A QUASI-STEADY CYLINDER METHOD FOR THE SIMULTANEOUS DETERMINATION OF HEAT CAPACITY, THERMAL CONDUCTIVITY AND THERMAL DIFFUSIVITY WITH THE USE OF TEMPERATURE MEASUREMENT IN ONE POINT ONLY

Wolfgang POESSNECKER and Ulrich GROSS*

Institute of Thermal Engineering, Technische Universität Bergakademie Freiberg D-09599 Freiberg, Germany, *corresponding author: gross@iwtt.tu-freiberg.de

Abstract

Heat capacity, thermal conductivity and thermal diffusivity are measured simultaneously using a hollow cylindrical sample with adiabatic boundary conditions at its outer surface in axial and radial direction. Starting from thermal equilibrium a constant heat flow is supplied to the inner surface of the sample. The temperatures throughout the sample are raised asymptotically approaching a certain constant radial profil which is time-linear shifting upwards. Measurements of the temperature at only one position allow the determination of all three properties and no temperature differences are needed.

Keywords: Simultaneous method, thermal conductivity, thermal diffusivity, specific heat, measuring technique

1. Introduction

For simultaneous determination of the thermophysical properties various transient, pulse, step-wise and periodic heat flow methods (wire and plane sources) are widely applied (for an overview see [1], [2]). The peculiarity of these methods mostly consists in considerable deviations between mathematical model and practical realisation. These deviations typically cause complicated formulas for the evaluation on the one hand and not seldom a strong limitation of exactness on the other hand. Because of it, a new simultaneous method with different than usual new features has been developed. A similar procedure for the

simultaneous measurement of the three properties has been published earlier by the present authors [1] where continous internal heating of a hollow adiabatic cylinder has been applied. The long-term behaviour has been evaluated based on temperature measurements at two radial positions and all three properties are obtained for bad conducting materials. In contrast to this the present method is focused on the short-time transient temperature at only one location.

2. Principle of the Method

A hollow cylindical sample is used ($R_1 = 0.008 \text{ m}$, $R_2 = 0.0175 \text{ m}$ as the inner and outer radius) with an axial borehole (at radius r) containing one single well contacted temperature sensor. Starting with uniform temperature, a constant heat flow is supplied to the inner surface of the sample. The temperature field develops depending on the thermophysical properties and also on axial and radial heat losses. The latter ones increase with time distorting the measurements. The resulting error can be reduced and finally avoided by application of controlled protection shields and/or application of long cylinders and short measuring times.

2.1 Mathematical Solution

Neglecting axial losses the temperature field is one dimensional and it can be calculated from the solution of the following initial boundary condition problem with thermal diffusivity a, thermal conductivity λ and the excess temperature "T" starting from time zero:

$$\frac{\partial T}{\partial t} = a \left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} \right) \quad \text{where} \quad -\lambda \left(\frac{\partial T}{\partial r} \right)_{R_1} = \dot{q} \quad \text{and} \quad -\lambda \left(\frac{\partial T}{\partial r} \right)_{R_2} = \alpha \left(T_{R_2} \right) \quad (1)$$

The exact analytical solution has been obtained by Laplace transformation. It is not included in this paper due to its space consuming complexity, however, it has been evaluated numerically for some examplary cases ($a = 9.25 \, 10^{-6} \, m^2 s^{-1}$, $\lambda = 22 \, Wm^{-1}K^{-1}$, inside heat flow rate $\dot{q} = 11659.7 \, Wm^{-2}$ and outside heat transfer coeffcients $0 \le \alpha \le 2000 \, Wm^{-2}K^{-1}$) and the resulting excess temperature is plotted in Figure 1 for one location close to the inner surface (at $r = 0.0084 \, m$).

In case of perfectly adiabatic conditions ($\alpha = 0 \text{ Wm}^{-2}\text{K}^{-1}$) the slope of the temperature increase (bold line) is found to approach a straight asymptote as expected which is reached

after a settling time of about $t_{set} = 4 \text{ s}$. This line is exactly represented by the quasi-stationary part of the extended solution (for $\alpha = 0 \text{ Wm}^{-2}\text{K}^{-1}$ and $t \rightarrow \infty$) with the transient $\dot{T} = AB$

$$T(\mathbf{r},\mathbf{t}) = \underbrace{\frac{\dot{q}R_{1}R_{2}}{\lambda(R_{2}^{2}-R_{1}^{2})}}_{A} \left\{ \underbrace{\frac{2a}{R_{2}}}_{B} \underbrace{-\frac{R_{2}\ln\frac{r}{R_{2}} - \frac{R_{2}}{2} + \frac{r^{2}}{2R_{2}} - \frac{\left[R_{1}R_{2}\ln\frac{R_{1}}{R_{2}} + \frac{1}{4}\left(\frac{R_{2}^{4}-R_{1}^{4}}{R_{1}R_{2}}\right)\right]R_{1}}{C} \right\} (2)$$

In the experiment, the sample will be positioned inside a device with or without special precautions to avoid radial heat losses. Evacuation will help to reduce heat conduction and convection effects to be negligible. Remaining radiation losses can be minimized by surrounding the sample by controlled radiation shields (see, e.g., [1]) to guarantee adiabatic conditions in a best possible way. Remaining radial losses will affect the temperature increase to be weaker exhibiting a saturation characteristic (horizontal asymptote) where heat input is balanced by radial heat losses (see the dash-dotted curve in Figure 1, as calculated from the detailed solution with $\alpha = 2000 \text{ Wm}^{-2} \text{K}^{-1}$ just for demonstration of a high heat loss case).



Figure 1: Temperature increase starting with time zero for various outside heat transfer condictions with t^* as the intersection time, see eq. (3)

In reality much smaller heat transfer coefficients have to be expected yielding negligible deviations from the adiabatic case at short times. Effective heat transfer coefficients up to $\alpha = 23 \text{ Wm}^{-2} \text{K}^{-1}$ (corresponding to radiation losses at the outer surface temperature 1800 K) have been applied to evaluate the actual temperature increase which proofs to keep below the accuracy limit of temperature measurements within the first 20 s. By this the application of the zero heat loss solution, eq. (2), for the thermophysical property calculations is enabled.

2.2 Evaluation of the Thermophysical Properties

As the first step the thermal diffusivity "a" will be evaluated from the intersection point t^* of the straight asymptote, eq. (2), with the time axis (T = 0, see Fig. 1)

$$Bt * + C = 0 \tag{3}$$

where t* is the respective negative(!) time obtained from the experiment. Combining the coefficient B from eq.(1) with eq.(3) the gained thermal diffusivity is easily obtained as

$$a = -\frac{CR_2}{2t^*} \tag{4}$$

with the parameter C, see eq.(2), only depending on the sample geometry. A practical way for finding t* consists in linear regression of the measured temperature increase with time for $t \ge t_{set}$, i.e. a plot analogeous to Fig. 1.

As the second step the thermal conductivity " λ " is obtained from eq. (2) with $\dot{T} = AB$

$$\lambda = \frac{2\dot{q}R_{1}a}{\dot{T}(R_{2}^{2} - R_{1}^{2})}$$
(5)

As the third step and in contrast to the evaluation of "a" and " λ ", the specific heat " c_p " can be calculated directly from its definition with \dot{Q} as the heat input and m as the mass of sample and device respectively

$$c_{p} = \frac{\dot{Q}}{m_{sample}\dot{T}} - \frac{(mc_{p})_{device}}{m_{sample}}$$
(6)

3. Consideration of Uncertainties by Heat Losses

Uncertainties by heat losses should nearly disappear for short times as discussed above for small values of α . By means of eq.(4), diffusivities and their deviations from the adiabatic case have been calculated for selected heat transfer conditions (see Table 1). The resulting error by heat losses is clearly shown to keep below about 1 % in any practical cases.

Table 1: Effect of outside heat transfer conditions on the resulting thermal diffusivity,

α (radiation losses)	0	0.9	3	7	13	23	$Wm^{-2}K^{-1}$
Thermal diffusivity	9.2514	9.2443	9.2320	9.1877	9.1309	9.0400	$10^{-6} \mathrm{m}^2 \mathrm{s}^{-1}$
deviations from the adiabatic case	-	0.08	0.21	0.69	1.32	2.34	%

Eqs. (6) and (5) for the determination of specific heat and thermal conductivity respectively are only valid for adiabatic conditions. In presence of heat losses the heating process (subscript H) has to be supplemented by considering the dynamic response of the system after switching off the heating elements, i.e. for the subsequent cooling process (subscript C) with a respective negative(!!) temperature transient.

For heating, subscript H is valid:

After switching off the input power is zero:

Substraction brings for the sample's specific heat

and analogeous for the thermal conductivity:

$$\dot{\mathbf{Q}} = ((\mathbf{mc}_{p})_{\text{sample}} + (\mathbf{mc}_{p})_{\text{device}})\dot{\mathbf{T}}_{H} + \dot{\mathbf{Q}}_{\text{heat loss}}$$
$$0 = ((\mathbf{mc}_{p})_{\text{sample}} + (\mathbf{mc}_{p})_{\text{device}})\dot{\mathbf{T}}_{C} + \dot{\mathbf{Q}}_{\text{heat loss}}$$
$$\mathbf{mc}_{p} = \frac{\dot{\mathbf{Q}}}{\left|\dot{\mathbf{T}}_{H}\right| + \left|\dot{\mathbf{T}}_{C}\right|} - (\mathbf{mc}_{p})_{\text{device}}$$
$$\lambda = \frac{2\dot{\mathbf{q}}\mathbf{R}_{1}\mathbf{a}}{\left(\left|\dot{\mathbf{T}}_{H}\right| + \left|\dot{\mathbf{T}}_{C}\right|\right)\left(\mathbf{R}_{2}^{2} - \mathbf{R}_{1}^{2}\right)}$$

4. First Measurements for Testing the Simultaneous Method

First measurements with Titanium for testing the method were carried out by very quick pushing of a precedently heated coil ($\dot{q} = 4973.6 \text{ Wm}^{-2}$) into the sample with the presence of air at room temperature. This coil is made from an oxidized Kantal wire housed in a quartz tube for prevention of short circuit (m = 60 g and c_p = 0.48 Jg⁻¹K⁻¹ for the device). A directly welded coated thermocouple was used as temperature sensor positioned in a narrow borehole near the inner surface. From the measured temperature history the following data

have been evaluated: $\dot{T} = 0.1383 \text{ Ks}^{-1}$ (slope) and $t^* = -4.2 \text{ s}$ (intersection time) and subsequently the properties from eqs. (4) to (6)

- thermal diffusivity: $a = 9.25 \ 10^{-6} \ m^2 s^{-1}$ (Touloukian [3]: $a = 9.25 \ 10^{-6} \ m^2 s^{-1}$)
- themal conductivity: $\lambda = 21.97 \text{ Wm}^{-1}\text{K}^{-1}$ (d'Ans-Lax [4]: $\lambda = 21.9 \text{ Wm}^{-1}\text{K}^{-1}$)
- specific heat: $c_p = 0.5236 \text{ Jg}^{-1}\text{K}^{-1}$ (d'Ans-Lax [4]: $c_p = 0.5232 \text{ Jg}^{-1}\text{K}^{-1}$)

which is in excellent agreement with the reported literature data.

5. Conclusions

The results received for Titanium at room temperature show very good agreement with the respective recommended values by Touloukian and D'Ans-Lax. The next aim is the further development of the method for higher temperatures. The heat losses which will increase at higher temperatures are to be avoided by compensation and additionally by commonly used constructive measures like automatically controlled protection shields.

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REFERENCES

- Poessnecker, W., Gross, U., A Quasi-steady Cylinder Method for Simultaneous Thermophysical Property Measurements up to 2000K, J. Thermal Analysis and Calorimetry, Vol. 86, pp. 809-817, 2006
- [2] Kubicar, L., Bohac, V., Review of several dynamic methods of measuring thermophysical parameters, Proc. 24th Int. Thermal Conductivity/ 12th Int. Thermal Expansion Symposium, eds. P.S. Gaal und D.E. Apostolescu, Technomic Publ. (Lancester/USA), pp135-149, 1999
- [3] Touloukian, Y.S., Thermophysical Properties of High Temperature Solids, IFI (New York, Washington) Vol. 4, 1970
- [4] D'Ans, J., Lax, E., Taschenbuch für Chemiker und Physiker (in german language),ed.: K. Schäfer und C. Synowietz, Springer-Verlag (Berlin) Vol. 3, 2006