

A Contact Method for Measuring Thermal Transport Properties of Solid Materials Using a Stamp Sensor

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A Contact Method for Measuring Thermal Transport Properties of Solid Materials Using a Stamp Sensor

by

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**A thesis submitted in partial fulfillment of the requirements
for the degree of Doctor of Engineering
in the Department of Mechanical Engineering**

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NOMENCLATURES

i	: Number of iteration
C_p	: Specific Heat at Constant Pressure (J/kg.K)
E	: Voltage (V)
I	: Current (A)
Q	: Heating power (W)
$SD_{\Delta T}$	: Standard deviation of difference between the calculated and measured temperatures (K)
T	: Temperature (K)
T_{cal}	: Calculated / theoretical temperature (K)
T_{exp}	: Measured temperature (K)
t	: Time (s)
t_{exp}	: Measured Time (s)
t_{cal}	: Calculated Time (s)
t_0	: Time at the start of heating (s)
α	: Thermal diffusivity (m ² /s)
Δt	: Difference in time (s)
δ	: Thickness of gel layer (m)
λ	: Thermal conductivity (W/m.K)
ρ	: Mass density (kg/m ³)

SUBSCRIPTS

cal	: calculated or theoretically obtained value
exp	: experiment
ref	: reference

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- [1] Hadi, S., Nishitani, M., Wijayanta, A.T., Kurata, K., and Takamatsu, H., Measurement of Thermal Conductivity and Thermal Diffusivity of Solid Materials Using a Novel Stamp Sensor: A Feasibility Study with Numerical Analysis, Journal of Thermal Science and Technology, JSME 7 (4) (2012) 536-548.
- [2] Hadi, S., Nishitani, M., Fukunaga, T., Kurata, K., and Takamatsu, H., A Stamp Sensor for Measurement of Thermal Conductivity and Thermal Diffusivity of Solid Materials, Proceeding of The Third International Forum on Heat Transfer, Nagasaki, Nov 2012, 089.
- [3] Hadi, S., Nishitani, M., Wijayanta, A.T., Kurata, K., and Takamatsu, H., Contact Measurement of Thermal Conductivity and Thermal Diffusivity of Solid Materials: Experimental Validation of Feasibility with a Prototype Sensor, International Journal of Heat and Mass Transfer (2013) Under Review.

1.1 Measurement of Thermal Transport Properties

There are a lot of potential needs of knowing thermal transport properties of soft materials including biological materials in medicine and food product engineering. In a cryosurgery or a tissue ablation by laser heating, thermal transport properties of tissues are important for designing protocols for successful treatments [1-4]. In the food product engineering, the thermal transport properties is required for predicting, designing, and optimizing every process in the food processing such as freezing, thawing, cooking and drying. Proper treatment will make processes optimized and produce high quality foods. Although materials treated in the food engineering are inhomogeneous or anisotropic in many cases, estimates of thermal transport properties would help designing production facilities [5-7].

A number of methods are available for measuring thermal transport properties of solid materials. They are categorized in two methods: a steady-state method and a transient method. The steady-state measurement technique is the simplest in principle, in which the thermal conductivity is determined by measuring the temperature drop and the heat flux in a sample. Temperature gradients are generated by heating one side of the sample with a heater and cooling at the other side (Fig. 1.1). Processes of heating and cooling at constant rates are needed with recording thermocouples outputs. Then recorded output is processed using simple mathematical model. High precision of the results reflects the high degree of control of experimental variables [8-10].

In the steady state method, there is a problem associated with the lateral heat loss that does not realize the directional heat flow. Therefore some complex apparatus must be incorporated to eliminate heat loss. The heat flux should be maintained at a constant value for long time throughout the measurements. The problem to reduce the

thermal contact resistance between samples and the heating or cooling substrate is another issue, and it could be achieved by pressing them tightly. However it does not work for soft materials.

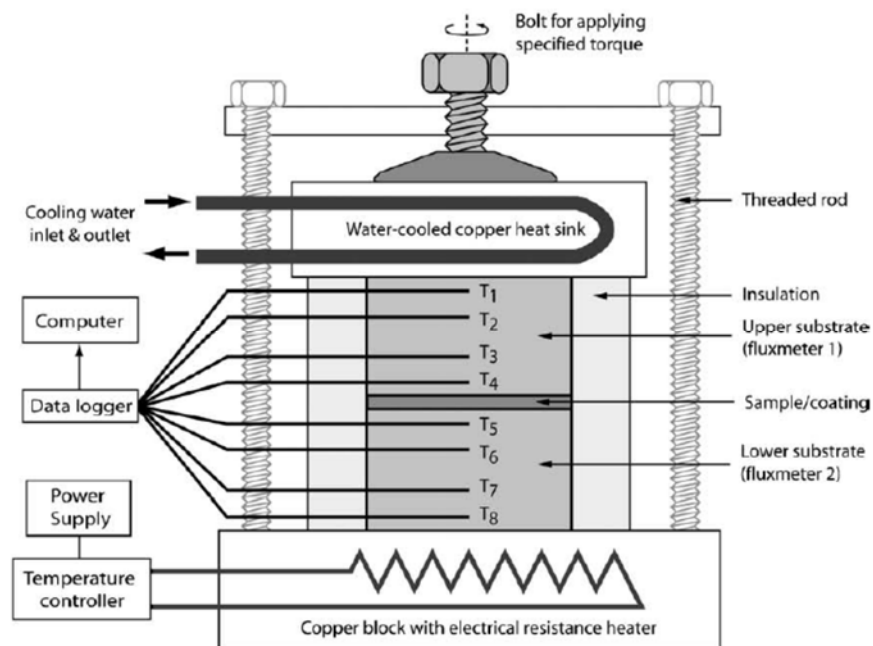


Fig. 1.1 Steady state method measurement [8]

The most popular method categorized in the transient method is the laser flash method (Fig. 1.2). In the method the thermal diffusivity is determined from the transient temperature rise at one side of a sample with pulse heating by laser irradiation at the other side [11-14]. Advantages of this method over the steady-state method are that the measurement is finished in a short time and does not require complicated mechanism to avoid heat losses. It has been used to measure a variety of solid materials: stainless steel, ceramic [15, 18], copper [16], aluminum [17], SiC_f composites [19], alumina, silicon [20], and glass materials [21, 22]. With a special treatment, it has also been applied to measure molten materials such as Li₂B₄O₇ and

KNbO₃ [23, 24]. In addition, thin solid materials between millimeters and micrometers in thickness have been measured [16, 17] by this method.

Since laser flash method utilizes laser heating, the material should be optically nontransparent to the laser light, and have high emissivity for absorbing the light at the surface and for measuring the surface temperature with infrared detectors [13]. In addition, preparation of a specimen with a precise thickness is important. For these reason, the laser flash method is not suitable for a gel or soft material including biomaterials.

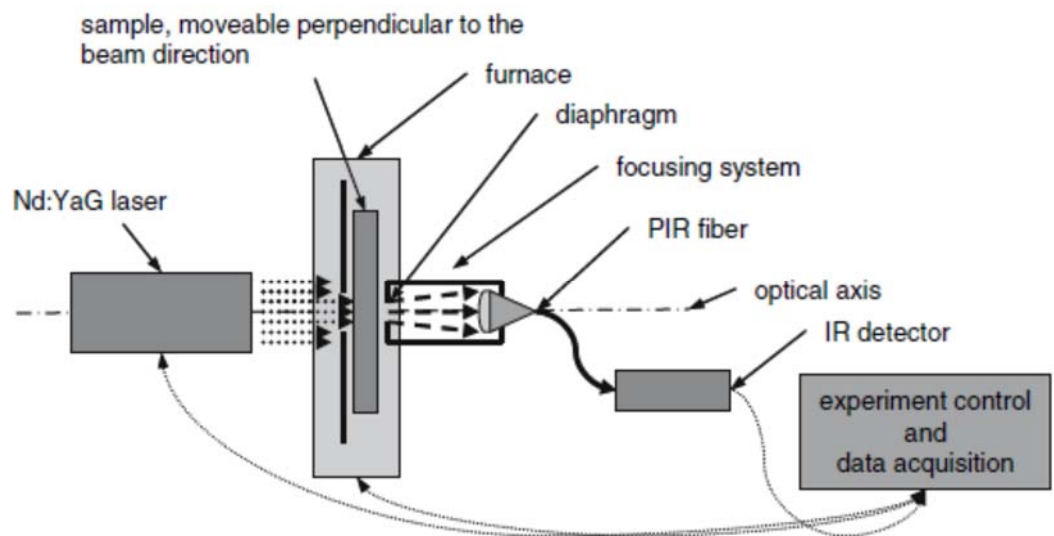


Fig. 1.2 Laser flash method [11]

The other popular method for measuring thermal transport properties of solids, gels and liquids easier than the laser flash method is a transient plane source technique that uses a thin heater that is sandwiched between two specimens (Fig. 1.3). The method is called a hot-disk or a hot-strip method depending on the shape of the heater. The temperature of the heater, which also works as a sensor, is determined by measuring the electrical resistance of the heater [25-32]. This method has been applied to measure a variety of materials such as insulation materials [33],

polyethylene foams [34], fused silica, carbon steels, halide crystal [35], agricultural materials [6], non-metals [36], coated papers [37], and aluminum foams [38]. While the transient plane source technique is categorized in a non-destructive measurement technique, it does require preparing two specimens of the same sizes.

The most important issue in this method is to minimize a thermal contact resistance between the sensor and the samples. This is generally solved by strongly pressing samples, polishing surface of samples [38], and putting a liquid between samples [25]. The transient hot-wire technique [39], instead of the hot-disk technique, has been used as well. The difference is that the hot wire assumes an infinitely long line source in a specimen as an ideal situation while the hot disk supposes a plane heat source.

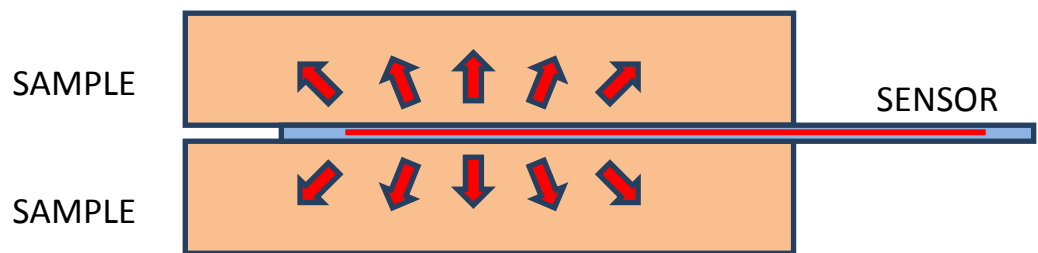


Fig. 1.3 Transient plane method

Another convenient method that is used for in-situ measurement is a point contact method [40-42]. Preparing a specimen with a fixed shape is not needed for this method. In this method a small heated spherical sensor is pressed to make a point contact with a specimen. Using a welded thermocouple as a small bead, the temperature response of the probe is observed for several seconds after the contact. This method has been used for the measurement of SUS304, aluminum, copper, acrylic resin, silicon rubber, and teflon. To reduce and control the effect of thermal contact resistance between the sensor and specimens the sensor is pressed at a

constant pressure to the specimen using a spring. However it is not appropriate for measuring soft materials including biological materials. This is because pressing a bead sensor on a soft sample would alter the contact area in the point contact-probe method, although the method assumes point contact between the sensor and the sample.

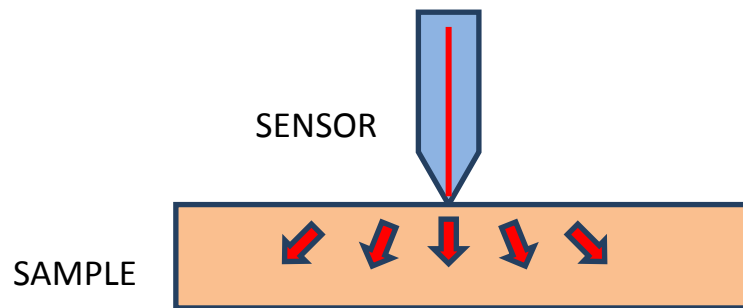


Fig. 1.4 Point contact method

The method that has been developed only for measuring thermal transport properties of biomaterials utilized a self-heated thermistor [43-45] (Fig. 1.5). A spherical bead thermistor that was used as both temperature probe and heat source was pierced in a sample, and heated with a d.c current to maintain its temperature constant. The thermal conductivity of materials was determined from the transient change of the input. Several types of materials have been measured: agar, water, glycerin, ethylene glycol, human blood [43], tissues [44], and gels [45]. Although this method was applied to tissues and biomaterials, it may involve an unknown effect from the contact between the bead and specimens. A gap could be formed at the interface during piercing process. For measurement of living tissue the effect of bleeding would not be evitable because this is an invasive method.

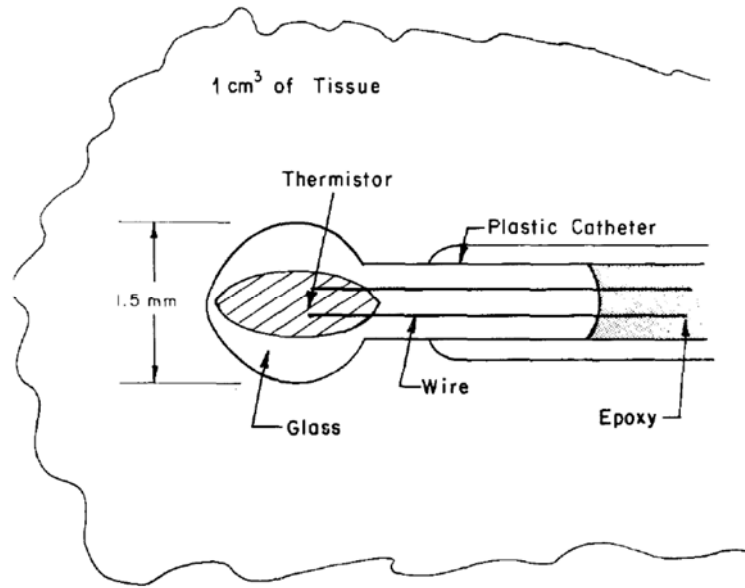


Fig. 1.5 Self-heated thermistor method

1.2 The Goal and Objectives

The methods mentioned above have been used for a variety of materials, while each method has advantages and limitations. However, none of these is appropriate for soft materials including biological materials except the invasive method that uses a bead sensor. Preparing a specimen of soft materials with precise dimension is difficult in the laser flash. Clamping soft specimens hard without deformation is difficult in the transient plane source method. Pressing a bead probe on a soft sample would alter the contact area in the point contact-probe method, although the method assumes point contact between the sensor and the sample. Even in the inserted bead technique, it may not completely avoid a problem resulted from the thermal contact resistance.

Hence the goal of this research is to develop a convenient method for measuring thermal transport properties of solids, particularly soft materials. The method proposed here uses a 'stamp sensor'. It is a sort of contact method that works for non-destructive in-situ measurement. The uniqueness of this method is to put a gel between a film heater/sensor and a sample to eliminate the thermal contact resistance.

In addition, a shallow cavity with given dimensions is prepared around the sensor for the gel.

This research proceeded in two steps. First, a numerical study was conducted to develop the principle of this method. The protocol to determining the thermal conductivity and the thermal diffusivity was checked using virtual experimental data that have been generated by adding an artificial scattering to a theoretical temperature change of the sensor. Then as the second step, the feasibility of the method was demonstrated with a prototype sensor by measuring several kinds of materials that have a wide range of thermal transport properties.

1.3 Outline of the Thesis

The thesis consists of five chapters.

Chapter 1 has introduced the background and the objective of this thesis.

Chapter 2 shows the feasibility study with a theoretical analysis. The conceptual design of the sensor, a model for theoretical analysis, the numerical solution and the protocol for determining thermal transport properties are presented. Based on the virtual experimental data, feasibility of the method is demonstrated.

Chapter 3 is prepared for showing fabrication of the sensor as well as the design of the experiment. The method to fabricate the stamp sensor, design of experimental apparatus, and the procedure of the experiment are explained. Method to determine the time at the start of heating, which is important in this method, is also discussed.

In chapter 4, the results of measuring thermal transport properties of different materials are summarized with some discussions.

Chapter 5 gives conclusions of the present study.

FEASIBILITY STUDY USING NUMERICAL ANALYSIS

2.1 Concept of Stamp Sensor and Measuring Method

The conceptual design of the proposed sensor is illustrated in Fig 2.1. A thin film heater that also works as a resistance thermometer is provided on the top of a flat surface as a contact sensor. When this sensor is used to measure thermal transport properties, it is pressed against a surface of a specimen with a gel in between to eliminate a contact thermal resistance. Then a transient temperature rise of the sensor is measured along with heating it by a direct current. Thermal transport properties are determined by theoretical analyses, which take into account the additional thermal resistance resulted from the gel layer. The point of the proposed sensor is to provide a shallow cavity of known dimensions for the gel to give its thickness, to be more precise, a preliminary assumption of the gel thickness in the theoretical model. The system is configured axially symmetric with a round heater at the bottom of a cylindrical device as shown in Fig. 2.1. The sensor was named the "stamp sensor" because of its supposed appearance and application.

The configuration and dimension of a supposed model, which will be fabricated as a prototype, are also shown in Fig. 2.1. A 40-nm thick platinum film heater 3 mm in diameter is deposited on a 150- μm thick glass substrate. The heater is coated by a 40-nm thick glass layer for purposes of insulation and protection. The shallow cavity for the gel is provided by a 50- μm thick rubber sheet with a round cutout attached on the glass surface. The glass substrate is held at its backside with a hollow cylinder.

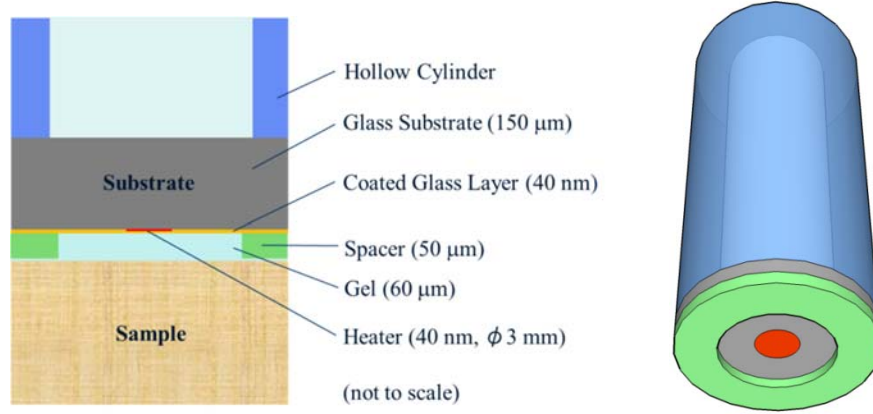


Fig. 2.1 Conceptual design and supposed dimension of a stamp sensor

After pressing the sensor against a specimen and waiting for the system to become thermal equilibrium, the sensor will be heated suddenly at a uniform and constant rate with measuring its temperature. The experiment is followed by comparison of the measured temperature with theoretical temperature rises obtained with assumed thermal conductivity and thermal diffusivity of the specimen as well as the thickness of the gel. These values, *i.e.* the thermal conductivity, the thermal diffusivity and the gel thickness, which would yield the minimum deviation from the measured temperature rise, are determined simultaneously by trial and error, which will be described later.

2.2 Physical Model

The physical model for the theoretical analysis is shown in Fig. 2.2. The system was defined by the cylindrical coordinate system with the origin at the center of the heater on the substrate surface. The heat conduction equation for each region is described by following equations:

[Substrate]

$$\rho_b C p_b \frac{\partial T}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left(\lambda_b r \frac{\partial T}{\partial r} \right) + \frac{\partial}{\partial z} \left(\lambda_b \frac{\partial T}{\partial z} \right) \quad (1)$$

[Sensor]

$$\rho_h C p_h \frac{\partial T}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left(\lambda_h r \frac{\partial T}{\partial r} \right) + \frac{\partial}{\partial z} \left(\lambda_h \frac{\partial T}{\partial z} \right) + \dot{q}_v \quad (2)$$

[Coated layer]

$$\rho_c C p_c \frac{\partial T}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left(\lambda_c r \frac{\partial T}{\partial r} \right) + \frac{\partial}{\partial z} \left(\lambda_c \frac{\partial T}{\partial z} \right) \quad (3)$$

[Gel]

$$\rho_g C p_g \frac{\partial T}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left(\lambda_g r \frac{\partial T}{\partial r} \right) + \frac{\partial}{\partial z} \left(\lambda_g \frac{\partial T}{\partial z} \right) \quad (4)$$

[Sample]

$$\rho_s C p_s \frac{\partial T}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left(\lambda_s r \frac{\partial T}{\partial r} \right) + \frac{\partial}{\partial z} \left(\lambda_s \frac{\partial T}{\partial z} \right) \quad (5)$$

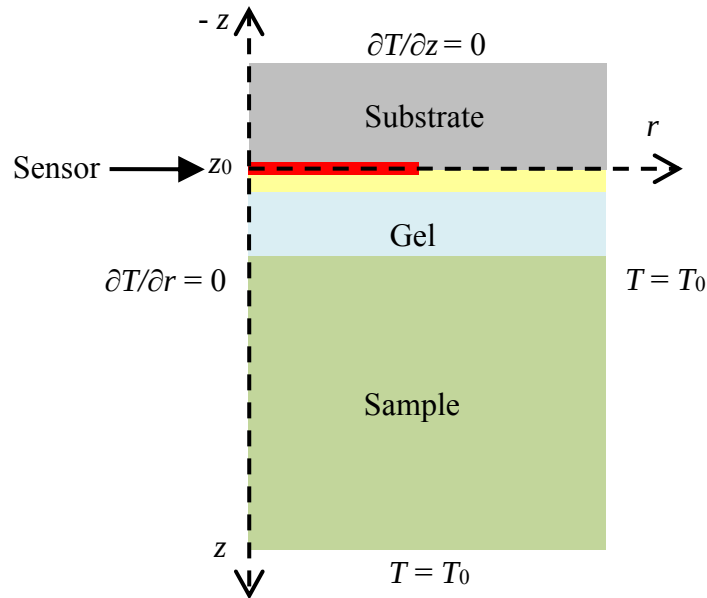


Fig. 2.2 Physical model and boundary conditions

Subscripts in the equations are; substrate b , sensor (heater) h , coated layer c , gel g , sample s . The rate of heat generation per unit volume $\dot{q}_v$ was incorporated only in

the equation for the heater, and was assumed to be uniform and constant. Physical properties of water was used for the gel layer. The initial condition is

$$T = T_0 \quad \text{at } t = 0 \quad (6)$$

and the boundary conditions are described as follows:

$$T = T_0 \quad \text{at } r \rightarrow \infty \text{ or } z \rightarrow \infty \quad (7)$$

$$\partial T / \partial r = 0 \quad \text{at } r = 0 \quad (8)$$

$$\partial T / \partial z = 0 \quad \text{at } z = -z_0 \quad (9)$$

The sensor side of the glass substrate, $z = -z_0$, that was exposed to the air inside the hollow cylinder was assumed to be adiabatic. The continuity of heat flux at the interfaces between different regions was also taken into account by:

$$\lambda_h \left. \frac{\partial T}{\partial z} \right|_h = \lambda_c \left. \frac{\partial T}{\partial z} \right|_c \quad \text{at } z = -\delta_h / 2 \quad (10)$$

$$\lambda_h \left. \frac{\partial T}{\partial z} \right|_h = \lambda_b \left. \frac{\partial T}{\partial z} \right|_b \quad \text{at } z = \delta_h / 2 \quad (11)$$

$$\lambda_b \left. \frac{\partial T}{\partial z} \right|_b = \lambda_g \left. \frac{\partial T}{\partial z} \right|_g \quad \text{at } z = \delta_h / 2 + \delta_b \quad (12)$$

$$\lambda_g \left. \frac{\partial T}{\partial z} \right|_g = \lambda_s \left. \frac{\partial T}{\partial z} \right|_s \quad \text{at } z = \delta_h / 2 + \delta_b + \delta \quad (13)$$

Numerical solution to Eq. (1) was obtained by a finite volume method with central difference for conduction terms and fully implicit formulation for unsteady terms. A schematic diagram of the grid is shown in Fig. 2.3. The boundary of the solution domain was defined at 0.2 mm from the sample surface in the z -directions and 15 mm in the radius. The solution domain was divided into 244×1000 meshes with variable sizes after examination of the effect of mesh size on the calculated result. Since the thickness of the sensor was very thin, the heater was covered by only one mesh in z -direction. The rest of 243 meshes in the z -direction consisted of 30 meshes in the substrate, 3 meshes in the coated layer, 10 meshes in the gel layer, and 200 meshes in

the sample. The size of the mesh increased with the z in the sample, while it was uniform in the other layer. The uniform meshes of the same size were used in the r -directions for all layers. Among 1000 meshes in total, 100 meshes were allocated to the radius of the heater. The computer program by FORTRAN is listed in the Appendix A.

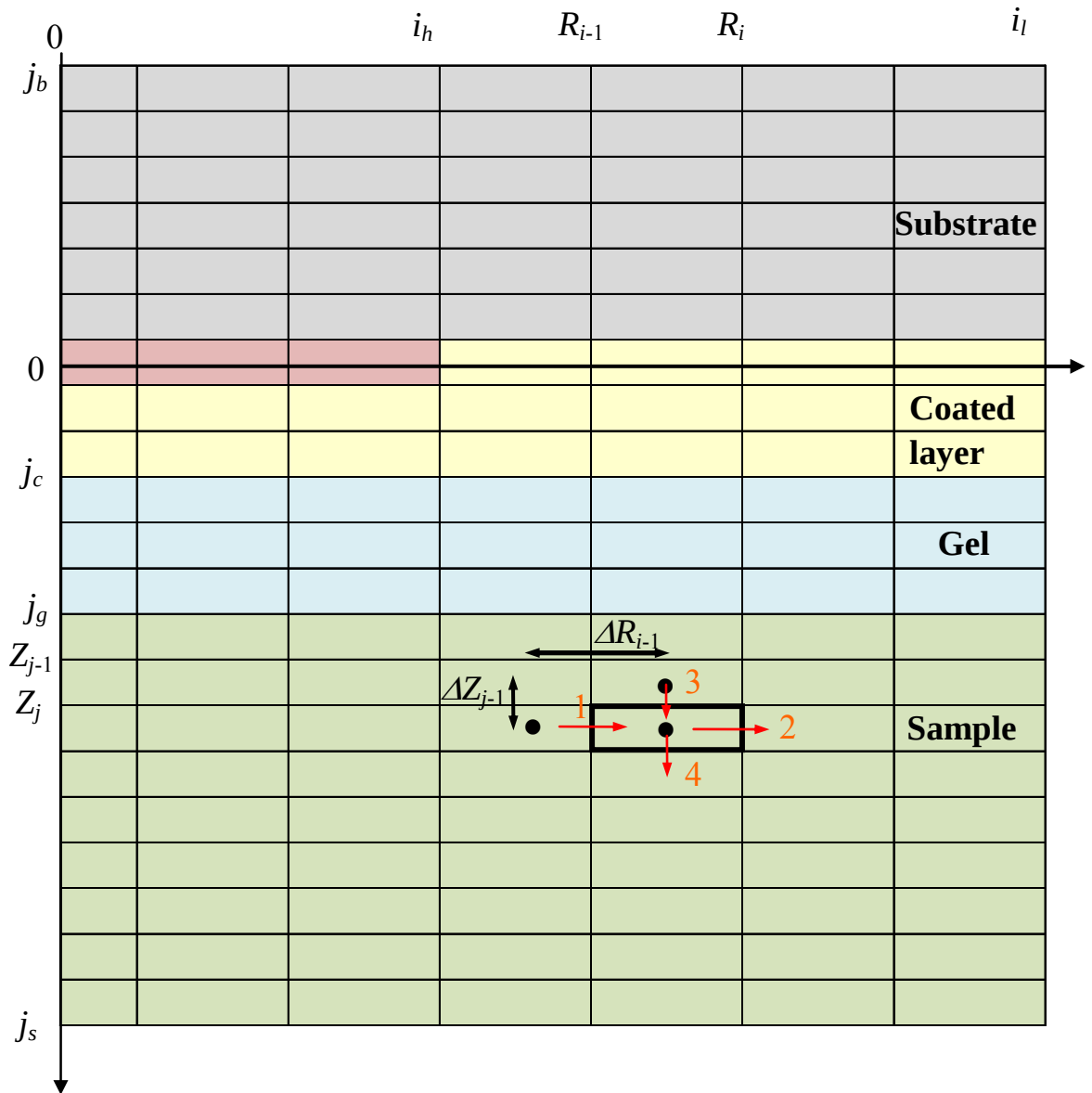


Fig. 2.3 Schematic of grid for numerical analysis

2.3 Protocol to Determine Thermal Transport Properties

Measurement of thermal transport properties includes a process to find the theoretical temperature rise that fits the experimental data, *i.e.* a process to determine the thermal conductivity λ , the thermal diffusivity α and the thickness δ of the gel layer which minimize the cumulative squared difference S in the average temperature of the sensor between the theory and experiment:

$$S = \sum_{i=1}^N (T_{exp,i} - T_{cal,i}(\lambda, \alpha, \delta))^2 \quad (32)$$

where N is the total number of experimental measurements, $T_{exp,i}$ is the i -th measured temperature, and $T_{cal,i}$ is the temperature obtained from the numerical solution at the time of measurement. The Gauss-Newton algorithm with a numerical approximation to the Jacobian was used for this nonlinear least-squares problems. The method for determination of λ and α in a short-hot-wire method [39] was extended to a three-parameters problem. The algorithm is as follows:

- (1) Guess λ , α and δ .
- (2) Solve Eq. (1) numerically to obtain transient average temperature $T_{cal,i}(\lambda, \alpha, \delta)$ ($i=1 \sim N$).
- (3) Solve Eq. (1) numerically to obtain three sets of temperatures for different combinations of λ , α and δ , *i.e.* $T'_{cal,i}(\lambda + \Delta\lambda, \alpha, \delta)$, $T''_{cal,i}(\lambda, \alpha + \Delta\alpha, \delta)$ and $T'''_{cal,i}(\lambda, \alpha, \delta + \Delta\delta)$ ($i=1 \sim N$).
- (4) Find x_λ , x_α and x_δ that satisfy the following equation :

$$S_{linear} = \sum_{i=1}^N \left[(T_{exp,i} - T_{cal,i}) - x_\lambda (T'_{cal,i} - T_{cal,i}) - x_\alpha (T''_{cal,i} - T_{cal,i}) - x_\delta (T'''_{cal,i} - T_{cal,i}) \right]^2 = \min \quad (33)$$

- (5) Set new values for λ , α and δ such that

$$\lambda^{new} = \lambda + x_\lambda \Delta\lambda, \quad \alpha^{new} = \alpha + x_\alpha \Delta\alpha, \quad \delta^{new} = \delta + x_\delta \Delta\delta \quad (34)$$

- (6) Repeat steps (2)-(5).

The values of $\Delta\lambda$, $\Delta\alpha$ and $\Delta\delta$ were taken to be 1 % of the current estimates λ , α and δ . The step (4) was carried out with the linear least-square method using Gram-Schmidt ortho-normalization and Q-R factorization.

The first guess was taken by comparing the experimental data with a prepared data set of calculated temperature rise. The average temperature rise of the sensor has been calculated as a function of time assuming a gel layer of the cavity depth, 50 μ m, for various combinations of λ and α , which are given in increments of 20 %. The values of λ and α that yield minimum difference from the experimental data were chosen from the prepared set as the first guess.

The standard deviation of the difference between the experimental data and calculated temperatures, *i.e.*

$$SD_{\Delta T} = \sqrt{\frac{S}{N}} = \sqrt{\frac{1}{N} \sum_{i=1}^N (T_{exp,i} - T_{cal,i}(\lambda, \alpha, \delta))^2} \quad (35)$$

was evaluated after step (4) of each iteration, and used as an index to convergence.

2.4 Simulated Experimental Data

Feasibility of the proposed method was examined using simulated experimental data, which were generated from the solution to Eq. (1)-(5). Taking into account the scattering that the experimental data inherently have, a variation to the calculated temperature rise was added using random numbers. The simulated experimental data were thus prepared at intervals of 0.03 s so that the data have a scattering of the standard deviation

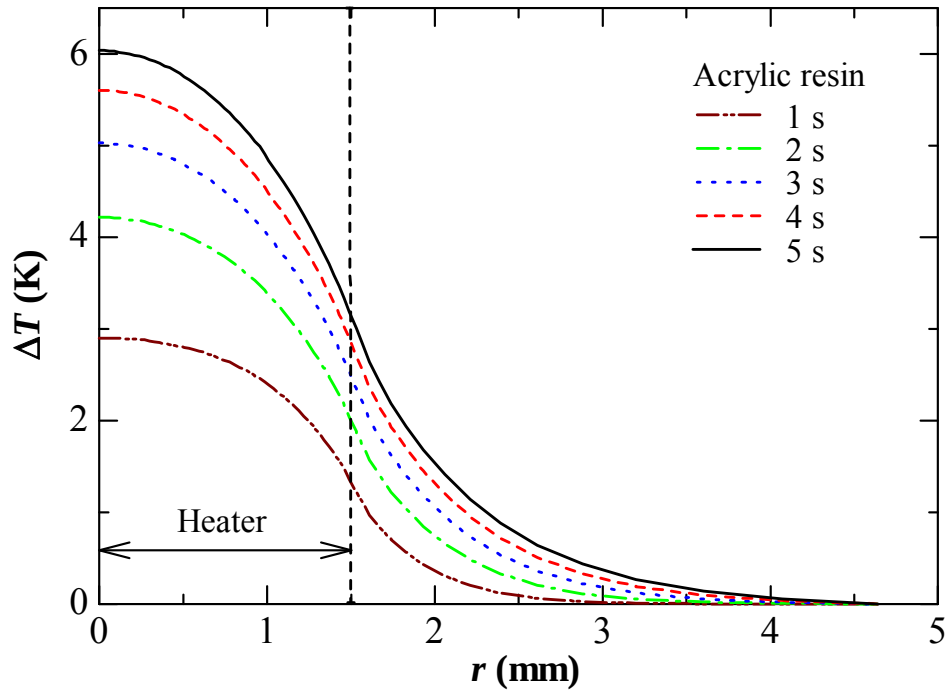
$$SD_{exp} = \sqrt{\frac{1}{N} \sum_{i=1}^N (T_{exp,i} - T_{theo,i})^2} \quad (36)$$

SD_{exp} of 0.015 K was assumed in most cases in the present study. The simulated experimental data with $SD_{exp} = 0$ K and 0.03 K were also used for examination of the effect of scattering. The acrylic resin was selected as a specimen to examine convergence process with iteration. The effect of heating power and time on the measurement error was also discussed using acrylic resin. To examine the range of λ

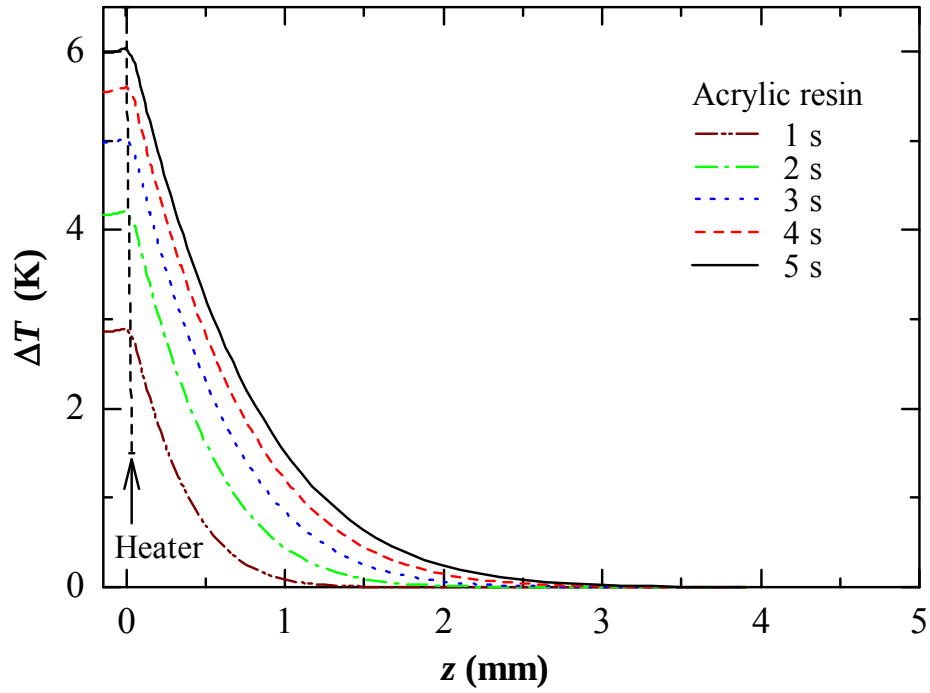
and α to be measured by the proposed method, water, zirconia and liver were also assumed as samples.

2.5 Calculated Results

The change of the temperature distribution, $\Delta T (= T - T_0)$, is shown in Fig. 2.4 for acrylic resin heated at 20 mW in term of the temperature rise. Figure 2.4 (a) shows the radial temperature distribution at the surface of substrate ($z = 0$). Since the thickness of the heater is extremely thin (40 nm), its temperature was the same as that at $r < 1.5$ mm shown in Fig. 2.4(a). The temperature rise at the center reached 6 K by heating for 5 s. However, a significant temperature distribution was found in the heater due to radial heat conduction; the temperature rise at the edge of the heater was approximately a half of that at the center. Thermally affected area increased with time but did not exceed 4.5 mm from the center even after 5 s.



(a) Radial temperature distribution at the substrate surface



(b) Temperature distribution along z-direction at the center

Fig. 2.4 Local temperature rise in the sample and the substrate for acrylic resin as a sample

The temperature distribution along z-direction at the center ($r = 0$) shown in Fig. 2.4(b) indicates that the temperature did not increase at the place 3 mm deep from the surface by heating for 5 s. The temperature at the surface of the glass substrate was lower than that of the heater due to conduction in the substrate, but the difference was smaller than 0.06 K.

The average temperature of the heater in the case shown in Fig. 2.4, for acrylic resin and 50- μm thick gel layer, is shown as a function of time in Fig. 2.5. Although the highest local temperature rise was 6 K at 5 s (Fig. 2.4), the average temperature rise was 4.8 K. In addition to this as a base temperature rise, Fig. 2.5 also shows six more lines calculated with different combinations of λ , α and δ to examine the effect of these parameters on the temperature rise. These lines were obtained with only one parameter deviated from the base by +10% or -10% keeping the other parameters the same. Comparison with the base line indicates that the temperature is

higher for smaller λ and larger α . It is also higher for smaller δ because of higher thermal conductivity of the gel compared to that of acrylic resin. A 10 % and –10% deviation of λ from the base resulted in 5.4 % and 6.1% differences in the temperature rise, respectively, at 5 s. In contrast, the influence of α and δ was much smaller. The temperature deviation from the base was only 2.1 % or 2.3 % for the difference in α by $\pm 10\%$, and smaller than 0.9 % for 10 % difference in δ . The result that showed significant difference in the influence of three parameters implies a difficulty in determining λ , α and δ within the same magnitude of errors.

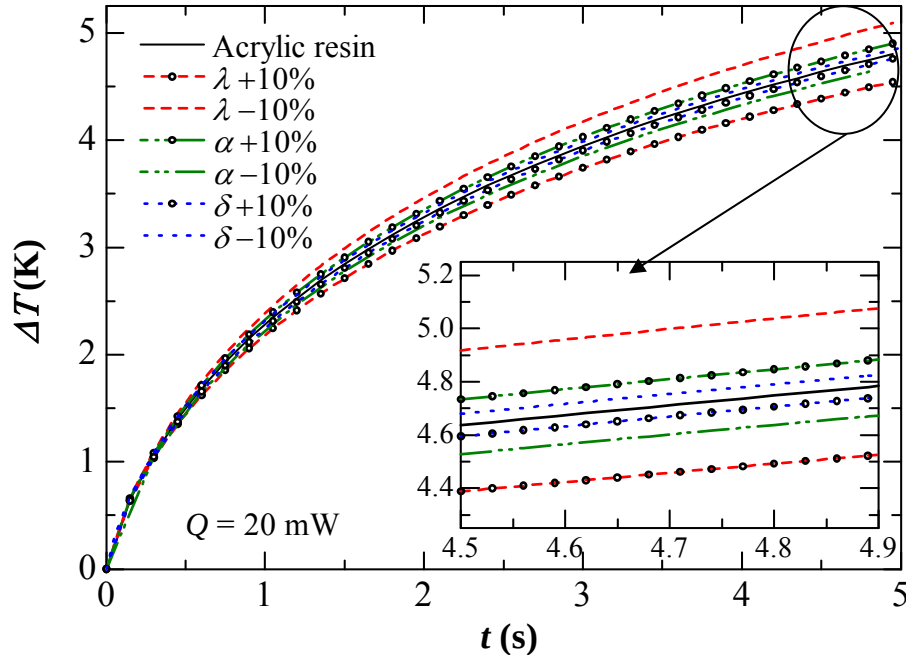


Fig. 2.5 Average temperature rise of the heater as a function of time for acrylic resin and its deviation by different λ , α and δ

2.6 Convergence Process to Obtain Final Values of λ , α , and δ

An example of simulated experimental data is shown in Fig. 2.6 for acrylic resin on the supposition of 60- μm thick gel layer. A scattering of the data was also assumed to be $SD_{exp} = 0.015$ K. The process to determine λ , α and δ based on this experimental data obtained for 5 s after the start of heating is summarized in Table 2.1 and Fig. 2.6. The first guess was $\lambda_1 = 0.244$ W/(mK), $\alpha_1 = 1.5 \times 10^{-7}$ m²/s and $\delta_1 =$

50 μm . Since the application of the Gauss-Newton algorithm with three variables from the first iteration sometimes did not work and gave negative δ value as the next guess, δ was fixed at 50 μm for the first several steps. Thus the iteration was carried out only with two variables, λ and α , until $SD_{\Delta T}$ reached nearly a minimum in every case. Although the deviation of the calculated temperature rise from the experimental data was larger for the second guess than the initial guess, it decreased with a progress of iteration with new guesses for λ and α . After the decrease of $SD_{\Delta T}$ became smaller than 10 %, from 0.019 K to 0.018 K in this case, δ was included as a variable in the Gauss-Newton algorithm. Although the difference between the temperature for the 4th guess and 7th guess is not distinguishable in Fig. 2.5, $SD_{\Delta T}$ did decrease with further iteration and converged to 0.016 K, which was the same magnitude as the scattering of the experimental data. The finally determined values were: $\lambda_7 = 0.210 \text{ W/(mK)}$, $\alpha_7 = 1.215 \times 10^{-7} \text{ m}^2/\text{s}$ and $\delta_7 = 61.4 \mu\text{m}$, which were different from the true values by 0.2 %, 1.3 % and 2.4 %, respectively.

2.7 Effect of Data Scattering on the Measurement Error

Since the measurement error could possibly depend on the scattering of the data, different magnitude of SD_{exp} , *i.e.* $\sim 0.015 \text{ K}$ and $\sim 0.03 \text{ K}$, were assumed as the inherent scattering of the experimental data, and three different data for each SD_{exp} were examined. In addition, the algorithm was also checked with the data that has no scattering. The difference between the finally determined value and the given true value is shown as a function of SD_{exp} in Fig. 2.7.

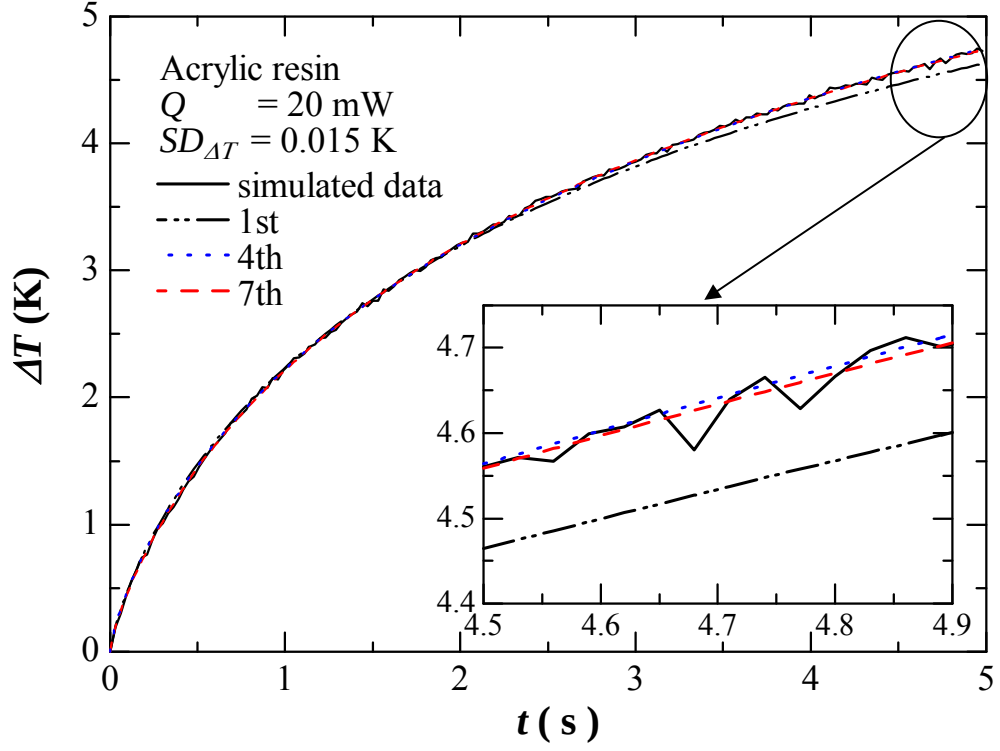


Fig. 2.6 Simulated experimental data for acrylic resin and the calculated temperature rise at some iteration steps.

Table 2.1 Values of λ , α and δ at iteration steps

i	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda_{cal} - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha_{cal} - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta_{cal} - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1	0.244	1.500	50	16.2	25.0	-16.7	0.057
2	0.182	0.639	50	-13.3	-46.8	-16.7	0.170
3	0.178	0.737	50	-15.3	-38.6	-16.7	0.019
4	0.179	0.750	50	-14.9	-37.5	-16.7	0.018
5	0.211	1.138	61.2	0.7	-5.1	2.0	0.063
6	0.210	1.213	61.5	-0.2	1.1	2.5	0.016
7	0.210	1.215	61.4	-0.2	1.3	2.4	0.016

The values determined by the present algorithm coincided with the given values when $SD_{exp} = 0$. However, the errors of all three variables, λ , α and δ , increased with SD_{exp} , and depended significantly on the specific experimental data. The error of δ was considerably large, *i.e.* up to 9.2 % for the data with $SD_{exp} = 0.03$

K. The error of α was 9.2 % for $SD_{exp} = 0.015$ K and 17.4 % for $SD_{exp} = 0.03$ K in the worst case. In contrast, λ was obtained within an error of 2.8 % for $SD_{exp} = 0.015$ K and 5.6 % for $SD_{\Delta T} = 0.03$ K. The difference in the error that depends on the variables was resulted from the difference in the sensitivity of the temperature rise to λ , α and δ , which was already demonstrated in Fig. 2.5. It is interesting that the largest error of δ was smaller than that of α though the temperature rise was more sensitive to α than δ . This is probably because the effect of parameters depends on the time after the start of heating. Although the error depends on the specific data, the results indicates that λ is able to be determined within a few percent error if the scattering has a magnitude of 0.015 K by the standard deviation, and the error of α would be larger. The error in δ would not be a critical concern because it is not a target of the measurement.

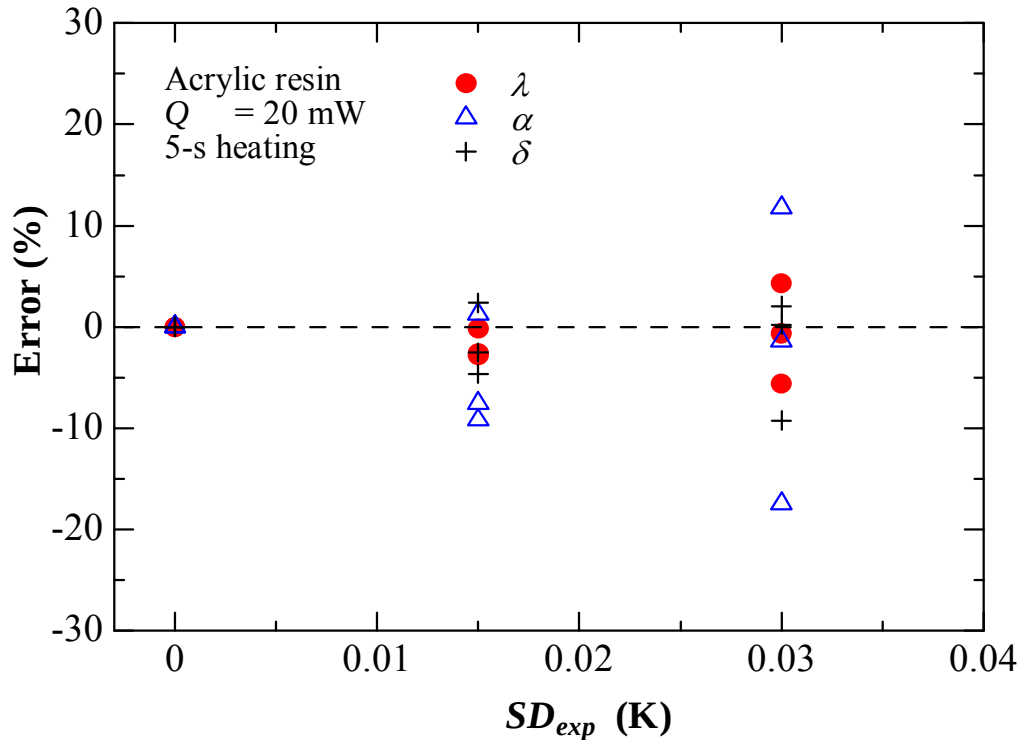


Fig. 2.7 Effect of scattering of experimental data on the measurement error

2.8 Effect of Heating Power on the Measurement Error

The effect of heating power Q on the measurement error was examined with simulated experimental data for $Q = 10, 20$, and 30 mW. The finally determined values of λ and α were compared with given true values, and the difference is shown in Fig. 2.8 for three different experiments for each Q . They are shown as a function of the final temperature rise at 5 s after the start of heating, since the error may depend directly on the magnitude of the temperature rise. Errors were different in each data again at the same heating power. However, the variation of the error, *i.e.* the largest error, decreased with increasing the heating power. The largest error of α , 29.0 % at $Q = 10$ mW, reduced to 12.0 % in the absolute value at $Q = 30$ mW. The error of λ was much smaller than α : 10.7, 5.6 and 3.8 % at 10, 20 and 30 mW, respectively, in the worst case.

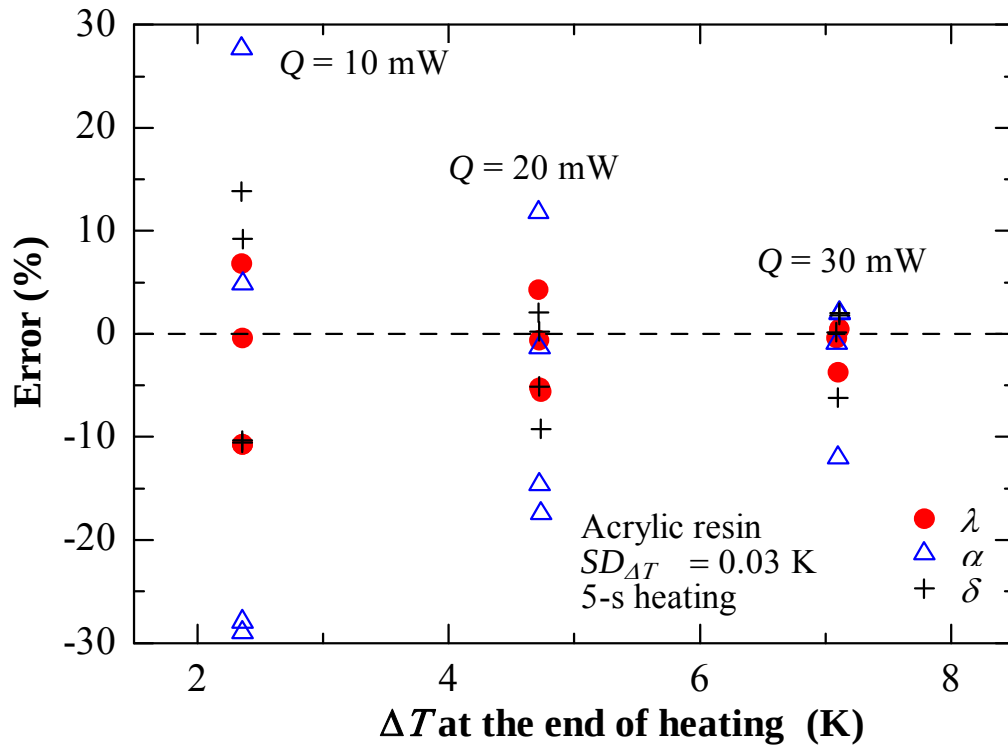


Fig. 2.8 Effect of heating power on the measurement error

The fact that the error depended on the heating power was probably resulted from the magnitude of scattering relative to the temperature rise. Even though SD_{exp} was approximately 0.015 K for all the data in Fig. 2.8, its relative value normalized by the temperature rise is smaller for larger Q . From this point of view, the larger heating power that yields larger temperature rise is preferable for the measurement. However the temperature dependence of the thermal transport properties is the other factor to be taken into account to determine the optimum magnitude of the temperature rise.

2.9 Effect of Heating Time on the Measurement Error

A longer heating also increases the heater temperature, which could have an influence on the measurement error. Figure 2.9 compares the results obtained from the data heated for 5 s and that for 3 s. The measurement error is shown again as a function of temperature rise at the end of heating. Heating acrylic resin at 20 mW for 3 s and 5 s respectively induced 3.9 and 4.7 K in the temperature rise. It is obvious that longer heating resulted in the smaller error of both λ and α . This suggests that heating the specimen for longer time would be favourable for the measurement. However, since the longer heating results in deeper penetration of thermal effect, the size of specimen is another factor to determine the heating time.

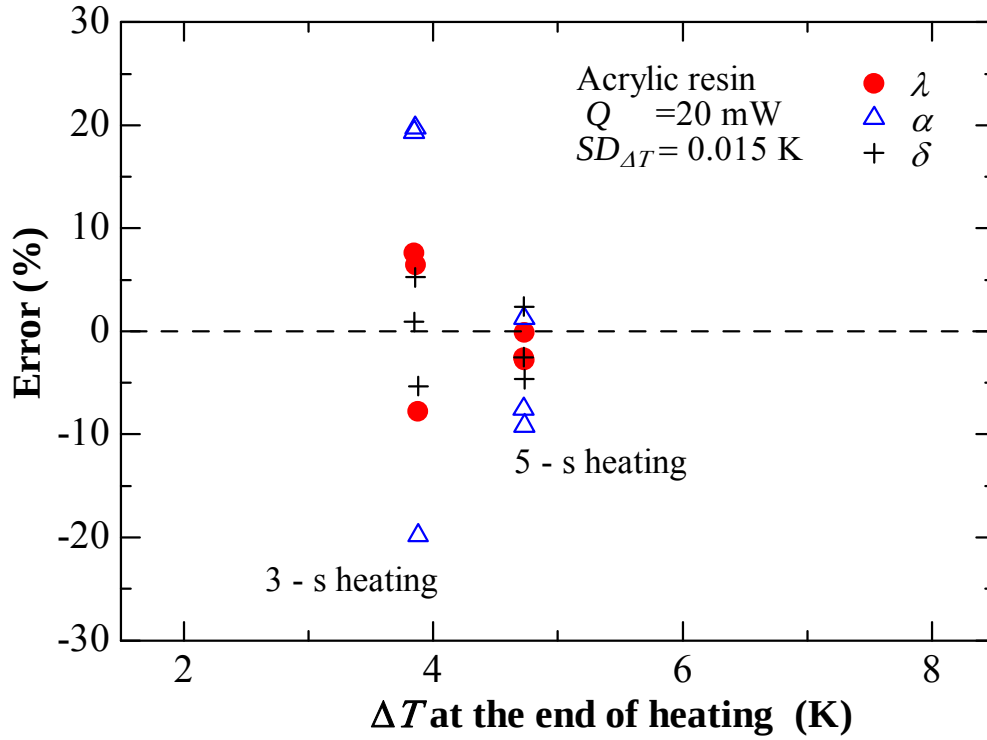


Fig. 2.9 Effect of the heating time on the measurement error

2.10 Virtual Measurement of Different Materials

In addition to the acrylic resin that was used for the examination of the present method, several other materials were tested as supposed specimens. Physical properties of supposed samples and measuring conditions are summarized in Table 2.2. Taking into account that one of the promising samples for this method are biological materials, the thermophysical property of water was assumed for a typical example. The thermal transport properties of a liver were cited from a literature [48] and used as a supposed sample. Zirconia was chosen from materials that have the thermal conductivity one order of magnitude higher than water and acrylic resin. The heating power was selected such that the temperature rise after 5 s reaches 3-5 K. The scattering of $SD_{exp} = 0.015 \text{ K}$ was assumed in all the simulated data.

Table 2.3 shows the errors of λ and α that were obtained from three simulated experiments. Seven to eight iterations were needed to determine λ and α . The standard deviation of the difference between the experimental data and the calculated

temperatures, $SD_{\Delta T}$, after the final iteration was ~ 0.015 K, which was approximately the same as the scattering of the data, SD_{exp} , for most cases. Both λ and α were determined within the error of 1.3 % for every material in the best case. The root mean square of the error of three trials were 0.5-2.2 % for λ and 1.8-6.9 % for α . The errors in λ and α of the average of three data were 0.1- -1.9 % and -1.4 - -5.2 %, respectively. The results suggest that a repetition of measurement and their average could reduce the error. Although the error of average α was larger than that of λ for every material, the errors of these data would be allowable for the present method.

Table 2.2 Supposed samples and measuring conditions

Material	λ (W/mK)	α (10^{-7} m ² /s)	Q (mW)	ΔT (K)	z_{aff} (mm)	r_{aff} (mm)
Water ⁽⁴⁹⁾	0.420	1.17	20	3.1	4.0	4.6
Acrylic ⁽⁴⁶⁾	0.210	1.20	20	4.7	4.0	4.6
Zirconia ⁽⁵⁰⁾	3.100	10.5	90	4.8	7.9	4.6
Liver ⁽⁴⁸⁾	0.417	1.05	30	4.5	4.0	4.6

Thermally affected region in the sample is also shown in Table 2.2 in terms of the depth z_{aff} from the surface of the sample and the radius r_{aff} at the surface where the temperature rise occurs after heating for 5 s. The values of z_{aff} were almost the same for the tested materials except for zirconia because of their similar thermal diffusivities. The information given in Table 2.2 is important for knowing the requirement for the sample size. The results indicate that a specimen must be larger than a cylinder 4.6 mm in diameter and 4 mm thick when the thermal transport properties are similar to that of water. Larger sample is required for zirconia and similar materials.

Table 2.3 Measured thermal conductivity and thermal diffusivity

Material	λ (W/mK)	α (10^{-7} m ² /s)	Iterations	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$SD_{\Delta T}$ (K)
Water	0.417	1.17	7	-0.7	0.2	0.0156
	0.409	1.07	7	-2.5	-8.2	0.0154
	0.415	1.10	7	-1.3	-5.7	0.0144
<i>Average</i>	<i>0.414</i>	<i>1.12</i>		<i>-1.5</i>	<i>-4.6</i>	
<i>r.m.s.</i>				<i>1.7</i>	<i>5.8</i>	
Acrylic	0.210	1.22	7	-0.2	1.3	0.0156
	0.204	1.09	7	-2.8	-9.2	0.0143
	0.205	1.11	7	-2.6	-7.5	0.0154
<i>Average</i>	<i>0.206</i>	<i>1.14</i>		<i>-1.9</i>	<i>-5.2</i>	
<i>r.m.s.</i>				<i>2.2</i>	<i>6.9</i>	
Zirconia	3.085	10.4	8	-0.5	-0.9	0.0156
	3.121	10.2	8	0.7	-3.0	0.0153
	3.098	10.5	8	-0.1	-0.3	0.0130
<i>Average</i>	<i>3.102</i>	<i>10.4</i>		<i>0.1</i>	<i>-1.4</i>	
<i>r.m.s.</i>				<i>0.5</i>	<i>1.8</i>	
Liver	0.414	1.05	7	-0.6	-0.1	0.0182
	0.410	0.99	7	-1.7	-5.7	0.0154
	0.414	1.01	7	-0.8	-3.9	0.0144
<i>Average</i>	<i>0.413</i>	<i>1.02</i>		<i>-1.1</i>	<i>-3.2</i>	
<i>r.m.s.</i>				<i>1.2</i>	<i>4.0</i>	

DESIGN OF THE SENSOR AND EXPERIMENT

3.1 Design of Stamp Sensor

The final design of the sensor would be a stamp-type device where a sensor is fabricated on the bottom of a cylindrical holder as shown in Fig. 2.1. However, in the present study a sensor pattern with electrodes was fabricated on a 0.16-mm thick 22 mm x 40 mm glass substrate by the physical vapor deposition (PVD) of platinum as shown in Fig. 3.1. A circular pattern of 3 mm in diameter was drawn with a single stroke of a 125 μm wide line. An annular silicone rubber sheet, 50- μm thick and 15 mm in the inner diameter, was used as the spacer. The glass substrate was held with a hollow cylinder (Fig. 3.2). A sample was heated not directly from the heater/sensor but through the substrate to avoid the sensor from being peeled off during cleaning after experiments, even though the sensor was coated by a glass layer.

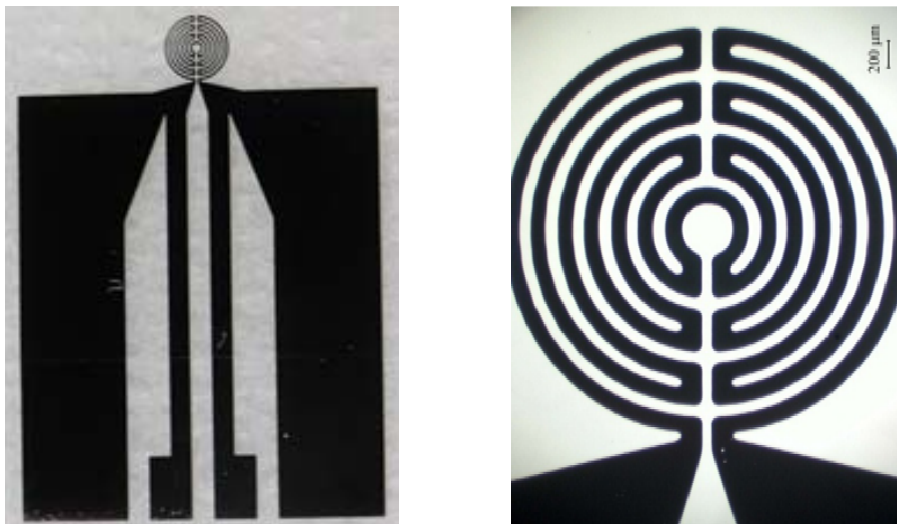


Fig. 3.1 Pattern of the sensor deposited on the glass substrate

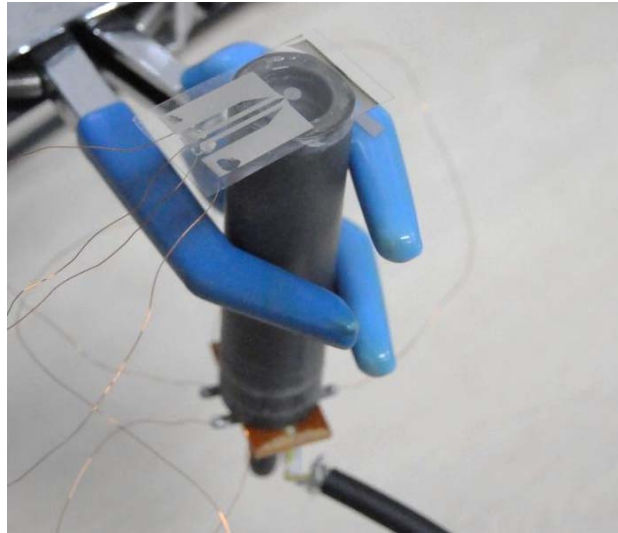


Figure 3.2 A glass substrate held with a hollow cylinder

3.2 Fabrication of Sensor

The sensor pattern was fabricated by following processes (Fig. 3.3):

1. Wash a glass substrate by acetone and dried in the air.
2. Spin-coat an image reversal photoresist (AZ-5214E, Clariant K.K.) on the substrate and prebake at 90°C for 90 seconds using a hot plate.
3. After putting a mask on the substrate, exposure it to UV-light.
4. Reversal bake the photoresist at 120°C for 100 seconds.
5. Expose the entire surface of the substrate to UV-light.
6. Wash the photoresist at unexposed area by a developer solution.
7. Deposite platinum film on the substrate by PVD (Physical Vapor Deposition).
8. Remove photoresist together with platinum film (lift-off) by a remover solution to obtain designed pattern of the platinum film.
9. Deposite a SiO_2 film by sputtering to provide protective film.

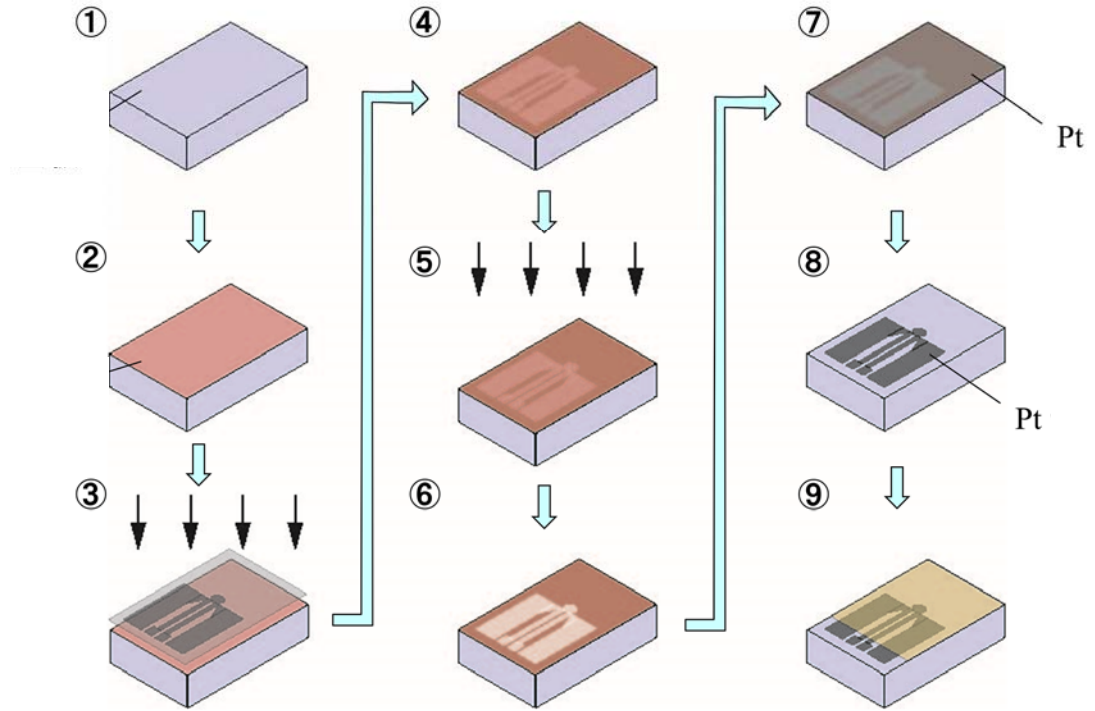


Fig. 3.3 Process to fabricate a pattern on the glass substrate

3.3 Experimental Apparatus and Procedure

The electric circuit and the setup for measurement are described in Figs. 3.4 and 3.5. The electrical resistance of the sensor was measured by a four-terminal method using a constant-current generator (R6245, Advantest) and two digital multimeters (Model 2002, Keithley); the voltage drops in the sensor and a standard resistor connected to the power line were measured at every 0.018 s.

A commercially available gel that is utilized in the medical ultrasonic echo was used in the experiment to eliminate thermal contact resistance. The sensor was pressed against a sample, more accurately, the sample was put on the device actually, after spreading a small amount of gel in a shallow cavity. Measurement was started after waiting for the system to reach thermal equilibrium. A constant current at 0.1 mA, which was negligibly small in regard of temperature rise of the sensor, was applied first for 5 s to measure the initial temperature of the system. The current was then increased stepwise to a prefixed value that is appropriate to obtain ~5K in the

temperature rise in 5 s. The measured voltage and current was recorded by a data acquisition system that was controlled by a computer.

Four different samples were prepared for measurement: an acrylic resin block of $40 \times 30 \times 15 \text{ mm}^3$, a stainless steel (SUS304) block of $40 \times 40 \times 30 \text{ mm}^3$, and a machinable ceramic cylinder of $\phi 30 \times 20 \text{ mm}^3$, and 1% agar gelated in a 20 mm deep plastic dish 100 mm in diameter. In the present experiment, the measuring device was set upside down to make it easy to handle taking into account its size and connected cables; a sample was put on the sensor-deposited glass plate that was held on the top of a hollow cylinder (See Figs. 3.2 and 3.5).

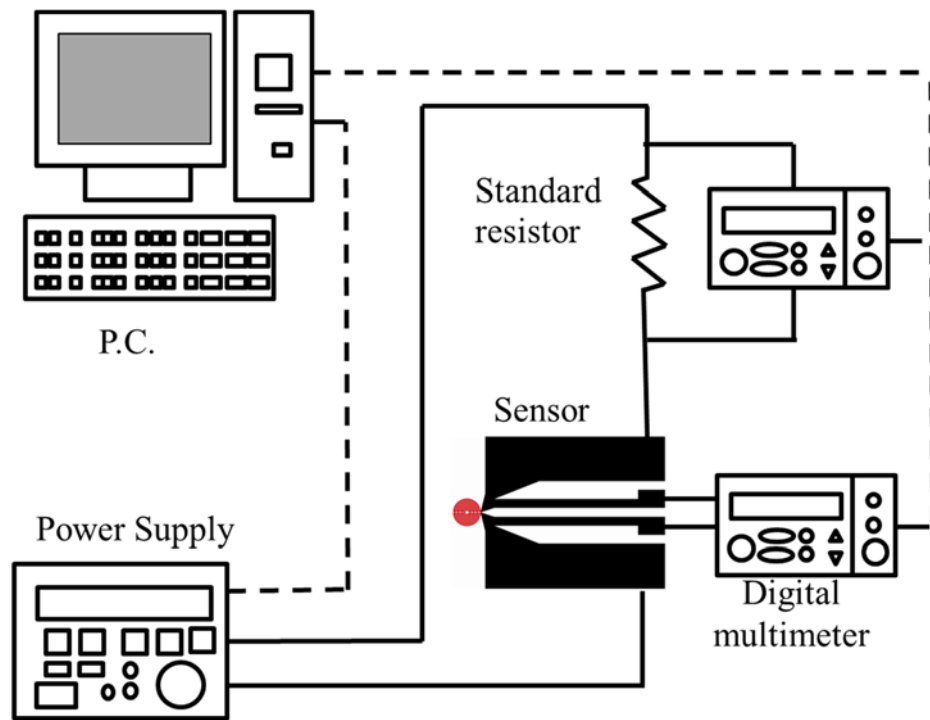


Fig. 3.4 Schematic of measurements system

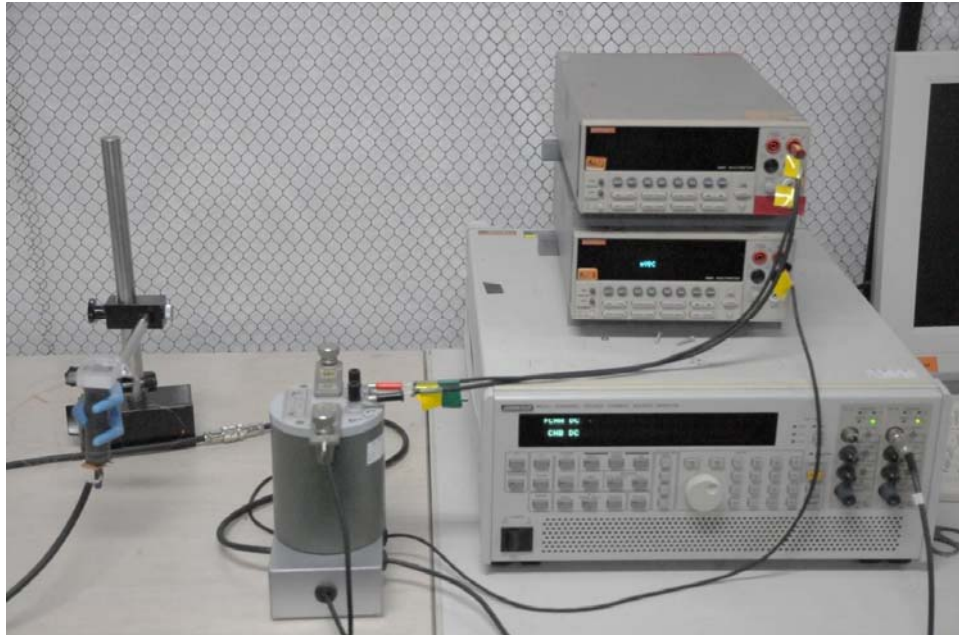


Figure 3.5 Setup of measurement apparatus



Fig. 3.6 The low temperature bath NCB series

3.4 Calibration of Sensor

The electrical resistance of the sensor was calibrated as a function of temperature by applying small currents to the sensor with keeping the sensor in a temperature-controlled dielectric liquid FC-84. A thermostat bath that maintains the liquid at $-30 - 80^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$ was used (Fig. 3.6.)

The results are shown in Table 3.1 and Fig. 3.7. The electrical resistance increased linearly with temperature and was correlated very well by a correlation shown in Fig. 3.7.

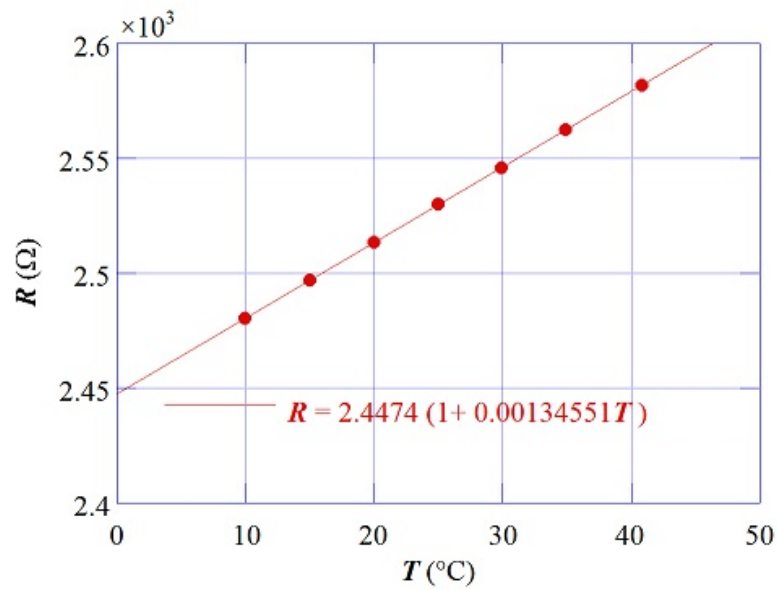


Fig. 3.7 Relationship between temperature and electrical resistance of the sensor

Table 3.1 Relationship between temperature and electrical resistance of the sensor

Preset Temperature (°C)	Actual Temperature (°C)	Electrical Resistance (Ω)
10	10.00	2480.4
20	20.03	2513.4
30	29.90	2546.0
40	40.83	2581.8
35	34.90	2562.3
25	25.02	2529.9
15	15.05	2496.9

3.5 The Time at the Start of Heating

One of the most important issues in the process of comparing the measured temperature rise with theoretical analysis is to properly determine the ‘time zero’ at the start of heating. Since the virtual experimental data used in the feasibility study that was described in Chapter 2 were based on the theoretical analysis, the time at the start of heating has been specified exactly. However, in the measurement, it would be sometime during an interval of measurement, 0.18 s in the present experiment. In addition, the measured data showed some anomalous behavior in measured voltages at the start of heating as shown in Fig. 3.8. This might be caused by switching the applied current from 0.1 mA for measuring initial temperature to significantly higher current for heating, 2.8 mA for acrylic resin as an example. A 1-ms difference in timing between the measurements of voltage and current might also induce undesirable effect. These differences caused a kinky behavior of measured temperature at the start of heating as shown in Fig. 3.9, which resulted in missing of one or two data, and made it more difficult to specify the time at the start of heating.

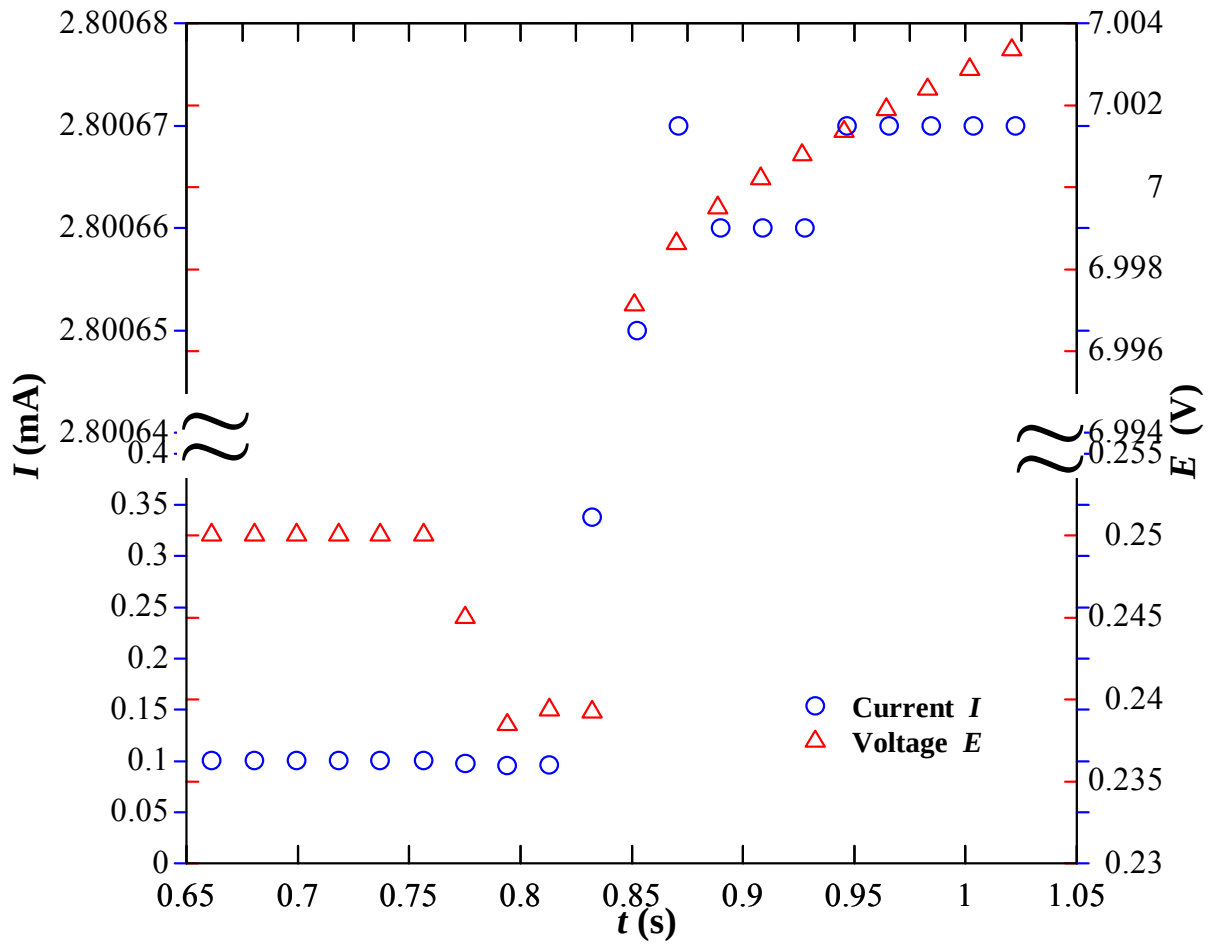


Fig. 3.8 An example of anomalous behavior of measured voltage and current at the start of heating

As the first trial, the time zero has determined at the point one interval of measurement, 0.018 s, ahead of the point that exhibited first temperature rise. However, it didn't work for obtaining final results depending on individual experiments. This was because the difference in the temperature between theory and experiment at the start of heating did not change significantly with changing λ and α . The example is shown in Fig. 3.10, which shows that the difference decreases with a progress of iteration with the exception of the time just after the start of heating. This implies that the time zero should be taken into account as a variable in addition

to λ , α , and δ . However, since this is unrealistic from the technical point of view at this stage of the study, the time zero with more straightforward way was determined. The difference in time between the experimental data and the temperature rise calculated with first assumption of λ and α was estimated for the first several points as shown in Fig. 3.11. Then the time zero was shifted by Δt , which was taken as an average of first three, i.e. Δt_1 , Δt_2 , and Δt_3 in Fig. 3.11 and Fig. 3.12.

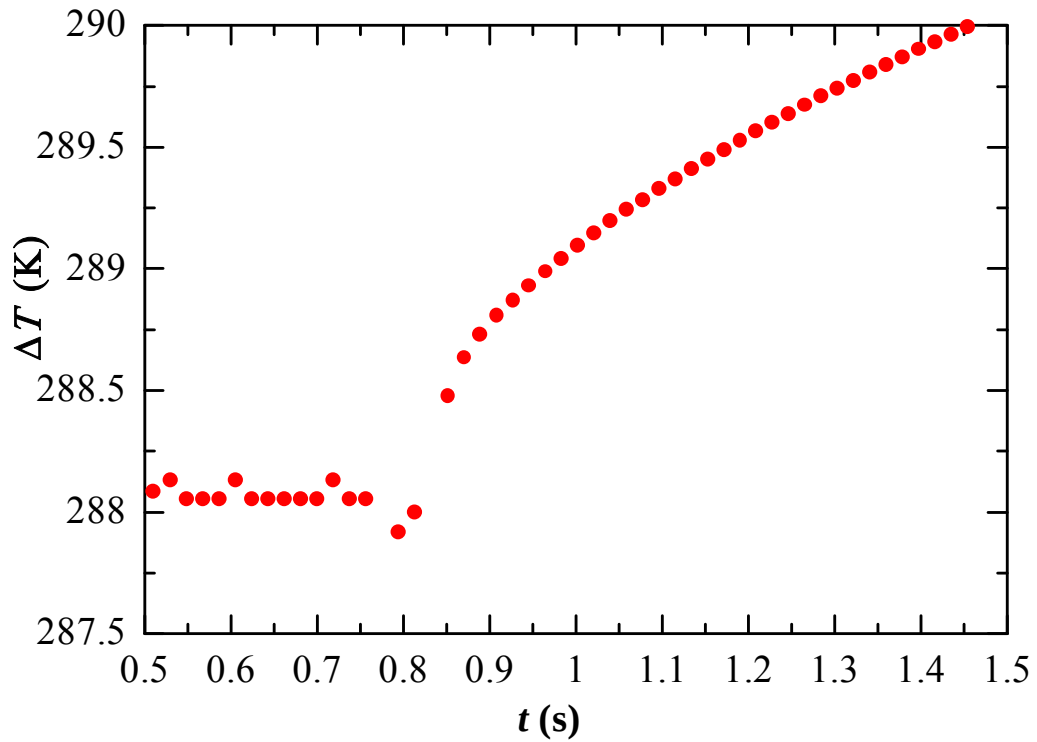


Fig. 3.9 Measured temperature change at the start of heating

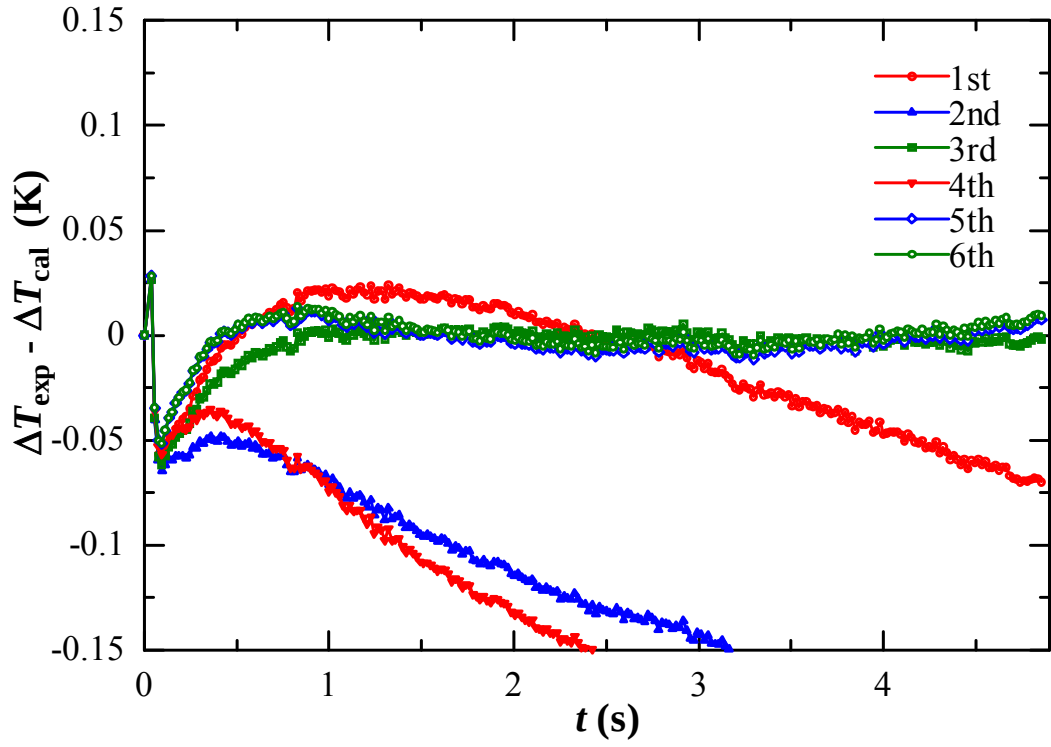


Fig. 3.10 Difference of the measured and calculated temperature

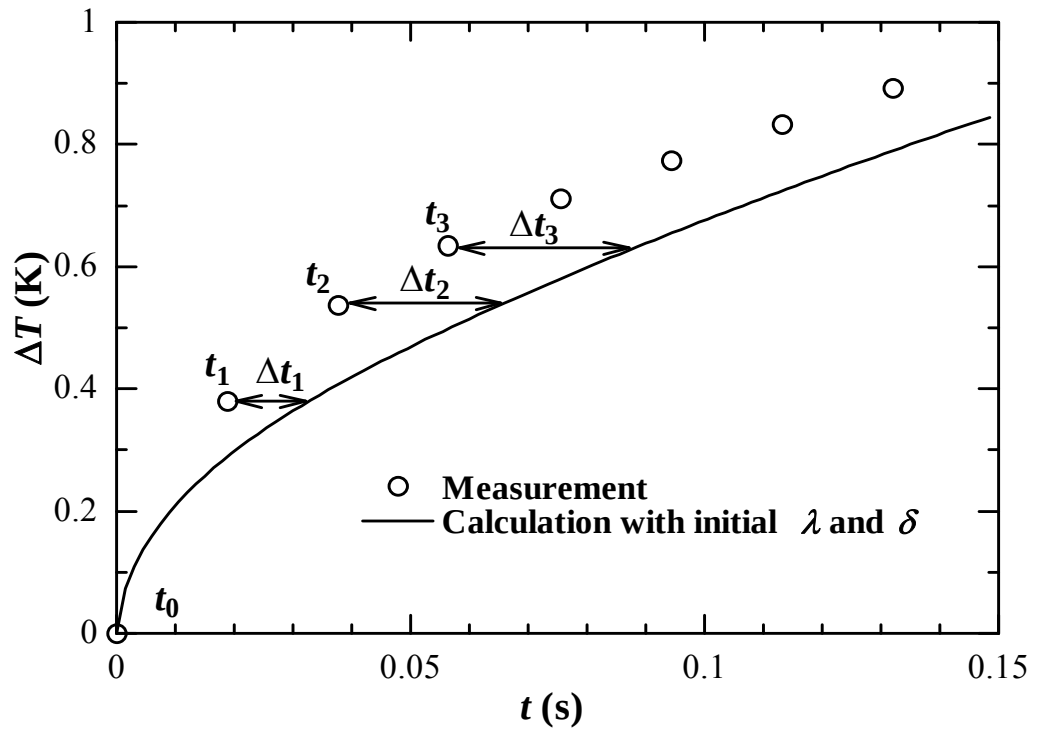


Fig. 3.11 Moved measured temperature rise data for acrylic resin

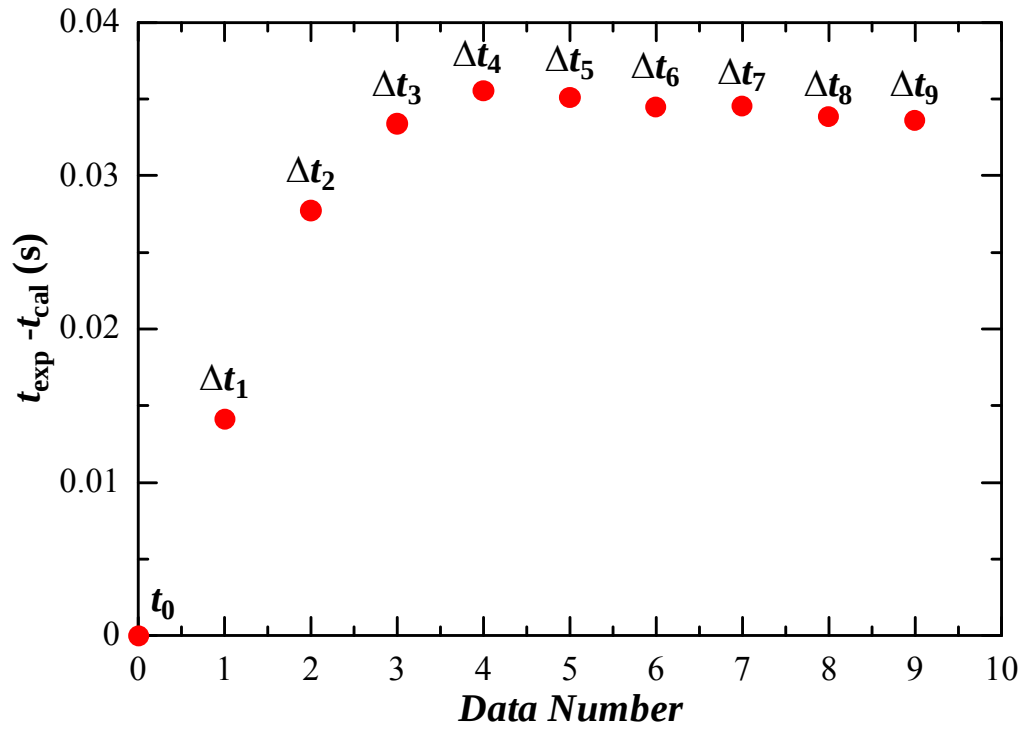


Fig. 3.12 The difference in time between measurement and calculation with first guess of λ and α at the same temperature rise

CHAPTER 4

FEASIBILITY VALIDATION WITH EXPERIMENTS

4.1 Iteration Process

The process for determining the thermal conductivity and the thermal diffusivity for measurement of acrylic resin is demonstrated in Fig. 4.1, and Table 4.1. Figure 4.1(a) shows the temperature as a function of time. Figure 4.1(b) shows the difference between measured and calculated temperature at some steps in the iteration process. The assumed data used in each step were summarized in Table 4.1 with the percent difference of these values and the deviation of calculated temperature rise from measurement defined by Eq. (10). As shown in Fig. 4.1(a), ~5-K rise in the temperature of the sensor within 5 s was obtained by heating at ~20 mW. The first assumption was $\lambda_1 = 0.244$ W/mK, $\alpha_1 = 1.50 \times 10^{-7}$ m²/s and $\delta_1 = 50$ μ m, which gave the smallest deviation of the temperature change from the measured temperature $SD_{\Delta T}$ among the prepared data set. The calculated temperature rise was lower than the measured temperature by ~0.08 K at 1 s and the difference increased gradually with time (Fig. 4.1(b)). The thickness of the gel, δ , was fixed at 50 μ m for the following iteration steps as long as $SD_{\Delta T}$ exceeded 0.03 K. In the case shown in Figs. 4.1, δ was incorporated as a variable at the third step after $SD_{\Delta T}$ reduced to 0.015 K at the second iteration (Table 4.1). While the difference between the measured and calculated temperatures increased once up to 0.15 K in $SD_{\Delta T}$ by incorporation of δ , it decreased again with further iteration (Fig. 4.1 and Table 4.1). Iteration was stopped after 6th trial because of no reduction in $SD_{\Delta T}$ from the 5th trial. Although the calculated temperature rise agreed well with the measured temperature within the difference only 0.003 K in $SD_{\Delta T}$, the differences of finally determined values from the literature were -3.0% for λ , -15.4% for α , and -39.1% for δ .

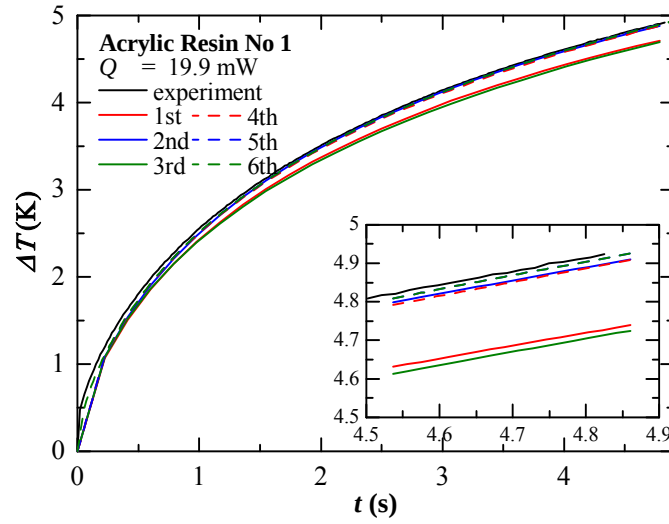
Figures 4.2-4.5 and Tables 4.2-4.5 show the iteration process of another four experimental trials with acrylic resin. The difference in the temperature rise between measurement and calculation at the first iteration step was significantly different for different experiments. It was smallest in experiment 2 (Fig. 4.2) and largest in experiment 5 (Fig. 4.5). Not only $SD_{\Delta T}$ but also the variation of $\Delta T_{cal} - \Delta T_{exp}$ with time depended considerably on experiments. However, λ and α were successfully determined with all experiments shown in Figs. 4.1-4.5 irrespective of different magnitude of $SD_{\Delta T}$ at the first step. Even though $SD_{\Delta T}$ at the last iteration step was acceptably small, it might probably depend on the data at the start of heating, which is clearly observed in Figs. 4.4 and 4.5.

Iteration processes for agar gel are shown in Figs. 4.6-4.10 and Tables 4.6-4.10. Again, the difference between measurement and calculation and how it decreased with iteration depended on each experimental run. It is apparent from Table 4.7 that smallest $SD_{\Delta T}$ at the first iteration step resulted in the smallest number of iteration steps, only three times, to obtain final result.

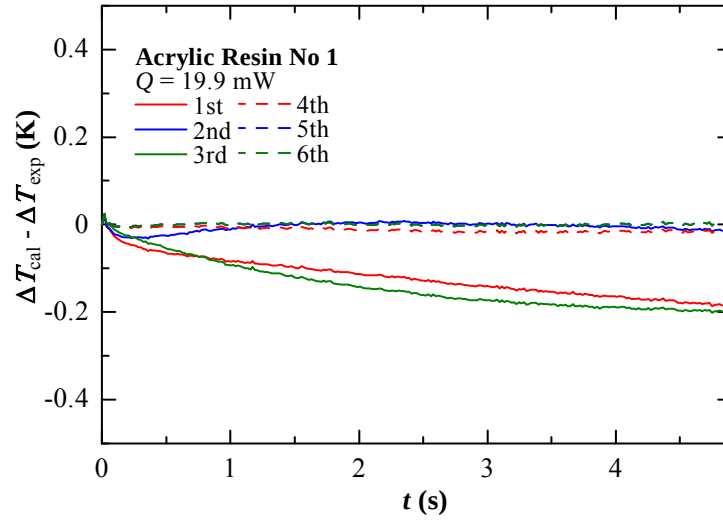
Figures 4.11-4.15 and Tables 4.11-4.15 show the results for machinable ceramics. There is no unique difference to be pointed out in the iteration process from acrylic resin and agar gel. However, generally speaking, the difference between measurement and calculation is smaller than the experiment with acrylic resin and agar gel and required fewer iteration steps to get final results.

Figures 4.16-4.20 and Tables 4.16-4.20 show the results of SUS304. Comparing with all the other materials, the temperature rise for SUS304 was much faster after the start of heating but slowed down immediately. The temperature difference between measurement and calculation at the first iteration step was also much larger than the experiment with the other materials. Probably because of this, the second guess for λ and α were considerably different from the first guesses. In general, the solution process needed more iterations than the other materials.

However, the final values of λ and α finally could be obtained with adequate small values of $SD_{\Delta T}$.



(a) Temperature rise

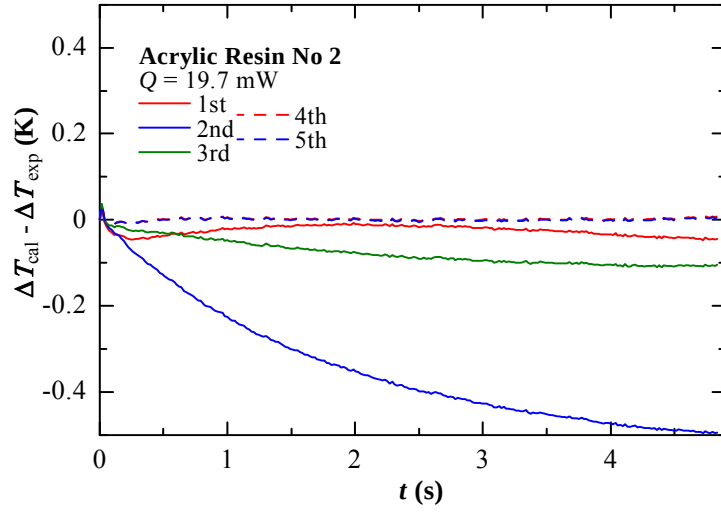


(b) Difference in temperature rise

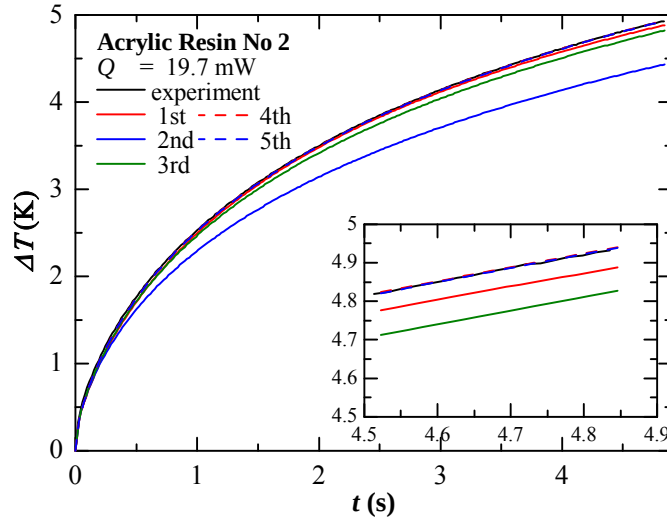
Fig. 4.1 Comparison between T_{cal} and T_{exp} rise (acrylic resin: Experiment 1)

Table 4.1 Values of λ , α , and δ at iteration steps (acrylic resin: Experiment 1)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.244	1.50	50	16.2	25.0	-16.7	0.13
2 nd	0.238	1.67	50	13.1	39.4	-16.7	0.01
3 rd	0.204	0.85	36	-2.8	-29.4	-39.5	0.15
4 th	0.204	1.01	37	-2.7	-15.9	-38.8	0.01
5 th	0.204	1.02	37	-3.0	-15.4	-39.1	0.00
6 th	0.204	1.02	37	-3.0	-15.4	-39.1	0.00



(a) Temperature rise

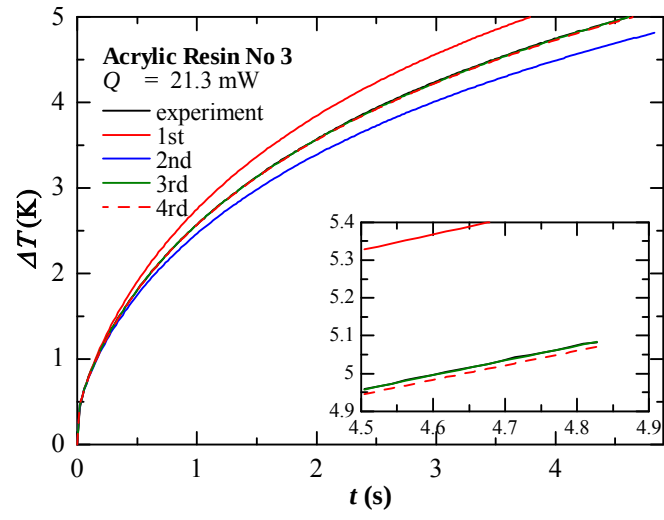


(b) Difference in temperature rise

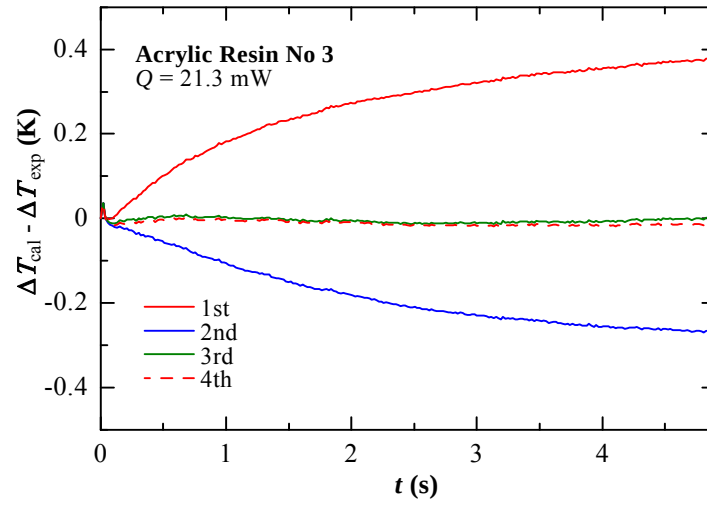
Fig. 4.2 Comparison between T_{cal} and T_{exp} rise (acrylic resin: Experiment 2)

Table 4.2 Values of λ , α , and δ at iteration steps (acrylic resin: Experiment 2)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.244	1.88	50	16.2	56.3	-16.7	0.03
2 nd	0.196	0.60	31	-6.9	-50.1	-47.7	0.37
3 rd	0.199	0.91	34	-5.1	-24.4	-43.8	0.08
4 th	0.194	0.93	32	-7.5	-22.3	-46.7	0.00
5 th	0.194	0.93	32	-7.4	-22.3	-46.7	0.00



Temperature rise

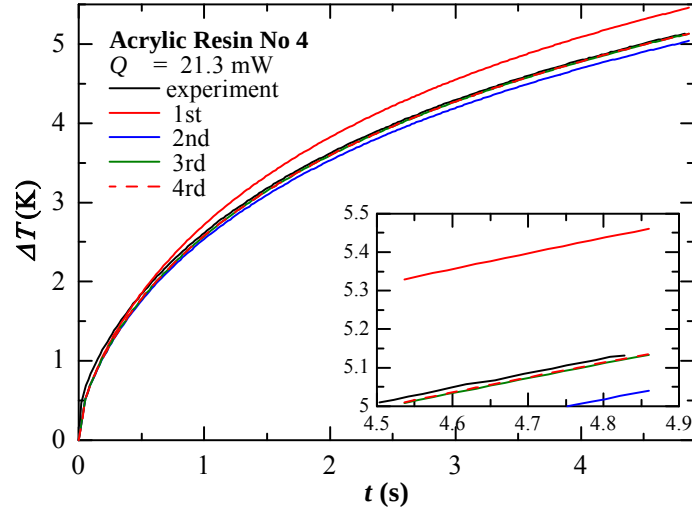


(b) Difference in temperature rise

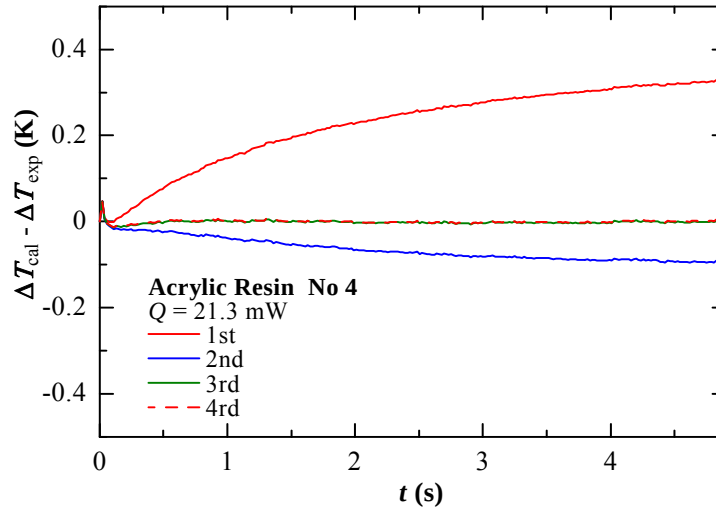
Fig. 4.3 Comparison between T_{cal} and T_{exp} rise (acrylic resin: Experiment 3)

Table 4.3 Values of λ , α , and δ at iteration steps (acrylic resin: Experiment 3)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.195	1.20	50	-7.1	0.00	-16.7	0.28
2 nd	0.181	0.56	50	-14.0	-53.4	-16.7	0.20
3 rd	0.174	0.65	50	-17.2	-46.1	-16.7	0.01
4 th	0.187	0.78	54	-11.0	-35.0	-10.5	0.01



(a) Temperature rise

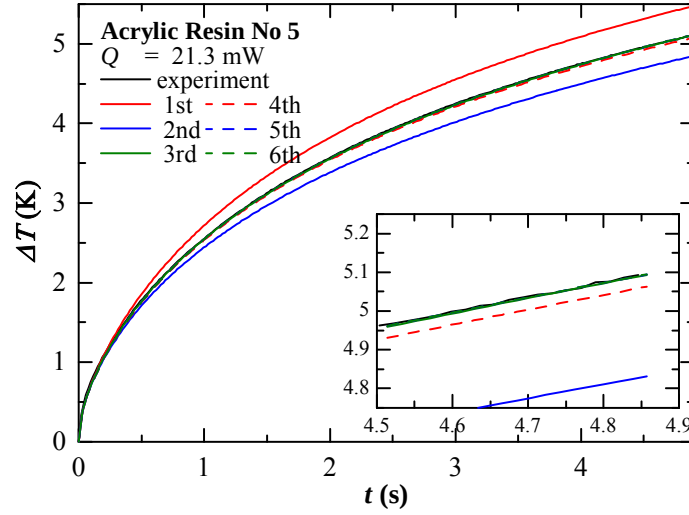


(b) Difference in temperature rise

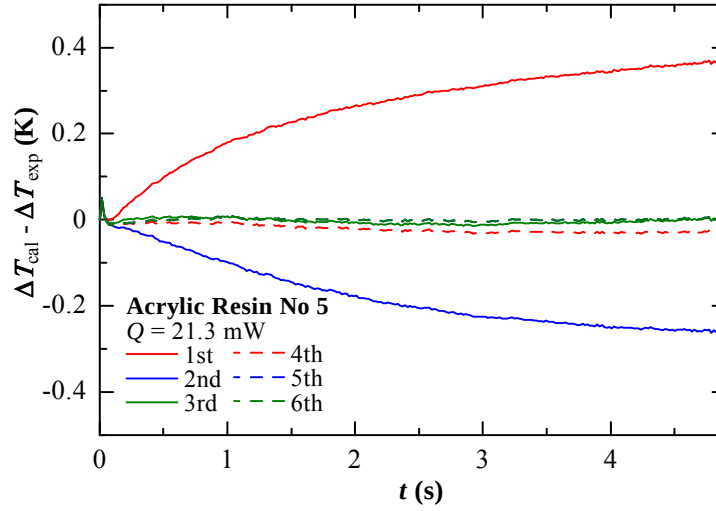
Fig. 4.4 Comparison between T_{cal} and T_{exp} rise (acrylic resin: Experiment 4)

Table 4.4 Values of λ , α , and δ at iteration steps (acrylic resin: Experiment 4)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.195	1.20	50	-7.1	0.0	-16.7	0.24
2 nd	0.192	0.78	50	-8.7	-34.7	-16.7	0.07
3 rd	0.190	0.84	50	-9.3	-30.1	-16.7	0.00
4 th	0.188	0.81	49	-10.5	-32.5	-18.1	0.00



(a) Temperature rise

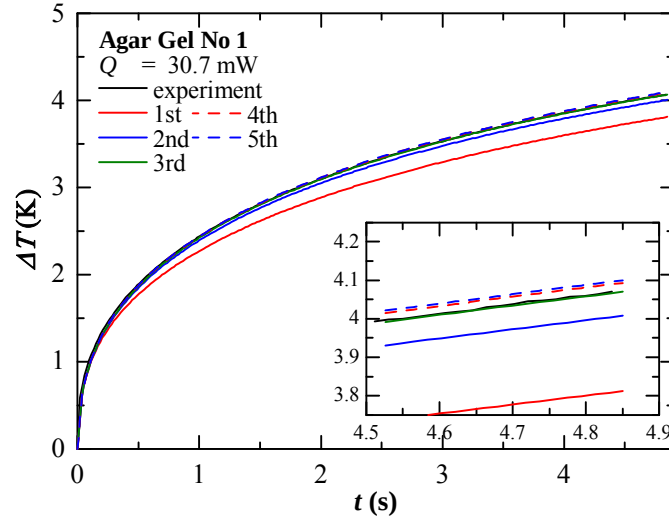


(b) Difference in temperature rise

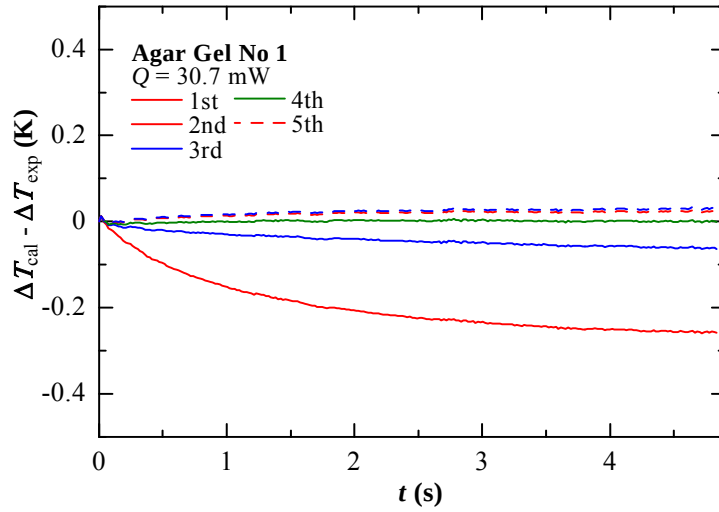
Fig. 4.5 Comparison between T_{cal} and T_{exp} rise (acrylic resin: Experiment 5)

Table 4.5 Values of λ , α , and δ at iteration steps (acrylic resin: Experiment 5)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.195	1.20	50	-7.1	0.0	-16.7	0.27
2 nd	0.180	0.57	50	-14.1	-52.9	-16.7	0.19
3 rd	0.174	0.65	50	-17.3	-45.7	-16.7	0.01
4 th	0.193	0.84	56	-8.3	-29.8	-7.4	0.02
5 th	0.192	0.86	56	-8.6	-28.2	-7.2	0.00
6 th	0.192	0.86	56	-8.6	-28.2	-7.2	0.00



(a) Temperature rise

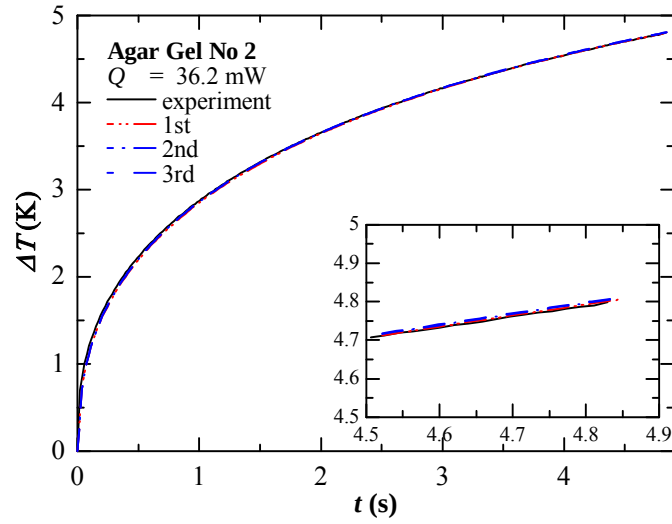


(b) Difference in temperature rise

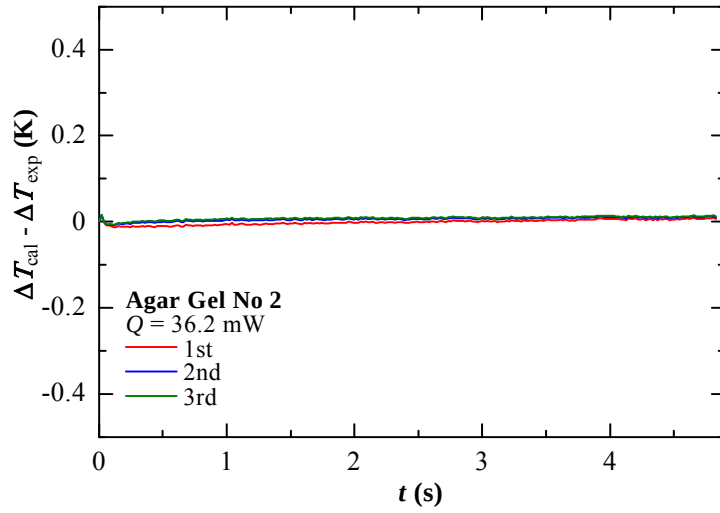
Fig. 4.6 Comparison between T_{cal} and T_{exp} rise (agar gel: Experiment 1)

Table 4.6 Values of λ , α , and δ at iteration steps (agar gel: Experiment 1)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.61	0.94	50	5.3	-29.1	-16.7	0.21
2 nd	0.64	1.32	50	10.2	-0.5	-16.7	0.04
3 rd	0.63	1.33	50	7.9	0.6	-16.7	0.00
4 th	0.62	1.31	69	6.7	-0.8	14.7	0.02
5 th	0.62	1.30	75	6.0	-2.0	25.4	0.02



(a) Temperature rise

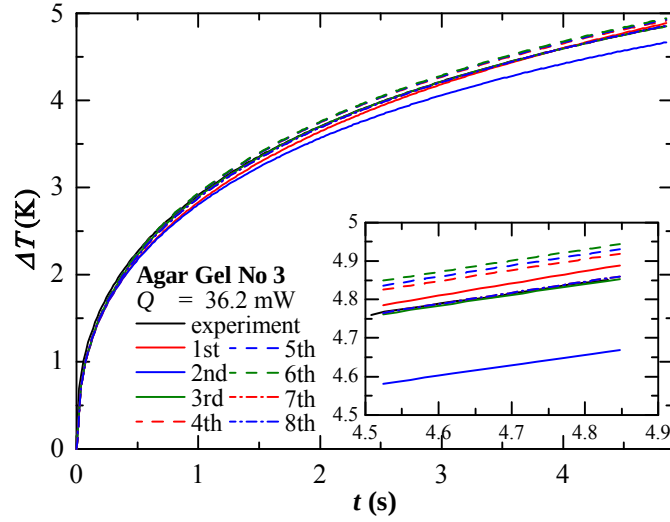


(b) Difference in temperature rise

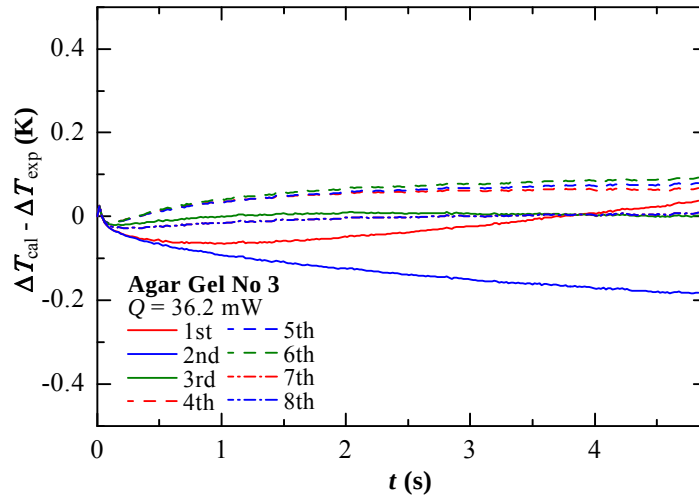
Fig. 4.7 Comparison between T_{cal} and T_{exp} rise (agar gel: Experiment 2)

Table 4.7 Values of λ , α , and δ at iteration steps (agar gel: Experiment 2)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.61	1.25	50	5.3	-5.4	-16.7	0.01
2 nd	0.61	1.25	74	5.4	-5.7	22.5	0.01
3 rd	0.61	1.24	83	5.3	-6.0	37.6	0.01



(a) Temperature rise

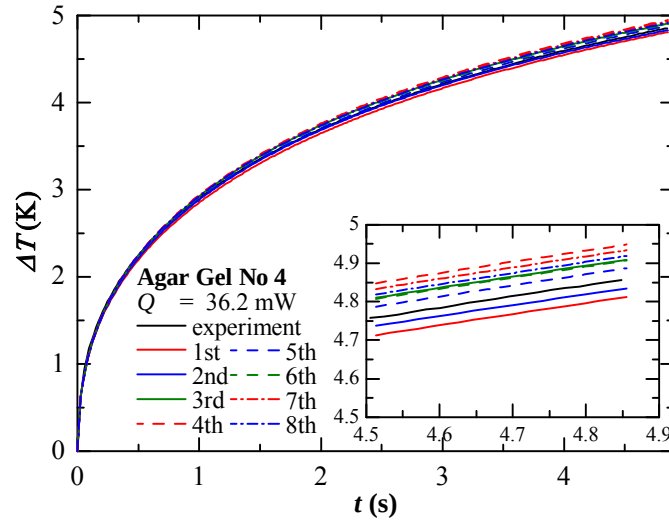


(b) Difference in temperature rise

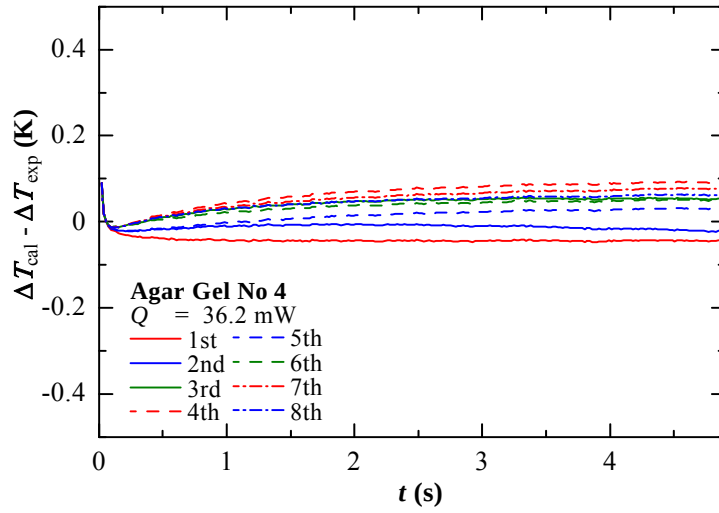
Fig. 4.8 Comparison between T_{cal} and T_{exp} rise (agar gel: Experiment 3)

Table 4.8 Values of λ , α , and δ at iteration steps (agar gel: Experiment 3)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.49	0.75	50	-15.7	-43.2	-16.7	0.04
2 nd	0.66	1.39	50	14.6	5.3	-16.7	0.13
3 rd	0.64	1.47	50	9.9	11.5	-16.7	0.01
4 th	0.62	1.42	155	6.8	7.2	158	0.05
5 th	0.61	1.37	130	4.8	3.9	118	0.06
6 th	0.60	1.33	181	3.1	0.3	202	0.07
7 th	0.61	1.33	36	5.7	0.7	-40.0	0.01
8 th	0.61	1.33	36	5.7	0.7	-40.0	0.01



(a) Temperature rise

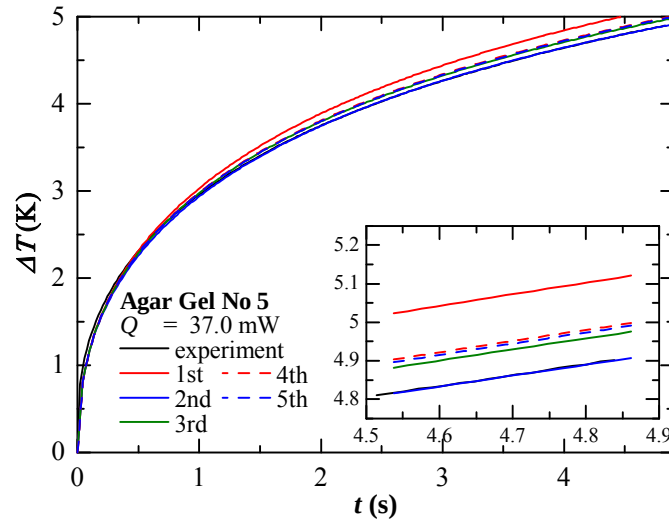


(b) Difference in temperature rise

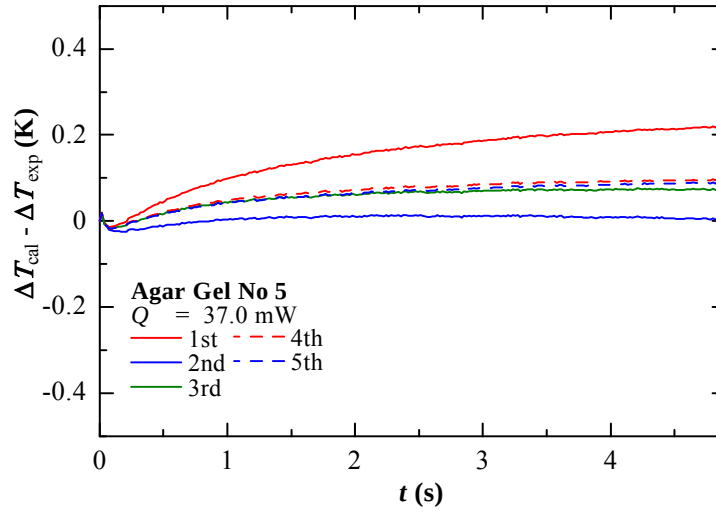
Fig. 4.9 Comparison between T_{cal} and T_{exp} rise (agar gel: Experiment 4)

Table 4.9 Values of λ , α , and δ at iteration steps (agar gel: Experiment 4)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.61	1.25	50	10.3	-0.1	-16.7	0.04
2 nd	0.64	1.47	50	16.0	17.7	-16.7	0.01
3 rd	0.62	1.42	136	12.4	13.4	128	0.05
4 th	0.60	1.36	182	8.94	8.4	204	0.07
5 th	0.61	1.35	37	10.4	7.6	-38.9	0.02
6 th	0.61	1.32	127	9.5	5.6	111	0.04
7 th	0.59	1.28	176	7.2	2.0	194	0.05



(a) Temperature rise

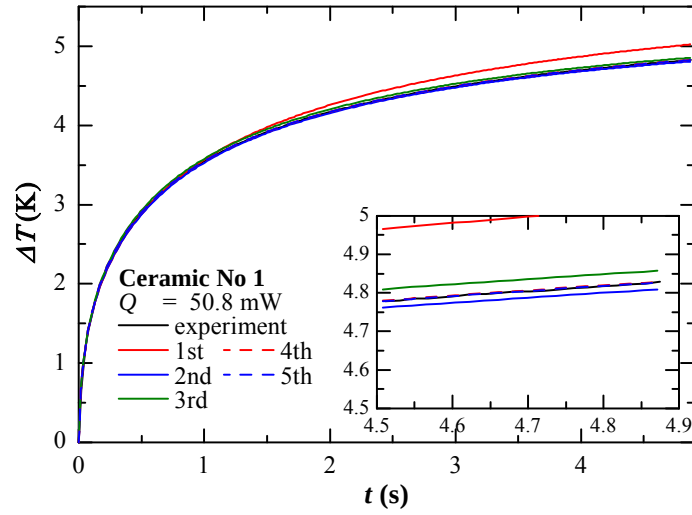


(b) Difference in temperature rise

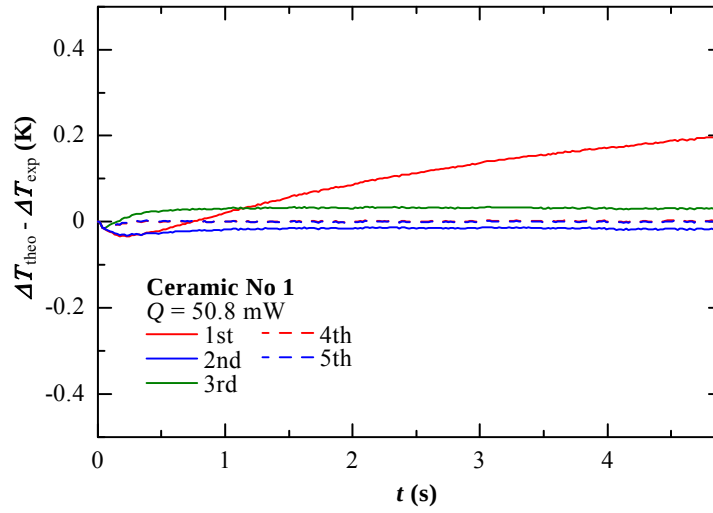
Fig. 4.10 Comparison between T_{cal} and T_{exp} rise (agar gel: Experiment 5)

Table 4.10 Values of λ , α , and δ at iteration steps (agar gel: Experiment 5)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	0.61	1.50	50	5.3	13.5	-16.7	0.16
2 nd	0.66	1.52	50	13.1	14.7	-16.7	0.01
3 rd	0.64	1.46	136	10.2	10.2	127	0.06
4 th	0.62	1.40	140	7.7	5.9	134	0.07
5 th	0.62	1.36	132	6.7	2.8	121	0.07



(a) Temperature rise

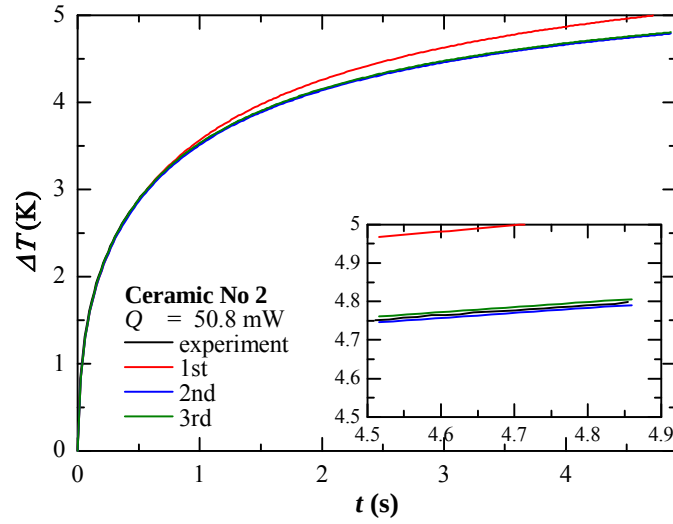


(b) Difference in temperature rise

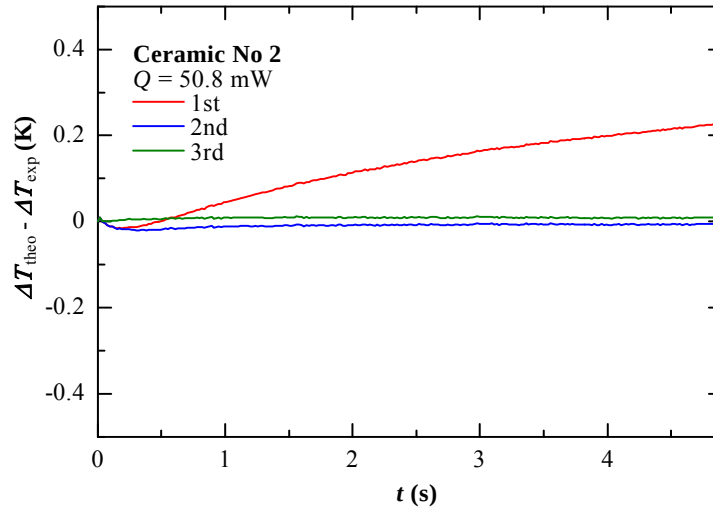
Fig. 4.11 Comparison between T_{cal} and T_{exp} rise (machinable ceramic: Experiment 1)

Table 4.11 Values of λ , α , and δ at iteration steps (machinable ceramic: Experiment 1)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	1.53	6.25	50	-4.7	-20.3	-16.7	0.12
2 nd	1.72	8.22	50	7.3	4.9	-16.7	0.02
3 rd	1.89	7.55	112	18.3	-3.7	87.0	0.03
4 th	1.85	7.43	98	15.5	-5.2	62.5	0.00
5 th	1.86	7.38	100	16.0	-5.9	66.3	0.00



(a) Temperature rise

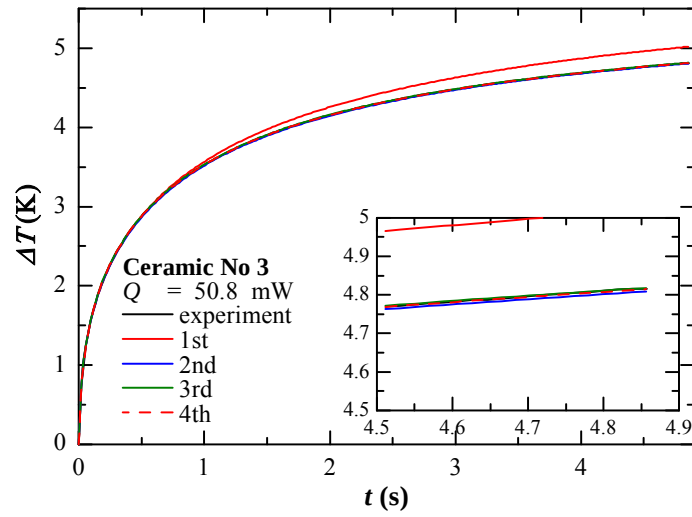


(b) Difference in temperature rise

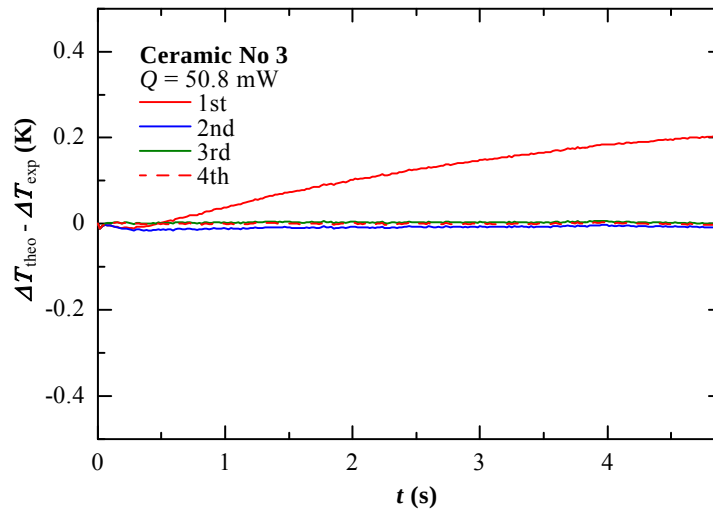
Fig. 4.12 Comparison between T_{cal} and T_{exp} rise (machinable ceramic: Experiment 2)

Table 4.12 Values of λ , α , and δ at iteration steps (machinable ceramic: Experiment 2)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	1.53	6.25	50	-4.7	-20.3	-16.7	0.14
2 nd	1.72	7.99	50	7.4	2.0	-16.7	0.01
3 rd	1.81	7.73	82	13.3	-1.5	36.0	0.01



(a) Temperature rise

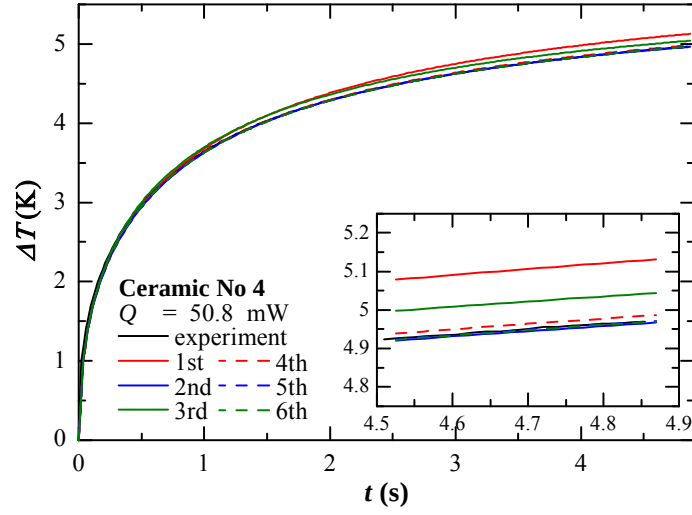


(b) Difference in temperature rise

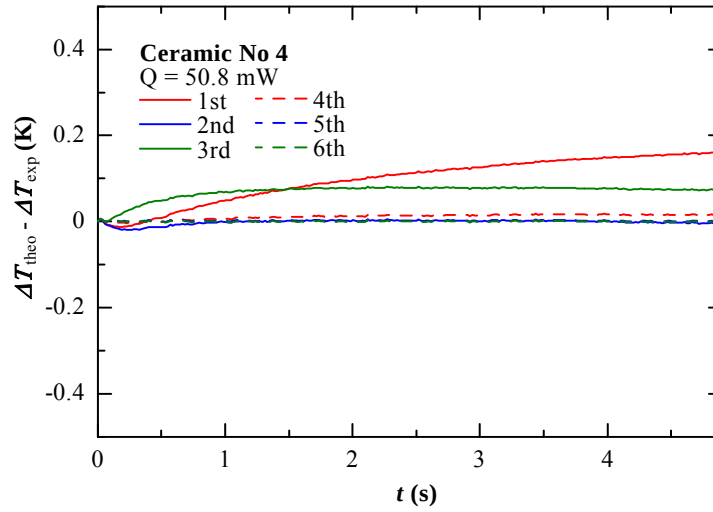
Fig. 4.13 Comparison between T_{cal} and T_{exp} rise (machinable ceramic: Experiment 3)

Table 4.13 Values of λ , α , and δ at iteration steps (machinable ceramic: Experiment 3)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	1.53	6.25	50	-4.7	-20.3	-16.7	0.13
2 nd	1.70	7.87	50	6.4	0.4	-16.7	0.01
3 rd	1.76	7.79	69	10.0	-0.7	15.5	0.00
4 th	1.76	7.74	69	10.0	-1.3	15.5	0.00



(a) Temperature rise

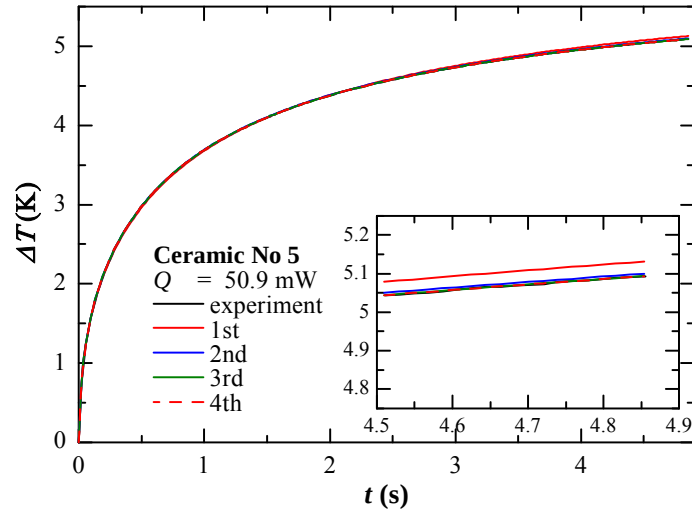


(b) Difference in temperature rise

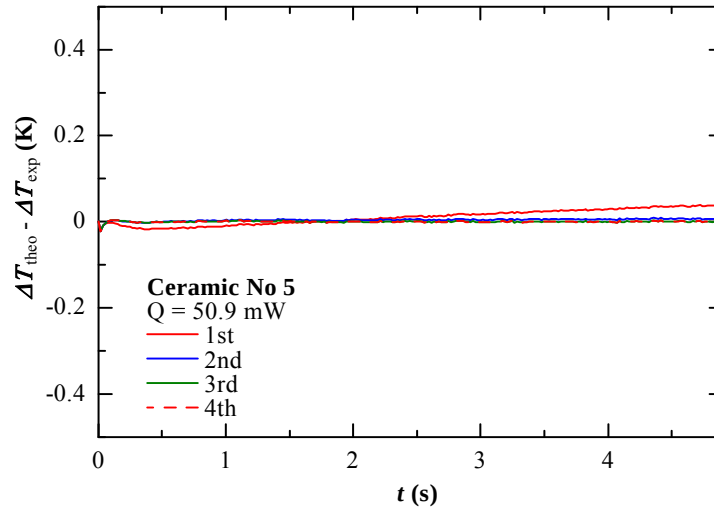
Fig. 4.14 Comparison between T_{cal} and T_{exp} rise (machinable ceramic: Experiment 4)

Table 4.14 Values of λ , α , and δ at iteration steps (machinable ceramic: Experiment 4)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	1.53	7.50	50	-4.7	-4.3	-16.7	0.11
2 nd	1.64	8.57	50	2.6	9.3	-16.7	0.01
3 rd	1.90	7.96	144	18.9	1.5	140.8	0.07
4 th	1.76	7.85	98	10.1	0.2	63.8	0.01
5 th	1.81	7.61	110	13.0	-2.9	84.8	0.00
6 th	1.80	7.60	109	12.7	-3.0	82.7	0.00



(a) Temperature rise

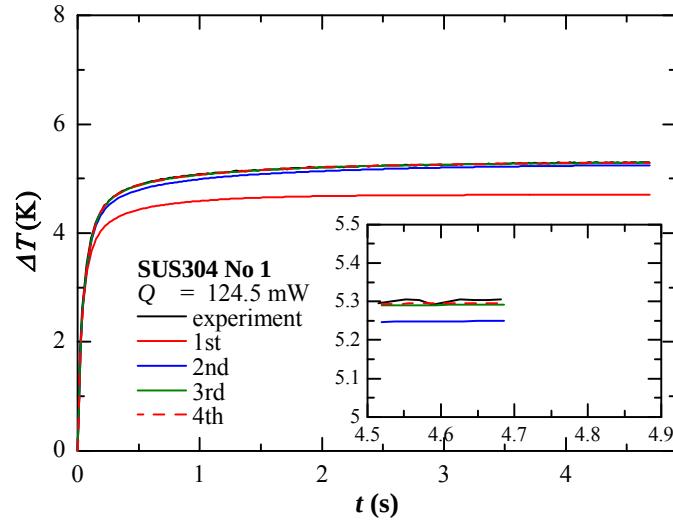


(b) Difference in temperature rise

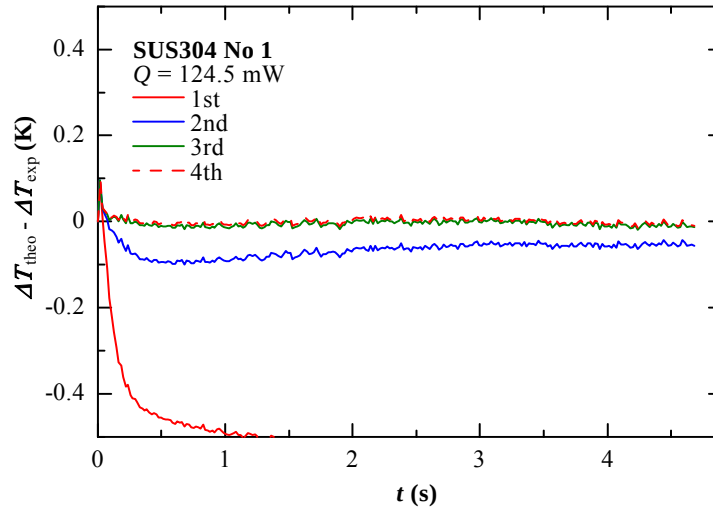
Fig. 4.15 Comparison between T_{cal} and T_{exp} rise (machinable ceramic: Experiment 5)

Table 4.15 Values of λ , α , and δ at iteration steps (machinable ceramic: Experiment 5)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	1.53	7.50	50	-4.7	-4.3	-16.7	0.02
2 nd	1.62	8.19	70	1.1	4.5	17.0	0.01
3 rd	1.64	8.19	75	2.2	4.4	25.1	0.00
4 th	1.63	8.18	75	2.1	4.4	24.6	0.00



(a) Temperature rise

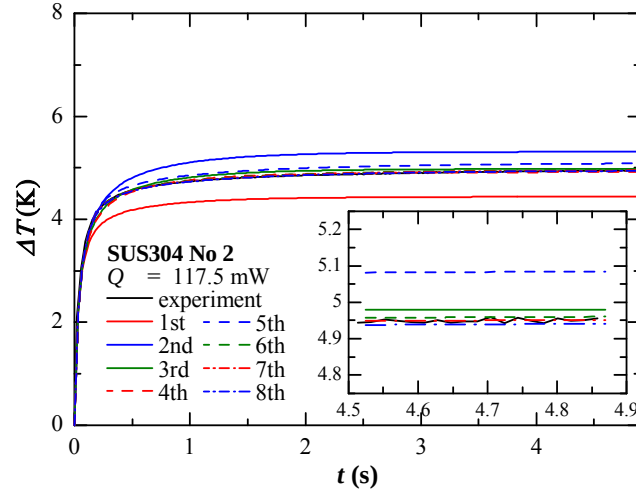


(b) Difference in temperature rise

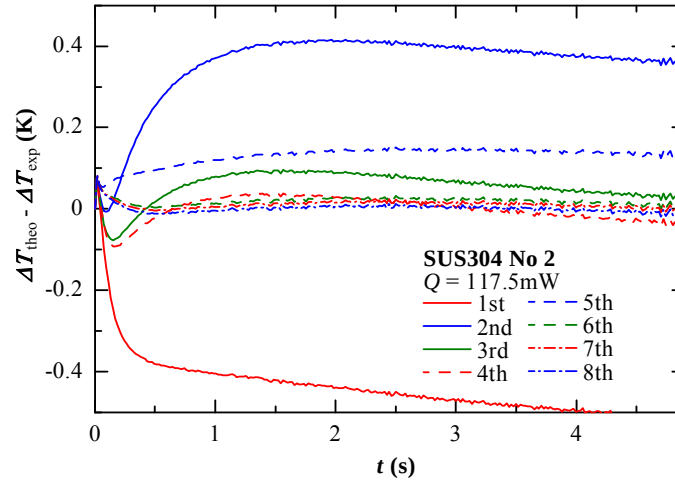
Fig. 4.16 Comparison between T_{cal} and T_{exp} rise (SUS304: Experiment 1)

Table 4.16 Values of λ , α , and δ at iteration steps (SUS304: Experiment 1)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	15.3	46.9	50	-6.6	15.0	-16.7	0.53
2 nd	15.6	17.8	77	-4.4	-56.3	27.8	0.07
3 rd	16.2	22.2	80	-0.7	-45.5	33.0	0.01
4 th	16.2	22.7	80	-0.9	-44.2	33.0	0.01



(a) Temperature rise

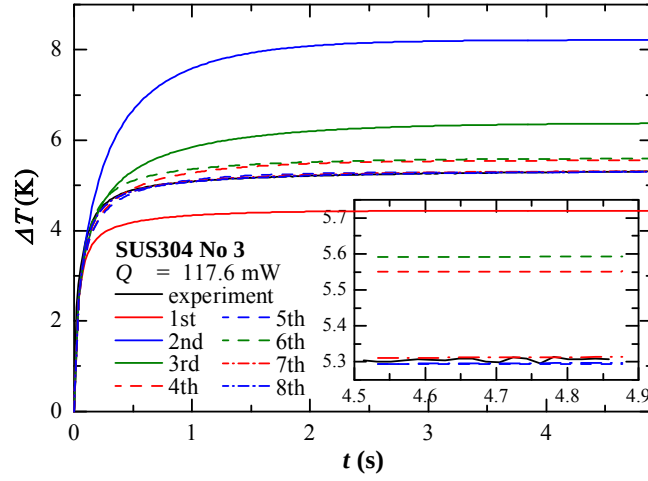


(b) Difference in temperature rise

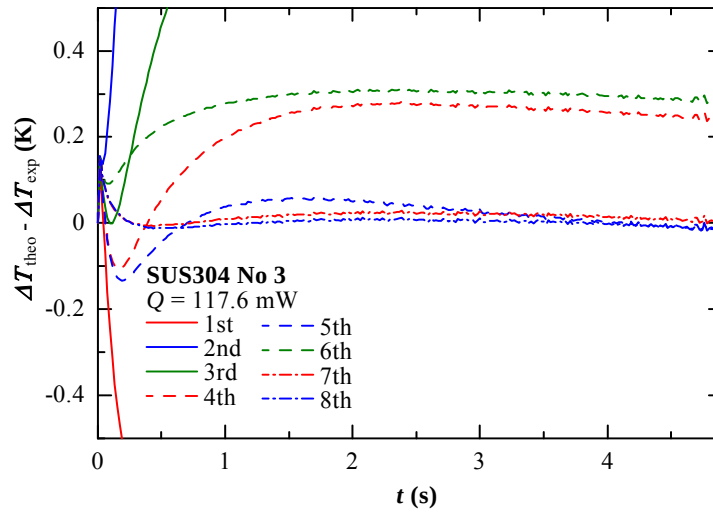
Fig. 4.17 Comparison between T_{cal} and T_{exp} rise (SUS304: Experiment 2)

Table 4.17 Values of λ , α , and δ at iteration steps (SUS304: Experiment 2)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	15.3	46.9	50	-6.6	29.5	-16.7	0.44
2 nd	8.31	50.3	50	-49.1	38.8	-16.7	0.37
3 rd	10.1	49.4	50	-38.4	36.4	-16.7	0.07
4 th	10.5	50.4	50	-35.8	39.4	-16.7	0.03
5 th	14.7	23.2	79	-9.8	-36.0	32.2	0.13
6 th	15.7	23.3	76	-3.6	-35.6	26.4	0.02
7 th	15.7485	23.409	76	-3.5	-35.3	27.1	0.01
8 th	15.8132	23.402	76	-3.1	-35.3	26.5	0.01



(a) Temperature rise

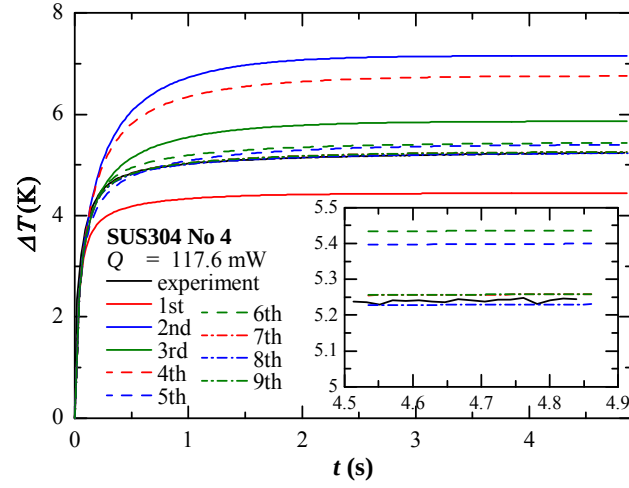


(b) Difference in temperature rise

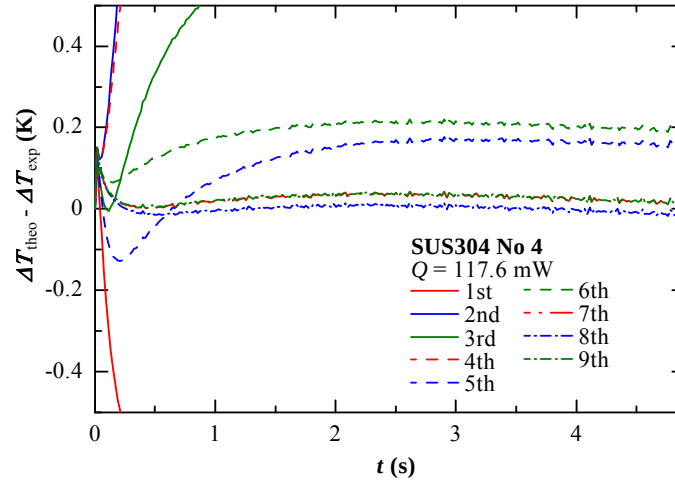
Fig. 4.18 Comparison between T_{cal} and T_{exp} rise (SUS304: Experiment 3)

Table 4.18 Values of λ , α , and δ at iteration steps (SUS304: Experiment 3)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	15.3	46.9	50	-6.6	29.5	-16.7	0.78
2 nd	3.23	65.9	50	-80.2	82.0	-16.7	2.68
3 rd	5.31	33.1	50	-67.5	-8.7	-16.7	0.93
4 th	7.39	44.0	50	-54.8	21.5	-16.7	0.24
5 th	8.41	57.1	50	-48.5	57.8	-16.7	0.04
6 th	12.8	30.4	96	-21.7	-16.1	59.7	0.28
7 th	14.6	25.6	90	-10.5	-29.4	49.6	0.02
8 th	15.1	25.1	91	-7.6	-30.6	50.9	0.02



(a) Temperature rise

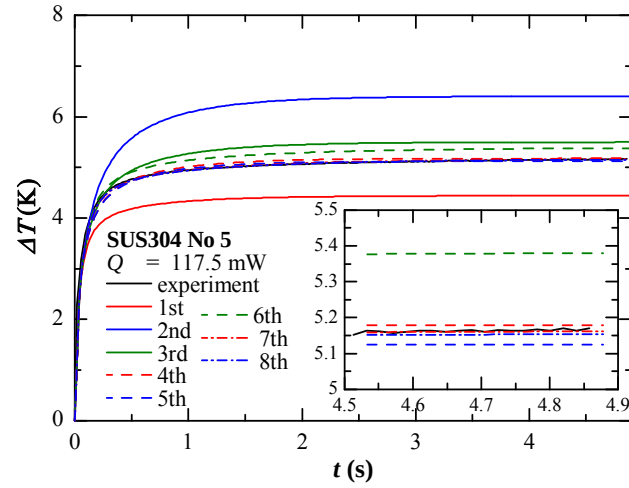


(b) Difference in temperature rise

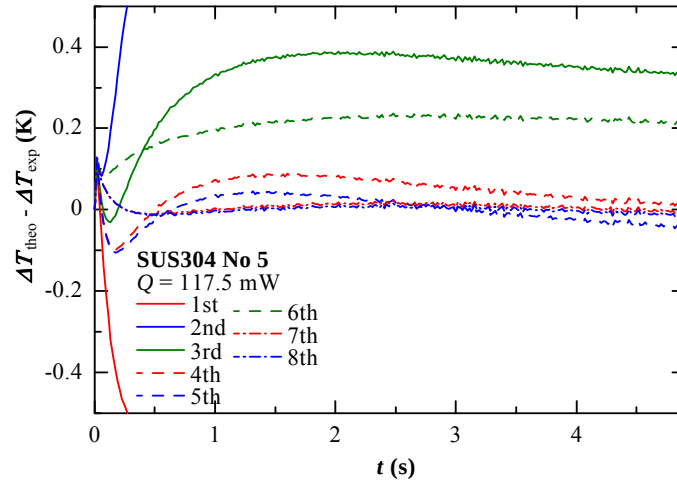
Fig. 4.19 Comparison between T_{cal} and T_{exp} rise (SUS304: Experiment 4)

Table 4.19 Values of λ , α , and δ at iteration steps (SUS304: Experiment 4)

No Iteration	λ (W/mK)	α (10^{-7} m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	15.3	46.9	50	-6.6	29.5	-16.7	0.72
2 nd	4.18	63.2	50	-74.4	74.6	-16.7	1.80
3 rd	6.44	45.7	50	-60.6	26.4	-16.7	0.58
4 th	6.49	44.4	96	-60.2	22.6	59.7	1.41
5 th	8.82	30.8	60	-46.0	-14.8	-0.5	0.14
6 th	12.5	28.5	87	-23.4	-21.4	44.8	0.19
7 th	14.7	25.6	87	-10.2	-29.4	45.4	0.03
8 th	15.1	25.3	87	-7.6	-30.2	45.5	0.02
9 th	15.1	25.3	87	-7.6	-30.2	45.5	0.02



(a) Temperature rise



(b) Difference in temperature rise

Fig. 4.20 Comparison between T_{cal} and T_{exp} rise (SUS304: Experiment 5)

Table 4.20 Values of λ , α , and δ at iteration steps (SUS304: Experiment 5)

No Iteration	λ (W/mK)	α ($10^{-7} \text{ m}^2/\text{s}$)	δ (μm)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	$\frac{\delta - \delta_{ref}}{\delta_{ref}}$ (%)	$SD_{\Delta T}$ (K)
1 st	15.3	46.9	50	-6.6	15.0	-16.7	0.65
2 nd	5.26	59.5	50	-67.8	45.9	-16.7	1.18
3 rd	7.56	50.9	50	-53.7	24.9	-16.7	0.34
4 th	8.95	55.9	50	-45.2	37.2	-16.7	0.06
5 th	9.23	59.3	50	-43.5	45.3	-16.7	0.04
6 th	13.7	25.4	90	-16.4	-37.7	49.1	0.21
7 th	15.0	24.7	84	-8.2	-39.4	39.7	0.02
8 th	15.2	24.7	84	-6.9	-39.3	40.1	0.01

4.2 Summary of Results and Discussion

The summary of finally obtained results for all tested materials is shown in Fig. 4.21 and Table 4.21 with literature values. Five individual measurements were carried out for each material. Even though only one sample was used for a given material, the results depended on each measurement partly because of the difference in preparation and setting. This indicates a need for replication to obtain thermal transport properties with the present method. The final results were obtained after three to nine iterations. The thermal conductivity determined from the average of five replicates was different from the literature value by -8.1% for acrylic resin, 6.2% for agar gel, 10.8% for machinable ceramic, and -5.2% for SUS304. This indicates that the measurement error could be less than 10% irrespective of the material. However, significantly larger error was found in thermal diffusivity for acrylic resin and SUS304; the difference from the literature value was -26.7% for acrylic resin and -35.9% for SUS304, while it was only -0.9% for agar gel and -1.5% for machinable ceramic. This resulted from the fact that the temperature rise is less sensitive to the thermal diffusivity than the thermal conductivity, which has been demonstrated in the discussion with theoretical analysis in Chapter 2.

Considerably small values of $SD_{\Delta T}$, smaller than 0.02 K in most cases and 0.07 K even at the largest case, indicate that the temperature rise obtained with finally determined λ and α agreed well with the measurement as shown in Figs. 4.1-4.20. Even so, the values of λ and α did not always agree well with literature values. In addition, smaller $SD_{\Delta T}$ did not imply better result. This is probably because that modifying λ and α does not significantly influence the difference between measured and calculated temperatures near the start of heating, and the difference here could be the major part of $SD_{\Delta T}$ at the final iteration steps. This could be another reason for the scattering of λ and α that depended on replicates, because the timing of the measurement relative to the actual start of heating was different in each experimental run. This suggests the importance of estimating the time zero to improve present method.

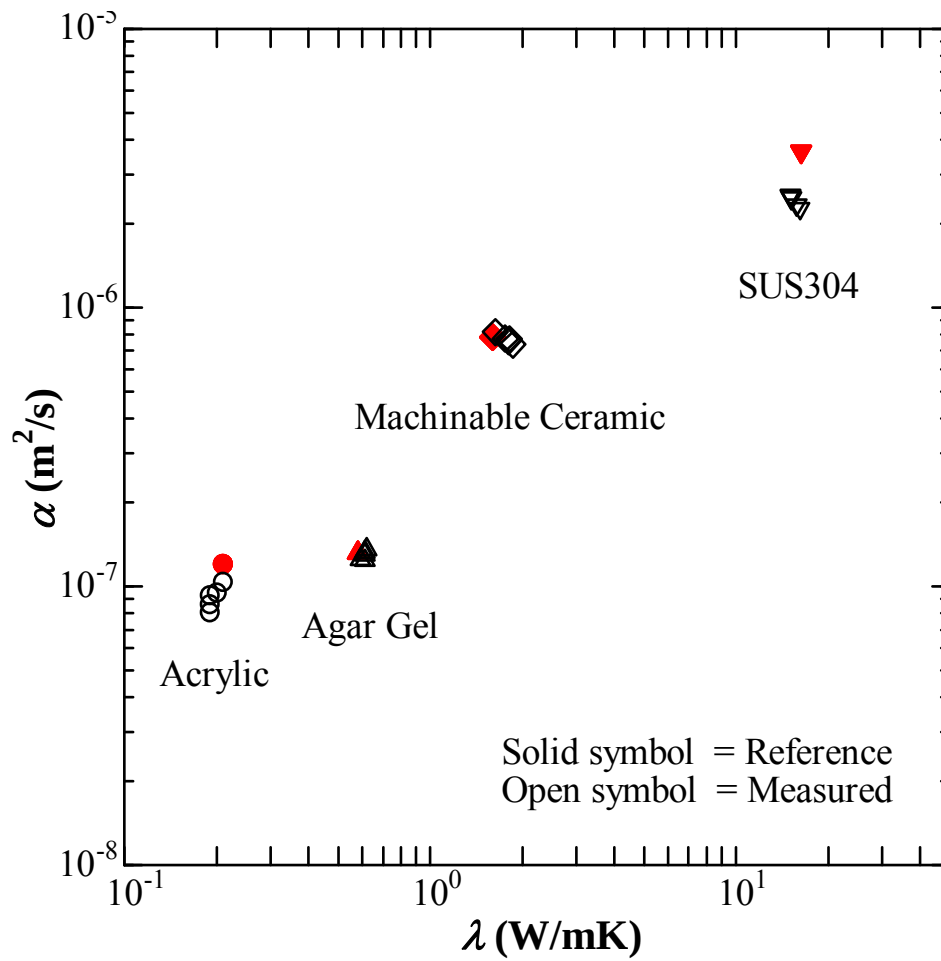


Fig. 4.21 Comparison of measured thermal conductivity and thermal diffusivity with literature values

The measurement error was much larger than those estimated in previous examination with virtual experimental data. One of the reasons is that the measured temperature rise do have some errors in temperatures, whereas the virtual experimental data have taken into account only scattering. A deviation from the assumed situation in the model, uniform thickness of the gel layer for an example, could also happen in the real system. The other major difference is that the exact time for the start of heating has been given in Chapter 2. Therefore significant reduction of

the measurement error might be achieved by introducing a better method for specifying the time at the start of heating.

The thickness of the gel layer δ varied much more than λ and α ; some of them were larger than the twice of the thickness of the spacer, and the others were smaller than the spacer thickness, which could not be the case theoretically. The scattering is the largest in the measurement of agar gel. This is reasonable because the gel has thermal transport properties similar to the agar gel; if thermal transport properties of the gel and the sample are identical, it is theoretically impossible to determine the thickness of the gel. The difficulty of determining δ is due to the fact that the temperature rise was not affected significantly by δ , which has been also demonstrated in our previous study. However, since the value of δ is no concern of the measurement, low sensitivity of the temperature rise to δ is preferable to the present method.

Table 4.21 Measured values of thermal conductivity and thermal diffusivity

Material	Q (mW)	i	λ (W/mK)	α (x 10 ⁻⁷ m ² /s)	δ (μ m)	$\frac{\lambda - \lambda_{ref}}{\lambda_{ref}}$ (%)	$\frac{\alpha - \alpha_{ref}}{\alpha_{ref}}$ (%)	SD _{ΔT} (K)
Acrilic Resin $\lambda_{ref} = 0.210$ W/mK $\alpha_{ref} = 1.20 \times 10^{-7}$ m ² /s [47]	19.9	6	0.204	1.02	37	-3.0	-15.4	0.00
	19.7	5	0.194	0.93	32	-7.4	-22.3	0.00
	21.3	4	0.188	0.81	54	-10.9	-35.0	0.00
	21.3	4	0.188	0.81	49	-10.5	-32.5	0.00
	21.3	6	0.192	0.86	56	-8.6	-28.2	0.01
<i>Average</i>	20.7	5	0.193	0.9	45	-8.1	-26.7	0.00
Agar $\lambda_{ref} = 0.58$ W/mK $\alpha_{ref} = 1.32 \times 10^{-7}$ m ² /s [52]	30.7	5	0.61	1.30	75	6.0	-1.9	0.02
	36.2	3	0.61	1.24	83	5.3	-6.0	0.01
	36.2	8	0.61	1.33	36	5.7	0.7	0.01
	36.2	8	0.59	1.25	184	7.2	0.1	0.05
	37.0	5	0.62	1.36	133	6.7	2.8	0.07
<i>Average</i>	35.3	6	0.61	1.30	102	6.2	-0.9	0.03
Machinable	50.8	5	1.86	7.38	100	16.0	-5.9	0.00
Ceramics $\lambda_{ref} = 1.60$ W/mK $\alpha_{ref} = 7.84 \times 10^{-7}$ m ² /s [47]	50.8	3	1.81	7.72	82	13.3	-1.5	0.01
	50.8	4	1.76	7.74	69	10.0	-1.3	0.00
	50.8	6	1.80	7.60	110	12.7	-3.0	0.00
	50.9	4	1.63	8.18	75	2.1	4.4	0.00
	50.9	4	1.63	8.18	75	2.1	4.4	0.00
<i>Average</i>	50.8	4	1.77	7.73	87	10.8	-1.5	0.00
SUS304 $\lambda_{ref} = 16.3$ W/mK $\alpha_{ref} = 36.2 \times 10^{-7}$ m ² /s [47]	124.5	4	16.2	22.7	80	-0.9	-44.2	0.01
	117.5	8	15.8	23.4	76	-3.1	-35.3	0.01
	117.6	8	15.1	25.1	91	-7.6	-30.6	0.02
	117.6	9	15.1	25.3	87	-7.6	-30.2	0.02
	117.5	9	15.2	24.7	84	-6.9	-39.3	0.02
<i>Average</i>	119.0	8	15.5	24.3	84	-5.2	-35.9	0.01

The final goal of the present research was to develop a method for measuring thermal transport properties of soft materials using a novel stamp sensor which could be used for in-situ non-invasive measurements. The feasibility of the proposed method was examined first with simulated experimental data that were generated based on the simulated temperature rise of the sensor with artificial scattering. Measurement was demonstrated with a model sensor using different kinds of samples with a wide range of thermal transport properties as test materials. The conclusions are as follows:

1. The thermal conductivity, the thermal diffusivity and the thickness of gel layer were determined successfully by the Gauss-Newton algorithm using real experimental data as well as virtual data. The calculated temperature agreed well with the measured temperature rise of the sensor. Finally obtained thermal conductivity for four different materials was different from literature value by ~ -7 to $\sim 11\%$ depending on the material. The average difference in the thermal diffusivity ranged from ~ -1 to $\sim -36\%$.
2. Determination of the time at the start of heating has a significant influence on the results. The method to determine the time which was proposed in the present paper resulted in the reasonable results.

The difference from the ideal situation assumed in the present study, *i.e.* non-uniform thickness of the gel layer and non-adiabatic condition at the top surface of the substrate, for instance, also influences the result. In addition, there are several points to be improved: preparation of the lists for the initial assumption, more effective iteration procedure, and better estimation of the time at start of heating.

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APPENDIX A

COMPUTER PROGRAM

In this research, fortran computer program has been used to do iteration the temperature rising (Appendix A) and fitting method (Appendix B).

```
!      Last change:  SH   27 Feb 2013    8:12 pm
program Temperature Rising
```

```
implicit none
```

```
! definition ---  ( base=b, heater=h, coating=c, gel=g, sample=s ) !
```

```
! definition ---  Size  thickness !
```

```
REAL(8),PARAMETER:: delta_b=156.0D-06, delta_h=52.81D-09, delta_c=150.0D-09, delta=5.0D-5
```

```
! definition ---  Size  Radius of Sensor !
```

```
REAL(8),PARAMETER:: D=1.50D-03
```

```
! definition ---  Thermal Property !
```

```
REAL(8),PARAMETER::      alpha_b=6.80D-07,      alpha_h=1.694D-05,      alpha_c=8.5D-07,
alpha_g=1.50D-7, alpha_s=1.8750D-07
```

```
REAL(8),PARAMETER::      lambda_b=1.10D0,      lambda_h=47.25D0,      lambda_c=1.38D0,
lambda_g=0.61D0, lambda_s=0.2440D0
```

```
! definition ---  Grid expansion coefficient !
```

```
REAL(8),PARAMETER:: k1=1.1D0, k2=1.027D0 ,k3=2.35D0, k4=1.1D0
```

```
! definition ---  Heating condition !
```

```
!REAL(8),PARAMETER::time_heating=5.0D0, Q=6.24988D10, T0=0.0D00, DELF0=1.0D-6
REAL(8),PARAMETER::time_heating=5.0D0, QmW=19.6742480D0 , T0=0.0D00, DELF0=1.0D-6
INTEGER::step=100 , step2=66666
```

```
! definition ---  the number of mesh !
```

```
REAL(8),PARAMETER::Ng=10.0      !GEL
REAL(8),PARAMETER::Ns=200.0     !SAMPLE
REAL(8),PARAMETER::Nb=30.0      !BASE
REAL(8),PARAMETER::Nrr=1000.0   !r
INTEGER,PARAMETER::ih=100       !r heater
```

```
REAL(8)::z_c,LL,MM,delz_min,u,DD
```

```
REAL(8)::THETA_AVEE
```

```
REAL(8)::LL_b,LL_s,LL_c,LL_h
```

```
REAL(8)::AA_b,AA_s,AA_c,AA_h
```

```
REAL(8)::C01,C02,C03,C04,C05,C06,C07,C08,C09,C010,Cb6
```

```
REAL(8)::C011,C012,C013,C014,C015,C016,C017,C018
```

```
REAL(8)::Tdepth,Q
```

```
INTEGER::js,jg,jb,ih2,jc
```

```

REAL(8)::z,X,H,Nrmin
REAL(8)::delz
REAL(8)::r,CN,OI,ITER
!REAL(8),DIMENSION(:),ALLOCATABLE::Nz
!REAL(8),DIMENSION(:),ALLOCATABLE::NZZ
REAL(8),DIMENSION(:),ALLOCATABLE::C1,C2
REAL(8),DIMENSION(:),ALLOCATABLE::C3,C4
REAL(8),DIMENSION(:,:),ALLOCATABLE::C_0
REAL(8),DIMENSION(:,:),ALLOCATABLE::C_1,C_2,C_3,C_4,C_5,ER
REAL(8),DIMENSION(:),ALLOCATABLE::NdelR,NR,Nr_cen
REAL(8),DIMENSION(:),ALLOCATABLE::Nz,Nz_cen
REAL(8),DIMENSION(:),ALLOCATABLE::Ndelz
REAL(8),DIMENSION(:),ALLOCATABLE::Nzg,Nzb

!REAL(8),DIMENSION(0:200)::NdelR,NR
!REAL(8),DIMENSION(jb:js)::Nz,Nz_cen
!REAL(8),DIMENSION(jb+1:js-1)::Ndelz
!REAL(8),DIMENSION(1:Ng)::Nzg

INTEGER::i,j,A,B,C,E,N,M,IER1,IER2,IER3,IER4,kk,kkk
INTEGER::il,jl,j_l,jcb,j_lo,K,O!,OO
REAL(8)::delta_con,delz_c,p,del_MIN,S,jcb_s,L,F,FF,THETA_0,TIME_S,Nrlim,Nzlim

REAL(8)::sos,pop

!non-demention
REAL(8)::Ndelta,Ndelta_c,Ndelta_h,Ndelta_b,Ndelta_con
REAL(8)::Nz_c,Ndelz_min,Ndelz_min_o,DEL_T_AVEE,DEL_T_AVEE2

REAL(8),dimension(:),ALLOCATABLE ::THETA_HEATER,T_HEATER,DEL_T,THETA_,DEL_T
2
REAL(8),DIMENSION(:,:),ALLOCATABLE::THETA,T,THETA_OLD
!-----
!initial parameter
!-----

!non-demention
Ndelta=delta/D
Ndelta_c=delta_c/D
Ndelta_h=delta_h/D
Ndelta_b=delta_b/D

LL_b=lambda_b/lambda_g
LL_s=lambda_s/lambda_g
LL_c=lambda_c/lambda_g
LL_h=lambda_h/lambda_g

AA_b=alpha_b/alpha_g
AA_s=alpha_s/alpha_g
AA_c=alpha_c/alpha_g
AA_h=alpha_h/alpha_g

!Initial Temperatures(Non-dimension)
THETA_0=(T0-T0)/(Q*(D**2)/LAMBDA_G)

```

!Initial Temperature(Non-dimension)

```
OPEN(UNIT=1,FILE="1Nz.txt",STATUS="UNKNOWN")
OPEN(UNIT=2,FILE="2Ndelz.txt",STATUS="UNKNOWN")
OPEN(UNIT=3,FILE="3Nz_cen.txt",STATUS="UNKNOWN")
OPEN(UNIT=4,FILE="4Nr.txt",STATUS="UNKNOWN")
OPEN(UNIT=5,FILE="5Ndelr.txt",STATUS="UNKNOWN")
OPEN(UNIT=7,FILE="7Nr_cen.txt",STATUS="UNKNOWN")
OPEN(UNIT=8,FILE="8DT_ave.txt",STATUS="UNKNOWN")
OPEN(UNIT=9,FILE="9T_r_center.txt",STATUS="UNKNOWN")
OPEN(UNIT=10,FILE="1019_10.csv",STATUS="UNKNOWN")
OPEN(UNIT=11,FILE="11DT_ave2.txt",STATUS="UNKNOWN")
OPEN(UNIT=12,FILE="12T_z_center.txt",STATUS="UNKNOWN")
OPEN(UNIT=13,FILE="13DTHETA_ave.txt",STATUS="UNKNOWN")
OPEN(UNIT=14,FILE="14T_distribution.csv",STATUS="UNKNOWN")
OPEN(UNIT=15,FILE="15DT_ave.txt",STATUS="UNKNOWN")
```

```
!OPEN(UNIT=16,FILE="1006_11.txt",STATUS="UNKNOWN")
!OPEN(UNIT=12,FILE="1019_51.csv",STATUS="UNKNOWN")
!OPEN(UNIT=13,FILE="1125_52.csv",STATUS="UNKNOWN")
!OPEN(UNIT=14,FILE="1125_63_8.txt",STATUS="UNKNOWN")
```

```
!!! Calculate (HeatingPower/Volume) Q from QmW
Q = QmW / ((D**2.0D0)*3.141592653589793238D0*delta_h)/1000.0D0
WRITE(*,*) 'Q=', Q
```

!!!!Coordinate of End point of mesh!!!!

```
jb=Nb
jg=Nb+Ng
js=Nb+Ng+Ns+2
jc=-3
```

```
ALLOCATE(Ndelz(jc:js-1))
ALLOCATE(Nz(jc-1:js))
ALLOCATE(Nz_cen(jc:js-1))
ALLOCATE(Nzg(1:Ng))
ALLOCATE(Nzb(1:Nb))
```

```
!!!size of (Heater & Coating) part
Ndelz(0)=Ndelta_h !DZ(0)
DO j=-1,jc,-1
    Ndelz(j)=Ndelta_c/(ABS(jc)) !DZ(-1)~
END DO
```

```
!!! Coordinate of (0 & Heater & Coating) part
Nz(0)=0.0 !Z(0)
Nz(1)=Nz(0)+Ndelz(0)/2.0D0 !Z(1)
Nz(-1)=Nz(0)-Ndelz(0)/2.0D0 !Z(-1)
DO j=-2,jc-1,-1
    Nz(j)=Nz(j+1)-Ndelz(j+1) !Z(-2)~
END DO
!Nz(-1)=Nz(0)-Ndelz(0)/2.0D0 !Z(-1)
!Nz(-2)=Nz(-1)-Ndelz(-1) !Z(-2)
```

!!!! BASE PART !!!!

```

!Nz(-1)=-Ndelta_h*0.5D0                                !Z(-1)

DO kkk=1,Nb
  Nzb(kkk)=Ndelta_b*(ABS(kkk)/ABS(Nb))**k3
END DO

DO j=2,jb+1
  Nz(j)=Nz(1)+Nzb(j-1)                                !Z(2)~Z(31)
END DO

      DO j=1,jb
        Ndelz(j)=ABS(Nz(j+1)-Nz(j))      !DZ(1)~DZ(30)
      END DO

!!!! GEL PART !!!!
!!! Coordinate of Gel part
!DO kk=1,Ng
!  Nzg(kk)=Ndelta*(kk/Ng)**k1
!END DO
      DO j=jb+1,jg                                !jb+1~jg
        Ndelz(j)=Ndelta/Ng                                !DZ(31)~DZ(40)
      END DO

DO j=jb+2,jg+1                                !jb+2 ~ jg+1
  Nz(j)=Nz(j-1)+Ndelz(j-1)
  !Nz(j)=Nz(jb+1)+Nzg(j-(jb+1))                                !Z(32) ~ Z(41)
END DO

!!! size of Gel part
!  DO j=jb+1,jg                                !jb+1~jg
!    Ndelz(j)=Nz(j+1)-Nz(j)                                !DZ(31)~DZ(40)
!  END DO

!!!! SAMPLE PART !!!!
!!! size of First mesh of Sample
Ndelz(jg+1)=Ndelz(jg)                                !DZ(41)=DZ(40)

!!! size of Sample part
DO j=jg+2,js-1
  Ndelz(j)=Ndelz(jg+1)*(k2**(j-(jg+1)))      !DZ(42)~DZ(241)
END DO

!!! Coordinate of Sample part
DO j=jg+2,js
  Nz(j)=Nz(j-1)+Ndelz(j-1)                                !Z(42)~Z(112)or
more
END DO

!DETERMINATION ~IH~ (+z Thickness of temperature penetration)
Tdepth=4.0D-3/D
IF (Tdepth<=((12.0D0*alpha_s*time_heating)**0.5D0)/D + Nz(jg+1)) THEN
  Tdepth=((12.0D0*alpha_s*time_heating)**0.5D0)/D + Nz(jg+1)
  WRITE(*,*) 'Analyzed Area needs more than 4mm!'
END IF

DO j=1,10000
  Nzlim=Nz(j)
  !write (*,*) 'j=',j, 'Nzlim=',Nzlim

```

```

        IF (Nzlim>=Tdepth) THEN
            write (*,*) 'Z-area=',Nzlim*D*1.0D3,'mm'
            write (*,*) 'OK!'
        EXIT
        !GOTO 30
    END IF
END DO
20    jl=j
    WRITE(*,*) 'jl=',jl

!!!!Coordinate of center point of mesh!!!!
    Nz_cen(0)=Nz(0)                                !Z_cen(0)

    DO j=1,jl-1
        Nz_cen(j)=Nz(j)+(Ndelz(j)/2.0)              !Z_cen(1)~Z_cen(111)
    END DO

    Nz_cen(-1)=Nz(-1)-(Ndelz(-1)/2.0)
    DO j=-2,jc,-1
        Nz_cen(j)=Nz(j)-(Ndelz(j)/2.0)              !Z_cen(-1)~Z_cen(-30)
    END DO

    !!! Check Nz & Ndelz
    WRITE(1,*) 'Nz'
    DO j=jc-1,jl
        WRITE (1,*)  j,Nz(j)
    END DO

    WRITE(2,*) 'Ndelz'
    DO j=jc,jl-1
        WRITE (2,*)  j,Ndelz(j)
    END DO

    WRITE(3,*) 'Nz_center'
    DO j=jc,jl-1
        WRITE (3,*)  j,Nz_cen(j)
    END DO

!!!!!r determine minimum size of mesh

    write (*,*) 'ih=',ih
    !ih2=ih*2.0D0
    !write (*,*) 'ih2=',ih2
    Nrmin=1.0D0/(ih+0.50D0)                            !!!caution

!r determine size of mesh
    ALLOCATE(NdelR(0:Nrr-1),NR(0:Nrr),Nr_cen(0:Nrr-1))

    !NdelR(0)=Nrmin*0.5D0
    DO i=0,ih,!ih2
        NdelR(i)=Nrmin
    END DO

    DO i=ih+1,Nrr,!ih2+1,Nrr
        NdelR(i)=Nrmin*k4**(i-ih)!ih2)
    END DO

    NR(0)=0.0
    NR(1)=NR(0)+NdelR(0)*0.5D0
    DO i=2,Nrr

```

```

        NR(i)=NR(i-1)+NdelR(i-1)
    END DO

    !write (*,*) '12at=',((12.0D0*alpha_c*time_heating)**0.5D0)/D
    !write (*,*) '12at+1=',((12.0D0*alpha_c*time_heating)**0.5D0)/D+1

!DETERMINATION ~IH,IL~ (+r Thickness of temperature penetration)
    DO i=1,10000
        Nrlim=NR(i)
        !write (*,*) 'i=',i, 'Nrlim=',Nrlim, 'NR(i)=',NR(i)
        IF (Nrlim>=((12.0D0*alpha_c*time_heating)**0.5D0)/D+1) THEN

            !write (*,*) 'OK!'
            EXIT
            !GOTO 30
        END IF
    END DO

30    il=i-8
!    il=143

    !write (*,*) 'NR(il)',NR(il)
    write (*,*) 'R-area=',NR(il)*D*1.0D3,'mm'
    write (*,*) 'OK!'
    write (*,*) 'il=',il

!!!!Coordinate of center point of mesh!!!!
    Nr_cen(0)=0.0

    DO i=1,il-1
        Nr_cen(i)=NR(i)+NdelR(i)*0.5D0
    END DO

!!! Check Nr & Ndelr
    WRITE(4,*) 'Nr'
    DO i=0,il
        WRITE (4,*) i,Nr(i)
    END DO

    WRITE(5,*) 'Ndelr'
    DO i=0,il-1
        WRITE (5,*) i,Ndelr(i)
    END DO

    WRITE(7,*) 'Nr_center'
    DO i=0,il-1
        WRITE (7,*) i,Nr_cen(i)
    END DO

!Temperatures
    ALLOCATE(T(0:il-1,jc:jl-1),T_HEATER(0:ih),STAT=IER1)

    ALLOCATE(THETA(0:il-1,jc:jl-1),THETA_OLD(0:il-1,jc:jl-1),THETA_HEATER(0:ih),THETA_(0:
ih),STAT=IER2)
    ALLOCATE(DEL_T(0:ih))
    !Coefficients

    ALLOCATE(C1(0:il-2),C2(0:il-2),C3(jc:jl-2),C4(jc:jl-2),C_0(0:il-2,jc:jl-2),C_1(0:il-2,jc:jl-2),STAT=I
ER3)

```

```

ALLOCATE(C_2(0:il-2,jc:jl-2),C_3(0:il-2,jc:jl-2),C_4(0:il-2,jc:jl-2),C_5(0:il-2,jc:jl-2),STAT=IER4)
IF (IER2==0.AND.IER3==0.AND.IER4==0) THEN
  WRITE (*,*) 'IER=',IER2,IER3,IER4;          !If IER=0 , Allocation succeeds
  WRITE(*,*) 'ALLOCATE SUCCESS!'
END if

```

```

!MASK      WRITE(*,*) '1 CLEAR'

```

```

!Set-up Coefficients

```

```

!constant number

```

```

C01=1.0D0/DELF0
C02=1.0D0/(DELF0*AA_b)
C03=1.0D0/(DELF0*AA_h)
C04=1.0D0/(DELF0*AA_s)
C05=1.0D0/(DELF0*AA_c)
C07=(2.0D0*LL_b)/(LL_h+LL_b)
C08=(2.0D0*LL_h)/(LL_h+LL_b)
C09=(2.0D0*LL_c)/(LL_c+LL_h)
C010=(2.0D0*LL_h)/(LL_h+LL_c)
C011=(2.0D0*LL_b)/(LL_c+LL_b)
C012=(2.0D0*LL_c)/(LL_c+LL_b)
C013=1.0D0/LL_h
C014=4.0D0/(Ndelr(0)**2.0D0)
C015=2.0D0/(LL_b+1.0D0)
C016=(2.0D0*LL_b)/(LL_b+1.0D0)
!C015=2.0D0/(LL_c+1.0D0)
!C016=(2.0D0*LL_c)/(LL_c+1.0D0)
C017=2.0D0/(LL_s+1.0D0)
C018=(2.0D0*LL_s)/(LL_s+1.0D0)
Cb6=2.0D0/((Ndelz(jb))**2)

```

```

!write (16,*)C01,C02,C03,C04,C05,C07,C08,C09,&
!      C010,C011,C012,C013,C014,C015,C016,C017,C018,Cb6

```

```

C1=0.0D0
C2=0.0D0
C3=0.0D0
C4=0.0D0

```

```

Ndelr(-1)=0.0D0

```

```

do i=0,il-2

```

```

  C1(i)=2.0D0 * (Nr(i)/(Nr(i+1)**2.0D0-Nr(i)**2.0D0)) * (2.0D0/(Ndelr(i-1)+Ndelr(i)))
  C2(i)=2.0D0 * (Nr(i+1)/(Nr(i+1)**2.0D0-Nr(i)**2.0D0)) * (2.0D0/(Ndelr(i)+Ndelr(i+1)))

```

```

end do

```

```

Ndelz(jc-1)=0.0D0

```

```

do j=jc,jl-2

```

```

  C3(j)=2.0D0/(Ndelz(j)*(Ndelz(j-1)+Ndelz(j)))
  C4(j)=2.0D0/(Ndelz(j)*(Ndelz(j)+Ndelz(j+1)))

```

```

end do

```

```

!MASK      WRITE(*,*) '2 CLEAR'

```

```

!Heat1

```

```

  i=0

```

```

  j=0

```

```

  C_0(i,j)=C03+C014+C09*C3(j)+C07*C4(j)

```

```

  C_1(i,j)=0          !Q1

```

```

  C_2(i,j)=C014       !Q2

```

```

C_3(i,j)=C3(j)*C09          !Q3
C_4(i,j)=C4(j)*C07          !Q4
C_5(i,j)=C03                !T(n-1)
!C_0(i,j)=C_1(i,j)+C_2(i,j)+C_3(i,j)+C_4(i,j)+C_5(i,j)
!Heat2
  do i=1,ih-1
    j=0
    C_0(i,j)=C03+C1(i)+C2(i)+C09*C3(j)+C07*C4(j)
    C_1(i,j)=C1(i)
    C_2(i,j)=C2(i)
    C_3(i,j)=C3(j)*C09
    C_4(i,j)=C4(j)*C07
    C_5(i,j)=C03
  end do
!Heat3
  i=ih
  j=0
  C_0(i,j)=C03+C1(i)+C09*C2(i)+C09*C3(j)+C07*C4(j)
  C_1(i,j)=C1(i)
  C_2(i,j)=C09*C2(i)
  C_3(i,j)=C3(j)*C09
  C_4(i,j)=C4(j)*C07
  C_5(i,j)=C03
!Cover1
  i=ih+1
  j=0
  C_0(i,j)=C05+C010*C1(i)+C2(i)+C3(j)+C4(j)*C011
  !C_0(i,j)=C05+C07*C1(i)+C2(i)+C011*C3(j)+C4(j)
  C_1(i,j)=C010*C1(i)
  !C_1(i,j)=C07*C1(i)
  C_2(i,j)=C2(i)
  C_3(i,j)=C3(j)
  C_4(i,j)=C4(j)*C011
  C_5(i,j)=C05
!Cover2
  DO i=ih+2,il-2
    j=0
    C_0(i,j)=C05+C1(i)+C2(i)+C3(j)+C4(j)*C011
    C_1(i,j)=C1(i)
    C_2(i,j)=C2(i)
    C_3(i,j)=C3(j)
    C_4(i,j)=C4(j)*C011
    C_5(i,j)=C05
  END DO
!Cover3
  i=0
  j=-1
  C_0(i,j)=C05+C014+C3(j)+C010*C4(j)
  C_1(i,j)=0
  C_2(i,j)=C014
  C_3(i,j)=C3(j)
  C_4(i,j)=C010*C4(j)
  C_5(i,j)=C05
!Cover4
  DO i=1,ih
    j=-1
    C_0(i,j)=C05+C1(i)+C2(i)+C3(j)+C010*C4(j)
    C_1(i,j)=C1(i)
    C_2(i,j)=C2(i)
    C_3(i,j)=C3(j)

```

```

C_4(i,j)=C010*C4(j)
C_5(i,j)=C05
END do
!Cover5
do i=ih+1,il-2
j=-1
C_0(i,j)=C05+C1(i)+C2(i)+C3(j)+C4(j)
C_1(i,j)=C1(i)
C_2(i,j)=C2(i)
C_3(i,j)=C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C05
END DO
!Cover6
DO j=-2,jc+1,-1
i=0
C_0(i,j)=C05+C2(i)+C3(j)+C4(j)
C_1(i,j)=0
C_2(i,j)=C2(i)
C_3(i,j)=C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C05
END DO
!Cover7
DO i=1,il-2
DO j=-2,jc+1,-1
C_0(i,j)=C05+C1(i)+C2(i)+C3(j)+C4(j)
C_1(i,j)=C1(i)
C_2(i,j)=C2(i)
C_3(i,j)=C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C05
END DO
END DO
!Cover8
j=jc
i=0
C_0(i,j)=C05+C2(i)+C4(j)
C_1(i,j)=0
C_2(i,j)=C2(i)
C_3(i,j)=0
C_4(i,j)=C4(j)
C_5(i,j)=C05
!Cover9
DO i=1,il-2
j=jc
C_0(i,j)=C05+C1(i)+C2(i)+C4(j)
C_1(i,j)=C1(i)
C_2(i,j)=C2(i)
C_3(i,j)=0
C_4(i,j)=C4(j)
C_5(i,j)=C05
END DO

!base1
i=0
j=1
C_0(i,j)=C02+C014+C08*C3(j)+C4(j)
C_1(i,j)=0
C_2(i,j)=C014

```

```

C_3(i,j)=C08*C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C02

!base2
do i=1,ih
  j=1
  C_0(i,j)=C02+C1(i)+C2(i)+C08*C3(j)+C4(j)
  C_1(i,j)=C1(i)
  C_2(i,j)=C2(i)
  C_3(i,j)=C08*C3(j)
  C_4(i,j)=C4(j)
  C_5(i,j)=C02
end do

!base3
do i=ih+1,il-2
  j=1
  C_0(i,j)=C02+C1(i)+C2(i)+C012*C3(j)+C4(j)
  C_1(i,j)=C1(i)
  C_2(i,j)=C2(i)
  C_3(i,j)=C012*C3(j)
  C_4(i,j)=C4(j)
  C_5(i,j)=C02
end do

!base4
do j=2,jb-1
  i=0
  C_0(i,j)=C02+C014+C3(j)+C4(j)
  C_1(i,j)=0
  C_2(i,j)=C014
  C_3(i,j)=C3(j)
  C_4(i,j)=C4(j)
  C_5(i,j)=C02
end do

!base5
do i=1,il-2
  do j=2,jb-1
    C_0(i,j)=C02+C1(i)+C2(i)+C3(j)+C4(j)
    C_1(i,j)=C1(i)
    C_2(i,j)=C2(i)
    C_3(i,j)=C3(j)
    C_4(i,j)=C4(j)
    C_5(i,j)=C02
  end do
end do

!base6
i=0
j=jb
C_0(i,j)=C02+C014+C3(j)+C4(j)*C015
C_1(i,j)=0
C_2(i,j)=C014
C_3(i,j)=C3(j)
C_4(i,j)=C4(j)*C015
C_5(i,j)=C02

!base7
do i=1,il-2
  j=jb
  C_0(i,j)=C02+C1(i)+C2(i)+C3(j)+C4(j)*C015
  C_1(i,j)=C1(i)
  C_2(i,j)=C2(i)

```

```

C_3(i,j)=C3(j)
C_4(i,j)=C4(j)*C015
C_5(i,j)=C02
end do
!gel1
i=0
j=jb+1
C_0(i,j)=C01+C014+C016*C3(j)+C4(j)
C_1(i,j)=0
C_2(i,j)=C014
C_3(i,j)=C016*C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C01
!gel2
do i=1,il-2
j=jb+1
C_0(i,j)=C01+C1(i)+C2(i)+C016*C3(j)+C4(j)
C_1(i,j)=C1(i)
C_2(i,j)=C2(i)
C_3(i,j)=C016*C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C01
end do
!gel3
do j=jb+2,jg-1
i=0
C_0(i,j)=C01+C014+C3(j)+C4(j)
C_1(i,j)=0
C_2(i,j)=C014
C_3(i,j)=C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C01
end do
!gel4
do i=1,il-2
do j=jb+2,jg-1
C_0(i,j)=C01+C1(i)+C2(i)+C3(j)+C4(j)
C_1(i,j)=C1(i)
C_2(i,j)=C2(i)
C_3(i,j)=C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C01
end do
end do

!gel5
i=0
j=jg
C_0(i,j)=C01+C014+C3(j)+C018*C4(j)
C_1(i,j)=0
C_2(i,j)=C014
C_3(i,j)=C3(j)
C_4(i,j)=C018*C4(j)
C_5(i,j)=C01
!gel6
do i=1,il-2
j=jg
C_0(i,j)=C01+C1(i)+C2(i)+C3(j)+C018*C4(j)
C_1(i,j)=C1(i)
C_2(i,j)=C2(i)

```

```

C_3(i,j)=C3(j)
C_4(i,j)=C018*C4(j)
C_5(i,j)=C01
end do
!sample1
i=0
j=jg+1
C_0(i,j)=C04+C014+C017*C3(j)+C4(j)
C_1(i,j)=0
C_2(i,j)=C014
C_3(i,j)=C017*C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C04
!sample2
do i=1,il-2
j=jg+1
C_0(i,j)=C04+C1(i)+C2(i)+C017*C3(j)+C4(j)
C_1(i,j)=C1(i)
C_2(i,j)=C2(i)
C_3(i,j)=C017*C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C04
end do
!sample3
do j=jg+2,jl-2
i=0
C_0(i,j)=C04+C014+C3(j)+C4(j)
C_1(i,j)=0
C_2(i,j)=C014
C_3(i,j)=C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C04
end do
!sample4
do i=1,il-2
do j=jg+2,jl-2
C_0(i,j)=C04+C1(i)+C2(i)+C3(j)+C4(j)
C_1(i,j)=C1(i)
C_2(i,j)=C2(i)
C_3(i,j)=C3(j)
C_4(i,j)=C4(j)
C_5(i,j)=C04
end do
end do

! DO i=0,il-2
! DO j=jc,jl-2
!   WRITE(16,*) C_1(i,j)
! END DO
! END DO

```

!Calculation

```

N=1
M=1
O=1
!OO=1
TIME_S=REAL(N,8)*DELF0*(D**2.0D0)/alpha_g

DO i=0,il-1
DO j=jc,jl-1

```

```

        THETA(i,j)=THETA_0
    END DO
END DO
WRITE(*,*) 'NOW calculating...'

WRITE(8,*) 0.0D0,0.0D0
WRITE(11,*) 0.0D0,0.0D0
WRITE(13,*) 0.0D0,0.0D0
WRITE(14,*) " ",Nr_cen(0:il-1)*1.5D0
WRITE(12,*) "Nz_cen=",Nz_cen(jc:jl-1)*1.5D0
WRITE(9,*) "Nr_cen=",Nr_cen(0:il-1)*1.5D0
WRITE(15,*) 0.0D0

DO WHILE(TIME_S<=TIME_HEATING)

    DO i=0,il-1
        DO j=jc,jl-1
            THETA_OLD(i,j)=THETA(i,j)
        END DO
    END DO

    !Boundary Conditions(T=T0 At Deep Layer)

    do j=jc,jl-1
        i=il-1
        !THETA(i,j)=THETA_0
        THETA(i,j)=0
    end do

    do i=0,il-1
        j=jl-1
        !THETA(i,j)=THETA_0
        THETA(i,j)=0
    end do

    !MASK    WRITE(*,*) '4 CLEAR'
    !do j=jc,jl-1
    !    write(16,*) j,THETA(0:il-1,j)
    !end do

    CALL
    SOR_METHOD(il,ih,jl,jb,jg,jc,C_0,C_1,C_2,C_3,C_4,C_5,C013,THETA_OLD,THETA,ITER)
    !CALL SOR_METHOD(il,ih,js,jb,jc,jg+2,C_0,C_1,C_2,C_3,C_4,C_5,C013,&
    !    THETA,THETA_OLD,ITER)

    !From Non-dimensional Order To dimensional Order
    DO i=0,il-1
        DO j=jc,jl-1
            T(i,j)=((D**2)*Q/lambda_g)*THETA(i,j)+T0
        END DO
    END DO

    !Average Heater Temperatures (From T(0,0) to T(IH,0))
    DO i=0,ih
        T_HEATER(i)=T(i,0)
    END DO
    do i=0,ih

DEL_T(i)=(T_HEATER(i)*(Nr(i)*Ndelr(i-1)+Nr(i)*Ndelr(i)+((Ndelr(i))**2-(Ndelr(i-1))**2)/4))-T0

```

```

end do
DEL_T_AVEE=SUM(DEL_T)

do i=0,ih
DEL_T2(i)=(T_HEATER(i)*(Nr(i+1)**2-Nr(i)**2))-T0
end do
DEL_T_AVEE2=SUM(DEL_T)
!Average Heater Non-dimensional Temperature (From THETA(0,0) to THETA(IH,0))
DO i=0,ih
THETA_HEATER(i)=THETA(i,0)
END DO
do i=0,ih

THETA_(i)=(THETA_HEATER(i)*(Nr(i)*Ndelr(i-1)+Nr(i)*Ndelr(i)+((Ndelr(i))**2-(Ndelr(i-1))**2)
/4))
end do
THETA_AVEE=SUM(THETA_)

!MARK WRITE(*,*) '6 CLEAR'

!Output T(0,0)
IF (M==step) THEN
WRITE(*,*) 'Passaged time =',TIME_S,'s'
WRITE(*,*) 'T(0,0) =',T(0,0),'ITER=',ITER
!WRITE(6,*) THETA(0,j_1:jl)
!WRITE(12,*) THETA(0,jb:jl-1)
!WRITE(9,*) THETA(0:il-1,0)
WRITE(*,*) DEL_T_AVEE
WRITE(*,*) THETA_AVEE
WRITE(8,*) TIME_S,DEL_T_AVEE
WRITE(15,*) DEL_T_AVEE
WRITE(13,*) TIME_S,THETA_AVEE
WRITE(11,*) TIME_S,DEL_T_AVEE2
! WRITE(*,*) ITER
!write(51,*) THETA(0:200,0)
WRITE(*,*) ''
WRITE(*,*) 'NOW calculating...'

M=0

END IF

if (O==step2) then
WRITE(10,*) TIME_S,"s"
do j=jc,jl-1
write(10,*) j,THETA(0:il-1,j)
end do
write(10,*) O
!WRITE(10,*) OO

O=0
!OO=OO+1

! T distribution of ALL AREA
WRITE(14,*) TIME_S,"s"
WRITE(14,*) " ",Nr_cen(0:il-1)*1.5D0
DO j=jc,jl-1
WRITE(14,*) Nz_cen(j)*1.5D0,T(0:il-1,j)
END DO

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[illegible]

APPENDIX B

COMPUTER PROGRAM

```
!      Last change:  HMT      1 Nov 2012      4:17 am
!-----
!
!      cmt (contact measurement techniques)Fit
!  Uses an iterative procedure to determine thermal conductivity
!      and diffusivity from cmt simulated data
!
!      A.Yoshida 2007
!-----

PROGRAM cmtFit
IMPLICIT NONE
INTEGER :: n,i,v
INTEGER :: nt      !No. of time steps for calculation
REAL(8),PARAMETER :: LAMBDA_G=0.60D0, ALPHA_G=1.50D-7, TIME_HEATING=5.0D0,
DELFO=1.0D-6
REAL(8),PARAMETER :: D=1.50D-3
REAL(8) :: LAMBDA_S, ALPHA_S, DELTA, LAMBDA_Sp, ALPHA_Sp, xL, xA, S
REAL(8),DIMENSION(:),ALLOCATABLE :: T_sim, T_AVE, T_L, T_A

OPEN(UNIT=1,FILE="T_sim_0.03.txt",STATUS="UNKNOWN")
OPEN(UNIT=2,FILE="T_AVE.txt",STATUS="UNKNOWN")
OPEN(UNIT=3,FILE="T_L.txt",STATUS="UNKNOWN")
OPEN(UNIT=4,FILE="T_A.txt",STATUS="UNKNOWN")
OPEN(UNIT=5,FILE="T_D.txt",STATUS="UNKNOWN")

! nt=INT((ALPHA_G*TIME_HEATING/DELFO/D**2.0D0)/2000.0D0)
! nt=nt+1
nt=256

ALLOCATE(T_sim(nt),T_AVE(nt),T_L(nt),T_A(nt))

DO n=1,nt
  READ(1,*) T_sim(n)
  READ(2,*) T_AVE(n)
  READ(3,*) T_L(n)
  READ(4,*) T_A(n)
END DO

WRITE(*,*) T_sim(1),T_AVE(1),T_A(1),T_L(1),nt

!LAMBDA_S = 0.42D0; ALPHA_S = 1.170D-7; DELTA = 2.5D-5      !True Value
LAMBDA_S = 0.2440D0; ALPHA_S = 1.8750D-7; DELTA = 5.0D-5      !Initial Value
LAMBDA_Sp=LAMBDA_S*1.01
ALPHA_Sp=ALPHA_S*1.01

OPEN(UNIT=6,FILE="i,DELTA_S, ALPHA_S, DELTA,SD=0.03.txt",STATUS="UNKNOWN")
```

```

OPEN(UNIT=7,FILE="LAMBDA_S, ALPHA_S.txt",STATUS="UNKNOWN")
! OPEN(UNIT=12,FILE="S,SD=0.03.txt",STATUS="UNKNOWN")

CALL leastsq(xL,xA,T_sim,T_L,T_A,T_AVE,nt)    !Solve a least-squares problem

LAMBDA_S = LAMBDA_S + xL*(LAMBDA_Sp-LAMBDA_S)
ALPHA_S = ALPHA_S + xA*(ALPHA_Sp-ALPHA_S)

WRITE(6,*) 'xL=',xL,'xA=',xA
WRITE(7,*) "LAMBDA_S=",LAMBDA_S, LAMBDA_S*1.01
WRITE(7,*) "ALPHA_S=", ALPHA_S, ALPHA_S*1.01

S = 0.0D0
DO n = 1, nt
    S = S + (T_sim(n)-T_AVE(n))*2.0D0
END DO

S = (S/REAL(nt,8))*0.50D0    !S = Standard daviation

WRITE (7,*) S
WRITE (*,*) 'S=',S

END PROGRAM cmtFit

!-----
SUBROUTINE leastsq(xL,xA,T_sim,T_L,T_A,T_AVE,nt)
!
! linear least squares method to estimate required adjustment
!
IMPLICIT NONE
REAL(8), INTENT(OUT) :: xL, xA
INTEGER, INTENT(IN) :: nt
REAL(8), INTENT(IN) :: T_sim(nt), T_L(nt), T_A(nt), T_AVE(nt)

REAL(8) :: x(2)
REAL(8) :: Aj(nt,2), y(nt)
INTEGER :: isingular, i, ierr

DO i = 1, nt
    y(i) = T_sim(i)-T_AVE(i)
    Aj(i,1) = T_L(i)-T_AVE(i)
    Aj(i,2) = T_A(i)-T_AVE(i)
END DO
CALL gslsq(x,Aj,y,nt,2,isingular)
xL = x(1)
xA = x(2)

RETURN
END SUBROUTINE leastsq

!-----
SUBROUTINE gslsq(x,Aj,y,m,n,isingular)
!
! Solves linear least squares problem
! [Aj] {x} = {y}
! where [Aj] is a m*n matrix (m>=n)
! {x} is the vector containing the solution
!
IMPLICIT NONE
INTEGER, INTENT(IN) :: m,n

```

```

    INTEGER, INTENT(OUT) :: isingular
    REAL(8), INTENT(OUT) :: x(n)
    REAL(8), INTENT(IN) :: Aj(m,n), y(m)

    REAL(8) :: R(n,n)
    IF(n>m) THEN
        WRITE(*,*) 'must have at least as many points as coeffs'
        STOP
    ENDIF
    CALL gsQR(Aj,R,m,n,isingular)
    IF(isingular/=1) CALL solveQR(x,y,Aj,R,m,n)
    RETURN
END SUBROUTINE gslsq
!-----
SUBROUTINE gsQR(Aj,R,m,n,isingular)
!
! Q R decomposition of [Aj] by modified Gram-Schmidt method
! Find [Q] and [R] such that
! [Aj] = [Q] [R]
! where [R] is upper triangle n*n and [Q] is m*n
! with columns orthogonal vectors
!
! [Aj] is overwritten by Q when returned
!
IMPLICIT NONE
INTEGER, INTENT(IN) :: m,n
INTEGER, INTENT(OUT) :: isingular
REAL(8), INTENT(INOUT) :: Aj(m,n)
REAL(8), INTENT(OUT) :: R(n,n)

REAL(8), PARAMETER :: rsmall = 1.0D-14
REAL(8) :: amag
INTEGER :: k, im, j
isingular = 0
DO k = 1, n
    ! find norm -----
    amag = 0.0D0
    DO im = 1, m
        amag = amag + Aj(im,k)**2
    END DO
    amag = SQRT(amag)
    R(k,k) = amag
    IF(amag<rsmall) THEN
        isingular = 1
        write(*,*) 'amag: ',amag, k
        RETURN
    ENDIF
    ! normalize column -----
    DO im = 1, m
        Aj(im,k) = Aj(im,k)/amag
    END DO
    ! make all other columns orthogonal - -
    ! to column k
    DO j = k+1, n
        R(k,j) = Aj(1,k)*Aj(1,j)
        DO im = 2, m
            R(k,j) = R(k,j) + Aj(im,k)*Aj(im,j)
        END DO
        DO im = 1, m
            Aj(im,j) = Aj(im,j) - R(k,j)*Aj(im,k)
        END DO
    END DO
END DO

```

```

        END DO
    END DO
! -----
END DO
RETURN
END SUBROUTINE gsqr
!-----
SUBROUTINE solveQR(x,y,Q,R,m,n)
!
! solve for {x}:
!           [Q][R] {x} = {y}
! [Q] has orthogonal columns so that [Q]^T [Q] = [I]
! [R] is upper triangle
! therefore: [R] {x} = [Q]^T {y}
! and {x} can be solved by back substitution
!
IMPLICIT NONE
INTEGER, INTENT(IN) :: m,n
REAL(8), INTENT(OUT) :: x(n)
REAL(8), INTENT(IN) :: y(m), Q(m,n), R(n,n)

REAL(8) :: totrhs
REAL(8), ALLOCATABLE :: z(:)
INTEGER :: i, im, j, ierr

ALLOCATE (z(n), STAT=ierr)
IF(ierr/=0) THEN
WRITE(*,*) ierr
STOP
ENDIF
! evaluate [Q]^T {y} -----
DO i = 1, n
    z(i) = Q(1,i)*y(1)
    DO im = 2, m
        z(i) = z(i) + Q(im,i)*y(im)
    END DO
END DO
! back substitute -----
x(n) = z(n)/R(n,n)
DO i = n-1, 1, -1
    totrhs = z(i)
    DO j = i+1, n
        totrhs = totrhs - R(i,j)*x(j)
    END DO
    x(i) = totrhs/R(i,i)
END DO
DEALLOCATE (z)
RETURN
END SUBROUTINE solveQR
!-----

```