental part

Since high purity graphite has high physical and chemical stability at the protective atmosphere, it is selected for this research. The experimental part is firstly used to determine the thermophysical parameters of high purity graphite for subsequent simulation studies.

The size of high purity graphite specimen used in the simulation validation is 12.7 mm in diameter and 3 mm in thickness, which is prepared by Shandong Non-Metallic Materials Institute. The thermal diffusivity of graphite is performed by LFA 427. This instrument is manufactured by NETZSCH and realizes measurements based on the principle of the laser flash method. During the measurement, helium is passed through the chamber to create a protective atmosphere. The pulsed laser flash generated by Nd:YAG laser system heats the bottom surface of the specimen. Then, an IR-detector is used to measuring the temperature response on the top surface of the specimen. The specimen holder is also made of graphite and supports the specimen through three small contacts to minimize thermal contact. During the measurement, the laser voltage is 600 V, while the laser pulse width is 0.2 ms. The method of baseline correction is linear, and the method of pulse-length correction is numerical. Then, in order to give the correct results, the fitting and model method for evaluation is Cape and Lehman.

Therefore, the thermal diffusivity, α, of high purity graphite with a diameter of 12.7 mm and a thickness of 3 mm is 107.137 mm^2/s. This measurement data is used to compare with the calculated results in the verification experiment.

Numerical part

Numerical model

The measurement theory of the laser flash method was initially presented by Parker *et al.* [9]. The schematic diagram is shown in fig. 1, in which the IR radiometer measures the temperature of the back surface of the specimen by a non-contact method.

When the front surface of the specimen is heated by a uniform laser pulse, its temperature rises rapidly. Then, the heat is transferred 1-D in the specimen. The temperature on the back side of the specimen varies with time. The half-temperature rise time is obtained from the temperature change on the back side of the specimen, which is used to calculate the thermal diffusivity of the material at the corresponding temperature.

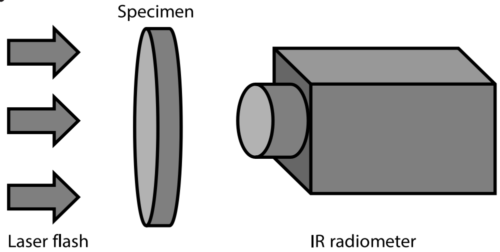

Figure 1. The schematic diagram of the laser flash method

Besides, the following assumptions need to be satisfied for the laser flash method. The heat transfer in the specimen is 1-D from the front surface to the back surface of the specimen, which means that there is no crosswise transfer of heat flow within the sample. The specimen is adiabatic and the external environment has no influence on the measurement. The specimen has good homogeneity. The front surface of the specimen is uniformly heated by laser and the laser pulse width is short enough. Therefore, according to the heat transfer theory, the heat balance equation in the specimen:

$$\nabla\left(\lambda\nabla T\right)=\rho C_p\frac{\partial T}{\partial t} \tag{1}$$

where T is the temperature of the specimen. Since the laser pulse width is short enough, the temperature rise generated by the specimen is small. It can be approximated that the thermal conductivity of the specimen remains constant during the measurement. Then, the eq. (1) is simplified and expressed:

$$\nabla^2 T=\frac{C_p\rho}{\lambda}\frac{\partial T}{\partial t}=\frac{1}{\alpha}\frac{\partial T}{\partial t} \tag{2}$$

Based on this, the rear surface of the specimen will produce a temperature response that varies with time, as shown in eq. (3), and the thermal diffusivity of the specimen can be calculated in eq. (4):

$$T\left(L,t\right)=\frac{Q}{\rho C_p d}\left[1+2\sum_{n=1}^{\infty}\left(-1\right)^n\exp\left(\frac{-n^2\pi^2}{d^2}\alpha t\right)\right] \tag{3}$$

$$\alpha=0.1388\frac{d^2}{t_{50}} \tag{4}$$

where Q is the laser pulse energy that the specimen absorbs, d – the thickness of the specimen, and t_{50} – the time corresponding to half temperature rise on the rear surface of the specimen.

During the numerical simulation, it is necessary to set appropriate laser parameters on the heating boundary condition. The laser pulse is a Gaussian heat source, and the expression is as follows [16], in which q is the heat flux distribution function, p refers to the laser power, R refers to the radius of the laser spot which should be larger than the radius of the specimen:

$$q = \frac{3p}{\pi R^2} \exp\left[\frac{-3\left(X^2 + Y^2\right)}{R^2}\right] \tag{5}$$

Verification of numerical model

In the ANSYS workbench simulation experiments, three thermophysical parameters including density, ρ, specific heat, C_p, and thermal conductivity, λ, need to be set. The density and specific heat of high purity graphite at room temperature are obtained from experimental measurements. The thermal conductivity of the sample is calculated by eq. (6), in which the thermal diffusivity, α, is measured by LFA 427:

$$\lambda = \alpha\rho C_p \tag{6}$$

Table 1 gives the thermal property parameters of high purity graphite set in the simulation at room temperature. Based on these thermal property parameters, a simulation model is established to carry out simulation experiments.

Table 1. Thermophysical properties of high purity graphite at room temperature

Parameters	Value
Density (set in the simulation)	1840 kg/m^3
Thermal conductivity (set in the simulation)	141.387 W/m°C
Specific heat (set in the simulation)	717.22 J/kg°C
Thermal diffusivity (used for comparison with simulation results)	107.137 mm^2/s

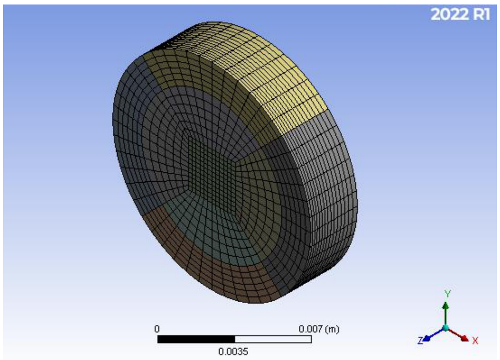

Figure 2. The meshing result of the specimen

The model of the numerical simulation is established by using ANSYS. The thickness of the specimen for verification is 3 mm, while the diameter is 12.7 mm. In the ANSYS simulation analysis, a smaller grid size and a shorter time step will make the simulation results more accurate, but the calculation time will be longer. Conversely, a larger grid size and a longer time step will result in lower simulation accuracy but shorter computation time [17]. In order to improve the simulation accuracy, the mesh layer is properly encrypted in the vicinity of the laser-heated surface. The meshing result is shown in fig. 2, which has 10800 grid cells with 46519 nodes.

The initial temperature of the specimen is 25 °C, and the periphery of the specimen is adiabatic. In the verification simulation, the spot diameter of the laser pulse is set to 13 mm, and the laser pulse width is set to 0.2 ms. The simulation experiment time is 0.1 second to ensure the specimen can reach a steady-state. In the simulation, the time step of the laser flash phase is set to 0.002 ms, while the time step of the heat transfer phase is 0.02 ms to improve the speed of numerical calculation. Figure 3 depicts the temperature distribution of the specimen at 0.02 ms, 0.2 ms, 0.01 second, and 0.1 second.

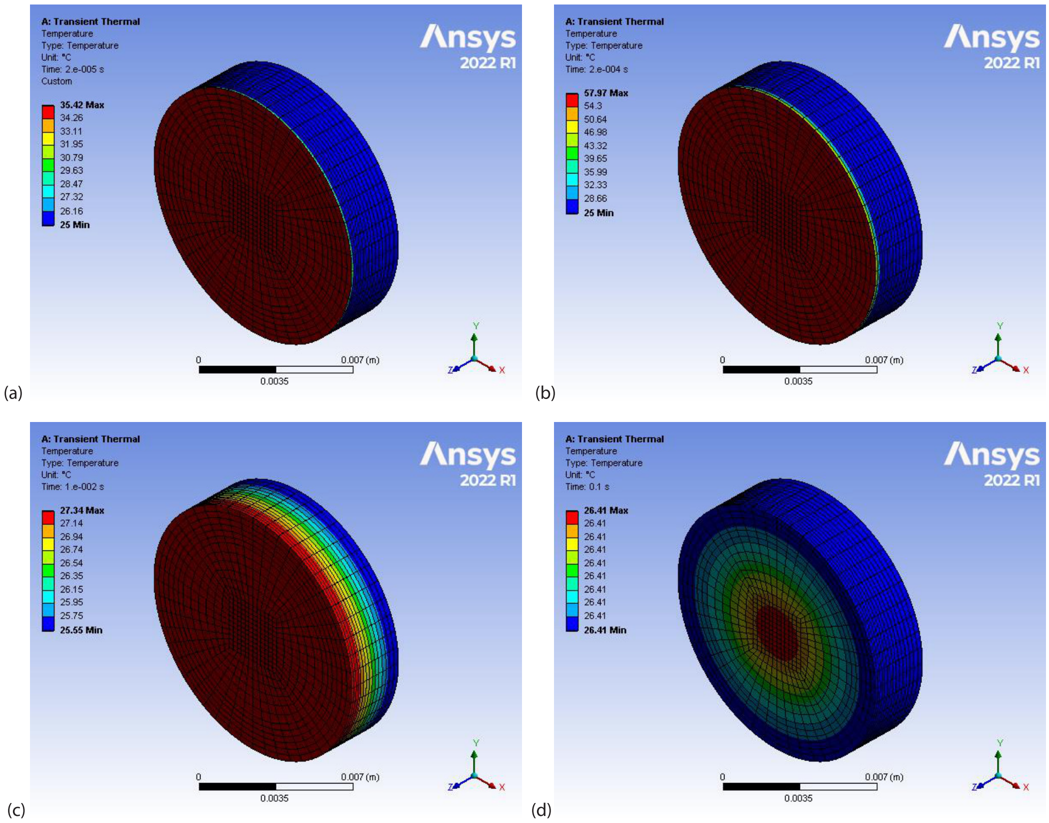

Figure 3. The temperature distribution of the specimen at 0.02 ms, 0.2 ms, 0.01 second, and 0.1 second

At 0.02 ms, the laser pulse starts. There is a very thin layer on the front surface of the specimen absorbs the laser energy. When the time comes to 0.2 ms, the laser pulse just stops. The temperature of the front surface of the specimen reaches a maximum value of 57.97 °C. As the heat from the front surface of the sample is gradually transferred to the back surface, the temperature of the specimen gradually converges to a steady-state temperature of 26.41 °C.

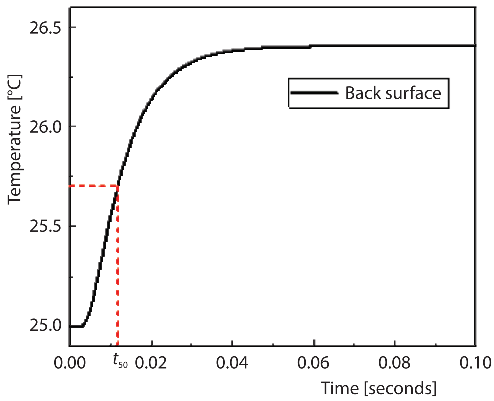

Figure 4. The temperature curve of the specimen backside varies with time

Figure 4 illustrates the temperature curve of the specimen backside varies with time. The thermal diffusivity can be calculated as 106.672 mm^2/s by using eq. (4). Since the thermal diffusivity calculated by the setting thermophysical parameters is 107.137 mm^2/s, the relative error is 0.434%. The source of the error may be the relative long laser pulse in comparison the half rise-time, the inherent error in the flash method principle, or the effect of the grid and time step on the simulation results. Consequently, this numerical model can realize the simulation for measuring thermal diffusivity of high purity graphite by laser flash method at room temperature, which provides a model foundation for subsequent investigations.

Results and discussions

Based on the numerical simulation model, the influence of the experimental parameters of the laser flash method on the measurement results of thermal diffusivity is analyzed by controlling variables. In this part, the influences of specimen thickness, laser pulse power, and laser pulse width on the measurement results are considered based on numerical simulation, and the correlations exhibited by the simulation are verified by experiments. Then, the degree of each influencing factor on the measurement results is discussed to provide theoretical guidance for the selection of experimental parameters of the laser flash method.

In this part, the thermophysical parameters of the high purity graphite specimen in the simulation vary with temperature. In order to obtain the simulation setting parameters at higher temperature, LFA 427 is used to measure thermal diffusivity of the 3 mm specimen at 100 °C, and the measurement results is 79.538 mm^2/s. Therefore, a linear relationship for the thermal diffusivity from 25-100 °C is assumed in the simulation account for the effect of the temperature variations on the simulation results.

Influence of specimen thickness

In order to study the influence of specimen thickness on the measurement results, six specimens with thickness of 1-6 mm are selected for investigation.

Simulation of the specimen thickness effect

Firstly, the simulation of the laser flash method of six thicknesses is carried out. In the simulation, the thermophysical parameters of the specimen vary with temperature. The boundary conditions are set the laser pulse power is 125 kW, the laser pulse width is 0.2 ms, the natural-convection heat transfer coefficient is 5 W/m^2, the emissivity of the specimen is 0.9, and the ambient temperature is 25 °C. Table 2 shows the numerical simulation results of the laser flash method under different thicknesses.

Table 2. The numerical simulation results for specimen with different thicknesses

Thickness of the specimen [mm]	Maximum temperature of the back surface temperature rise [°C]	α [mm^{2}s^{-1}]
1	29.18	97.969
2	27.10	105.319
3	26.40	106.063
4	26.05	106.496
5	25.84	106.991
6	25.70	107.037

It can be seen that the temperature rise on the back surface of the specimen decreases significantly with the increase of the specimen thickness. Since the thermal diffusivity of the specimen decreases with increasing temperature, thicker specimen may provide more accurate results. However, the calculation result of the specimen with 1 mm in thickness is produced significant differences from the results for the other thickness. The reason may be that the laser pulse width is not short enough relative to the half temperature rise time on the back surface of the specimen. For the specimen with 3 mm, the compared to the model validation experiments in section *Verification of numerical model*, the thermophysical parameters of this part of the sample vary with temperature, which leads to a decrease in the results due to an increase in the temperature of the specimen during the simulation.

Comparison of patterns between experimental and numerical results

The LFA 427 is employed to determine the thermal diffusivity of specimens with these six thicknesses. The laser pulse voltage and the laser pulse width are configured to 600 V and 0.2 ms, respectively. The measurement uncertainty of LFA 427 is 3% [18]. Figure 5 shows the experimental measurement results and the numerical simulation results for different thicknesses.

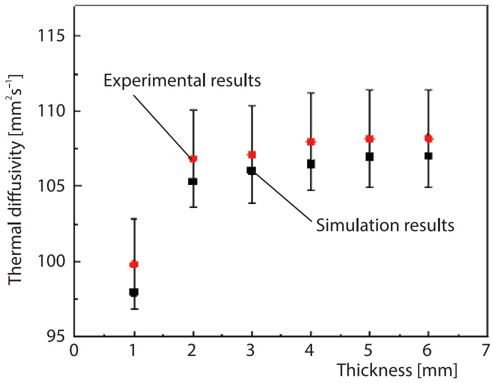

Figure 5. The experimental and simulation results of thermal diffusivity for different thicknesses

Since the experimental data are set as the initial thermophysical parameters in the simulation, the numerical results will be lower than the experiment results after adding the temperature-dependent thermophysical parameters. Besides, the measurement result of the specimen with a thickness of 1 mm is significantly lower than those of other thicknesses. The reason for this phenomenon may be that the laser energy breaks down the specimen or the heat transfer time in the specimen is too short. Moreover, the thermal diffusivity measurement results gradually approached a constant value with increasing thickness. Therefore, the selection of the specimen thickness needs to comprehensively consider the influence of the factors such as measurement accuracy, specimen fabrication difficulty, and cost.

Influence of laser pulse power

Simulation of the laser pulse power effect

In investigating of the influence of the laser pulse power, five laser pulse powers of 125 kW, 150 kW, 175 kW, 200 kW, and 225 kW are selected for numerical simulation experiments. The thermophysical properties of the specimen still vary with temperature. The thickness of the specimen is 3 mm. The natural-convection heat transfer coefficient is 5 W/m^2, while the emissivity of the specimen is 0.9. The laser pulse width is configured to 0.2 ms. Table 3 depicts the numerical simulation results of thermal diffusivity under different laser pulse powers, and fig. 6(a) gives the graph of the numerical results.

Table 3. The numerical simulation results of thermal diffusivity under different laser pulse powers

Laser pulse power [kW]	Maximum temperature of the back surface temperature rise [°C]	α [mm^{2}s^{-1}]
125	26.40	106.063
150	26.68	105.840
175	26.96	105.701
200	27.24	105.562
225	27.51	105.423

It is found that the maximum temperature of the back surface temperature rise increases while the laser pulse power increases. Moreover, since the thermal diffusivity of the specimen decreases with the increasing temperature, the results of thermal diffusivity calculated by the simulation gradually drop as the increasing flash laser power.

Comparison of patterns between experimental and numerical results

For further comparison of the influence on flash laser energy on thermal diffusivity measurement, the experimental researches are undertaken by LFA 427. The laser voltages of LFA 427 are set to 600 V, 640 V, 680 V, 720 V, and 750 V, respectively. The laser pulse width is setting to 0.2 ms. The experimental results are depicted in fig. 6(b).

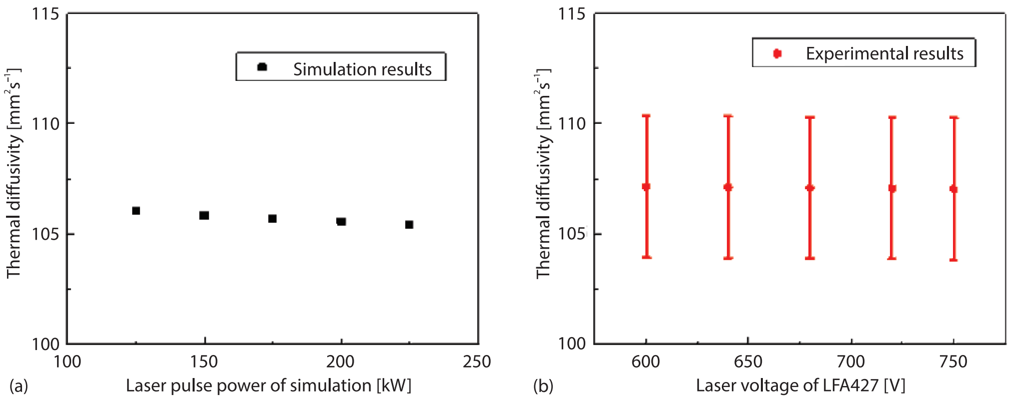

Figure 6. Comparison between the simulation the experimental results with different laser pulse powers

It could be observed that higher laser pulse voltage will lead to lower measurement results. This conclusion matches that obtained by numerical simulation. Since the laser pulse power can bring greater measurement error, this parameter cannot be set too high in the experiment. However, the laser pulse power needs to deliver enough power to the specimen so that the infrared detector can detect the energy change on the back surface of the specimen. In addition, from the data in fig. 6, it is found that the measurement results in both simulation and experiment show a linear relationship. Through a linear fitting, the results of thermal diffusivity at zero laser pulse energy could be obtained, which are 106.808 mm^2/s and 107.517 mm^2/s for simulation and experiment, respectively. Therefore, in the laser flash method, a smaller laser pulse voltage or multiple measurements with linear fitting could be selected to achieve an accurate measurement result.

Influence of laser pulse width

Simulation of the laser pulse width effect

Except for the specimen thickness and the laser pulse power, the laser pulse width may also affect the measurement results of the laser flash method. Four laser pulse widths are chosen for numerical simulations, including 0.2 ms, 0.4 ms, 0.6 ms, 0.8 ms, and 1.0 ms. Other boundary conditions are the laser pulse power is 125 kW, the natural-convection heat transfer coefficient is 5 W/m^2, and the emissivity of the specimen is 0.9. The thermophysical parameters of the specimen depend on temperature. Table 4 gives the numerical calculation results of thermal diffusivity under different laser pulse widths.

Table 4. The numerical calculation results of thermal diffusivity under different laser pulse widths

Laser pulse width [ms]	Maximum temperature of the back surface temperature rise [°C]	α [mm^{2}s^{-1}]
0.2	26.40	106.063
0.4	27.80	104.430
0.6	29.18	102.946
0.8	30.56	101.520
1.0	31.93	100.147

It could be concluded from that under the condition of the constant laser pulse power, the maximum temperature of the back surface temperature rise of the specimen also increases with the increase of the laser pulse width. The pattern demonstrated is consistent with that in section *Simulation of the laser pulse power effect*, that is, both raising the laser pulse voltage and enhancing the laser pulse width are able to increasing the laser pulse energy, which consequently affects the temperature of the specimen.

Comparison of patterns between experimental and numerical results

The measurement experiments of LFA 427 are utilized to verify the variation tendency of the simulation results. The experiments are also carried out at room temperature. During the investigation, the laser pulse voltage is set to 600 V. Then, the laser pulse widths of 0.2 ms, 0.4 ms, 0.6 ms, 0.8 ms, and 1.0 ms are considered, respectively, to investigate their influences on the measurement results of thermal diffusivity. Figure 7 shows the comparison between the simulation results and the experimental results under different laser pulse widths, in which fig. 7(a) depicts the simulation results while fig. 7(b) gives the experimental results.

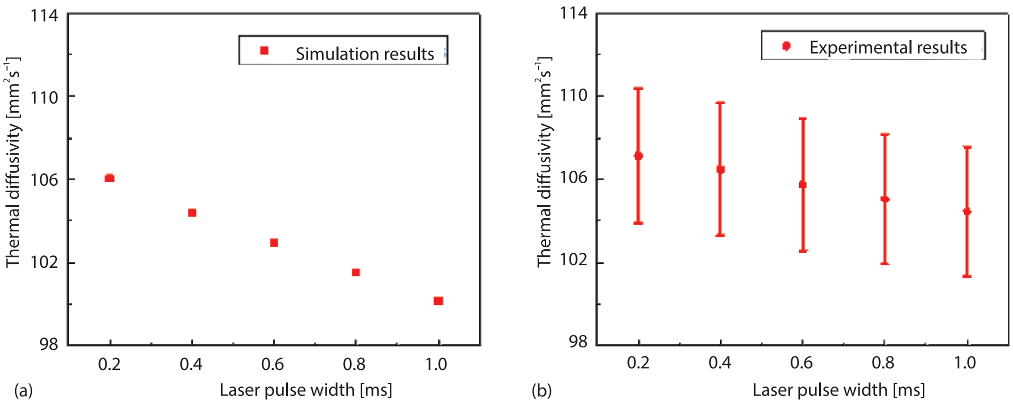

Figure 7. Comparison between the simulation and experimental results under different laser pulse widths

It is obvious that there are differences between the experimental value and the simulation value, especially since the laser pulse widths are set to 0.8 ms and 1.0 ms. The possible reason for this phenomenon is that the laser power configured in the simulation is higher than the laser power generated by the laser pulse voltage in the experiment, which leads to a higher temperature on the back surface of the specimen in the simulation and produces a more significant deviation. However, after linear fitting these two groups of data generated by experiments and simulations separately, the intercepts of the simulation results and the experimental results are

107.444 mm^2/s and 107.811 mm^2/s, respectively. It means that the thermal diffusivity obtained by the simulation and the experiment are close when the laser pulse width is 0 ms. Furthermore, it can be found that a short laser pulse width is conducive to improving the accuracy of thermal diffusivity measurements. Yet, it should be considered that a minimal laser pulse width is not conducive to the detection of the back surface temperature of the specimen. Therefore, a comprehensive consideration is required to select the test parameters.

Analysis of the influence degree of each factor

Finally, an orthogonal design method is employed to investigate the influence degree of three factors involved in the laser flash method on the measurement accuracy of thermal diffusivity. The three factors include specimen thickness, laser pulse power, and laser pulse width. Three levels of each factor are selected to carry out the orthogonal test. Thus, the three factors and three levels of the orthogonal design method are shown in tab. 5.

Table 5. Three factors and three levels of the orthogonal design method

Factors	Specimen thickness, *A*	Laser pulse power, *B*	Laser pulse width, *C*
Level 1	5 mm	225 kW	0.2 ms
Level 2	4 mm	175 kW	0.4 ms
Level 3	3 mm	125 kW	0.6 ms

According to the principle of permutation and combination, twenty-seven experiments are required to perform complete permutation experiments with the data in tab. 5. The orthogonal design method can analyze typical data using the idea of balanced distribution, which can effectively reduce the number of experimental tests. Based on the three factors and three levels indicated in tab. 5, an orthogonal design method is conducted using the standard $L_9(3^4)$ orthogonal test table shown in tab. 6. Besides, the calculated results of the thermal diffusivity for each case by simulation are also listed in this table.

Table 6. Experimental arrangements and results

Case	*A* [mm]	*B* [kW]	*C* [ms]	*D*	Thermal diffusivity [mm^{2}s^{-1}]
1	5	225	0.2	1	106.356
2	5	175	0.4	2	105.617
3	5	125	0.6	3	105.225
4	4	225	0.4	3	104.654
5	4	175	0.6	1	103.888
6	4	125	0.2	2	106.496
7	3	225	0.6	2	101.478
8	3	175	0.2	3	105.701
9	3	125	0.4	1	104.430

The extreme difference analysis is employed to analyze the degree of the influence of *A*, *B*, and *C* on the thermal diffusivity measurements. Table 7 depicts the extreme difference analysis results, in which K_i represents the mean value of each level for each factor and *R* refers to the extreme difference value.

Table 7. The extreme difference analysis results

Factors	*A*	*B*	*C*
K_1	105.733 mm²/s	104.163 mm²/s	106.184 mm²/s
K_2	105.013 mm²/s	105.069 mm²/s	104.900 mm²/s
K_3	103.870 mm²/s	105.384 mm²/s	103.530 mm²/s
R	1.863 mm²/s	1.221 mm²/s	2.654 mm²/s

According to the extreme difference data in tab. 7, it can be found that the laser pulse power has a minimum extreme difference value of 1.221 mm²/s, and the laser pulse width has a maximum extreme difference value of 2.654 mm²/s. Therefore, within the test range, the laser pulse width has the most significant influence on the thermal diffusivity measurement. In contrast, the laser pulse power gives the least impact on the thermal diffusivity measurement. This conclusion is the same as that of the previously mentioned trends for each factor, which demonstrates the reliability of the orthogonal design method.

Conclusion

This paper investigates the influence of the selection of experimental parameters on measurement results based on laser flash method for measuring thermal diffusivity of graphite, and the results are only valid for well conducting materials at room temperature. The evaluated experimental parameters include specimen thickness, laser pulse power, and laser pulse width. The results of the thermal diffusivity are compared utilizing simulations and experiments, respectively. Based on the orthogonal experimental design of the simulation results, it could be concluded that the laser pulse width is more likely to influence the measurement accuracy of the laser flash method. In comparison, the laser pulse power has the least influence on measurement accuracy. Therefore, for measuring thermal diffusivity of high purity graphite at room temperature, it is necessary to select thicker samples and set a lower laser pulse power and a smaller laser pulse width to achieve a more accurate result.

Acknowledgment

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Nomenclature

C_p – specific heat, [Jkg^{-1}°C^{-1}]
d – thickness, [m]
T – temperature, [°C]
p – laser power, [Wm^{-2}]
Q – laser pulse energy, [Jm^{-2}]
q – heat flux, [Wm^{-2}]
R – radius of the laser spot, [m]
t – time, [s]

Greek symbols

α – thermal diffusivity, [Wm^{-2}]
λ – thermal conductivity, [Wm^{-1}°C^{-1}]
ρ – density, [kgm^{-3}]

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