

A QUASI-STEADY CYLINDER METHOD FOR SIMULTANEOUS THERMOPHYSICAL PROPERTY MEASUREMENTS UP TO 2000 K

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For the simultaneous measurements of thermophysical properties a cylindrical sample is heated up in radial direction in a quasi-steady way. After a short settling time, the radial temperature profile will have established in the sample, which does not change with time anymore if the thermophysical and geometrical parameters of the sample remain constant. By this, one gets three properties (λ , a , c_p) directly without knowledge of the density, with only one sample, in only one measuring course, at temperatures up to 2000 K. However, two preconditions must be fulfilled for the simultaneous measurement of the three properties: firstly the heating mechanism of the sample to be measured has to be adiabatic and secondly the temperature difference within the sample must be high enough (for the measurement of the specific heat alone the second precondition is not necessary). A respective instrument has been designed and tested with molybdenum, titanium and alumina.

Keywords: *adiabatic scanning calorimeter, specific heat, thermal conductivity, thermal diffusivity*

Introduction

The increasing possibilities, abilities and needs for technical process simulations lead to a fast growing demand for thermophysical properties. Among these thermal conductivity and diffusivity, specific heat and also enthalpy and temperature behaviour of physical transformations have to be measured with high accuracy and reliability in an appropriately fast and economic way – and often at extremely high temperatures.

Apart from the application of well established measuring procedures for the individual properties, methods have been developed in the past for the simultaneous determination of at least two of them. Such procedures are always operated in a transient or quasi-steady way (see, e.g. [1] and more recently [2]).

Most frequently, ‘stepwise transient methods’ are applied including, e.g., the well known hot-wire, hot-strip and hot-disk procedures. A very thin line or foil shaped heat source is completely surrounded by an extended sample at initially uniform temperature. Starting from time zero the heat source delivers a constant heat flow by electrical heating. As the system’s response the temperature rise with time is measured and based on the well-known mathematical solution thermal conductivity and diffusivity can be determined by parameter identification - the latter property, however, with strongly reduced accuracy. These methods have been applied to gases, liquids and bad conducting solids since more than one hundred years.

An only short heating pulse is released in case of the ‘pulse transient methods’ which allow for the determination of specific heat and thermal diffusivity by fitting the mathematical solution to the dynamic response. Kubičar [3], Nastaj [4] and Hammer-schmidt [5] applied plane and strip-like sensors respectively. The evaluation procedure of the pulse methods is more complex, the measuring accuracy however is considerably improved.

A well-known and often used alternative is the ‘flash procedure’ usually with a laser source and with a flatcylindrical sample. One front side is subjected briefly with a radiation pulse, and the temperature history of the turned away side (or of the illuminated front, but seldom used) is measured and evaluated to obtain the thermal diffusivity. There are only a few successful attempts, e.g. by Kobayasi and Kobayashi [6], Kiss *et al.* [7] and Shinzato and Baba [8], to measure thereby also the specific heat which usually failed in such experiments due to heat losses.

In the ‘periodic heat flow methods’ oscillating heating is applied instead of the gradual or the pulse-type heat supply. A respective temperature oscillation develops inside the adjacent medium being damped and phase shifted with increasing distance from the heat source. Temporally and locally dissolved temperature measurements yield thermal conductivity, thermal diffusivity, specific heat and also contact resistance. There are some variants, starting with the ‘longitudinal heat flow method’ suggested by

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Angström [9]. More recently Champoussin [10] and Bierer *et al.* [11] presented investigations with oscillating electrical and convective heating respectively, Emelyanov [12] with modulated electron beam and Czarnetzki and Roetzel [13] with modulated laser heating. Lots of applications are reported for the ‘radial heat flow method’ with a hollow cylinder heated from inside or outside, and more rarely the ‘hot wire method’ with periodic heating (e.g. Fukai *et al.* [14]).

In the ‘subsecond pulse heating method’ described, e.g., by Seifter *et al.* [15] and Wilthan *et al.* [16], the sample consists of a thin wire, which is electrically heated to very high temperatures in an extremely fast way. Specific heat and electrical resistance are obtained from electric current, tension and contactless temperature measurements, and after that with the help of the Wiedemann–Franz law the thermal conductivity and also the thermal diffusivity may be obtained if the density is available. The validity of the Wiedemann–Franz law, however, is restricted.

With all these procedures an initially steady state of the measuring system is disturbed by constant, pulse type or oscillating heating. The temperature history is measured at one or more positions as the dynamic system’s response and from this the various properties (thermal diffusivity, thermal conductivity, specific heat) may be evaluated. Two of them are usually measured – the respective third one can then be obtained from $\lambda = acp$ with, however, reduced accuracy due to the accumulation of the inevitable measuring errors.

In this contribution a new quasi-steady method is presented which allows the simultaneous and direct measurement of all three properties in only one or rather two measurements without using $\lambda = acp$.

Theory

Principle of the quasi-steady measurements

Figure 1 shows the underlying principle with the cylindrical sample (pos. 1; radius R , length $2L$) positioned inside a furnace (outer heating tube, pos. 2), whose central section (pos. 4) is heated up with linear rise of temperature T_F . Inside the sample a transient temperature field develops approaching an invariable radial temperature profile with temperature differences and transients which allow the determination of the various properties. There are two lateral guard heaters (pos. 5 and 6) operated and controlled for minimization of the axial temperature gradients.

The furnace contains the measuring device consisting of a separate electrical heater (internal heating tube, pos. 3) and the sample which both have the same length. Adiabatic conditions in radial direction are

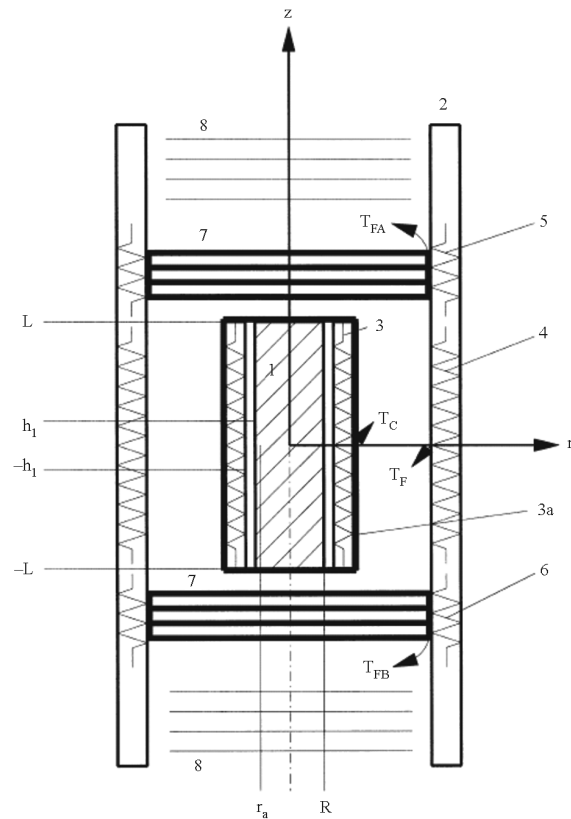


Fig. 1 Scheme of the method of measurement. 1: sample, 2: outer heating tube, 3: internal heating tube, 4: central section of the outer heating tube 2, 5: upper section of the outer heating tube 2, 6: lower section of the outer heating tube 2, 7: thermal conducting multiple inner screen system, 8: radiation protection screen system

produced by controlled heating for the temperature difference ($T_C - T_F$) to disappear. Thus, the heat flow rate is completely supplied to the sample – if axial heat losses are avoided. This critical condition is realized with very good approximation (see results) by application of two-stage radiation protection screens (pos. 7 and 8) which reduce the axial losses in the best possible way, whereby in particular the thermally conducting multiple inner screen system (pos. 7) brings the temperatures T_{FA} and T_{FB} with good approximation direct to the front surfaces of the internal heating system.

Literature review

The first instrument based on this principle has been developed at the National Bureau of Standards in the USA [17] for thermal diffusivity measurements of bad-conducting materials. Cape *et al.* [18] were the next to report a modification of the principle and its application to diffusivity measurements of well conducting materials like Ta, ZrC and TiC. In the 1970’s there were some Japanese investigations of metal

diffusivities based on the principle whereby the time-linear heating had partly to be gone off (Kosaka *et al.* [19, 20], Sasaki *et al.* [21]). The heat losses of the sample have been recognized as an unsurmountable problem making the determination of, e.g., the specific heat to be doubtful.

The application of the quasi-steady procedure for the presented simultaneous determination of thermal conductivity, thermal diffusivity and specific heat within very wide ranges of temperature and material properties is essentially new (Poessnecker *et al.* [22]; Poessnecker and Gross [23]). Only one report and one further conference paper have been found where the simultaneous procedure has been applied previously (Chen and Ge [24] and Fujino and Honda [25], respectively), however restricted to temperature below 400 K using a plate shaped sample heated from one side with a constant heat flow rate and using the resulting back-side temperature transient for the properties evaluation. In this, the adiabatic conditions have been achieved by differentiated control of the thermal environment, a method which is restricted to moderate temperatures.

Determination of the properties

Fourier's equation

$$\frac{\partial T}{\partial t} = a \left[\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2} \right] \quad (1)$$

is solved for constant lateral heating with the assumption of ideal adiabatic conditions in axial direction

$$-\lambda \left(\frac{dT}{dr} \right)_{r=R} = -\dot{q} \quad \text{and} \quad \left(\frac{dT}{dz} \right)_{z=L} = 0$$

yielding the following expression (according to Tautz [26]) for the temperature increase inside the cylindrical sample with the uniform starting temperature which equals the invariant ambient temperature T_{amb} :

$$T(r,t) = T_{\text{amb}} + \frac{\dot{q}R}{\lambda} \left[2Fo + \frac{1}{2} \left(\frac{r}{R} \right)^2 - \frac{1}{4} - 2 \sum_{k=1}^{\infty} \frac{1}{v_k^2} \frac{J_0(v_k r/R)}{J_0(v_k)} \exp(-v_k^2 Fo) \right] \quad (2)$$

with $Fo = at/R^2$ and the eigenvalues v_k derived from $J_1(v_k) = 0$.

A time-invariant radial temperature profile will be established after a short settling time converging to the quasi-steady temperature rise

$$T(r,t) = T_{\text{amb}} + \frac{\dot{q}R}{\lambda} \left[2Fo + \frac{1}{2} \left(\frac{r}{R} \right)^2 - \frac{1}{4} \right] \quad (3)$$

and from this the properties can be derived as:

$$T_{r=r_a} - T_{r=0} = \frac{\dot{q}r_a^2}{2\lambda R} \Rightarrow \lambda = \frac{\dot{Q}}{8\pi L(T_{r=r_a} - T_{r=0})} \left(\frac{r_a}{R} \right)^2 \quad (4)$$

$$\dot{T} = \frac{dT}{dt} = \frac{2\dot{q}a}{\lambda R} \Rightarrow a = \frac{r_a^2 \dot{T}}{4(T_{r=r_a} - T_{r=0})} \quad (5)$$

and with

$$c = \frac{\lambda}{a\rho} \Rightarrow c = \frac{\dot{Q}}{m\dot{T}} \quad (6)$$

For a given rate of the temperature rise \dot{T} (heat-up speed) and the measured heat flow rate to the sample $\dot{Q} = 4\pi RL\dot{q}$ thermal conductivity and diffusivity and also the specific heat can be determined from these equations without knowledge of the temperature-dependent density which is needed in case of the various other simultaneous methods.

Computation of the settling time

The intake disturbance must have faded out before evaluable measurements begin. The maximum radial temperature difference within the cylindrical sample can serve as a measure for this:

$$\begin{aligned} \Delta T(t) &= T_{r=R}(t) - T_{r=0}(t) = \\ &= \frac{\dot{q}R}{\lambda} \left[\frac{1}{2} - 2 \sum_{k=1}^{\infty} \left[\left(1 - \frac{1}{J_0(v_k)} \right) \frac{\exp(-v_k^2 Fo)}{v_k^2} \right] \right] \end{aligned} \quad (7)$$

which asymptotically increases from zero to its constant final value $\Delta T_{t \rightarrow \infty} = \dot{q}R/2\lambda$. The remaining difference

$$\begin{aligned} \Delta T_{t \rightarrow \infty} - \Delta T(t) &= \Delta T_{\text{Residuum}}(t) = \\ &= \frac{2\dot{q}R}{\lambda} \sum_{k=1}^{\infty} \left[\left(1 - \frac{1}{J_0(v_k)} \right) \frac{\exp(-v_k^2 Fo)}{v_k^2} \right] \end{aligned} \quad (8)$$

decreases with time depending on cylinder radius, material properties and heating conditions. By reference to the final value one gets the relative settling error as

$$\frac{\Delta T_{\text{Residuum}}}{\Delta T_{t \rightarrow \infty}} = 4 \sum_{k=1}^{\infty} \left[\left(1 - \frac{1}{J_0(v_k)} \right) \frac{\exp(-v_k^2 Fo)}{v_k^2} \right] \quad (9)$$

If this is accepted to be 1%, e.g., the respective settling Fourier number can numerically be obtained from Eq. (9) as

$$Fo_{\text{settling}} = \frac{at}{R^2} = 0.31 \quad (10)$$

Table 1 Lower bounds for settling time and heat-up speed for various thermal diffusivities a

Lower bounds for...		Settling time <i>t/s</i>			Heat-up speed <i>T/K min⁻¹</i>		
	Sample radius	<i>R</i> =0.01 m	0.02 m	0.04 m	<i>R</i> =0.01 m	0.02 m	0.04 m
Copper	$a=1.2 \cdot 10^{-4} \text{m}^2 \text{s}^{-1}$	0.3	1.0	4.1	≥ 57.6	≥ 14.4	≥ 3.6
Iron	$a=0.2 \cdot 10^{-4} \text{m}^2 \text{s}^{-1}$	1.6	6.2	24.8	≥ 9.6	≥ 2.4	≥ 0.6
Glass	$a=0.0072 \cdot 10^{-4} \text{m}^2 \text{s}^{-1}$	43.1	172.2	688.9	≥ 0.35	≥ 0.09	≥ 0.02

enabling determination of the settling time duration. Some examples are given in Table 1 for various materials and sample radii.

Heat-up speed

The attainable accuracy of thermophysical property measurements is most sensitive upon the temperature difference within the sample. Lower bounds for heat flow rate and heat-up speed can be derived from Eqs (4) and (5) respectively depending on geometrical and thermophysical properties and also on the temperature difference to be measured. Preconditions for reliable measurements are accurate positioning, very good contacting and regular calibration of the temperature sensors. With, e.g., $\Delta T=0.2$ K as a lower limit for the radial temperature difference between surface and axis of the sample, lower bounds for the heat-up speed are obtained (Table 1).

The settling time is found to be rather long for bad conducting materials, while the heat-up speed grows unacceptable high for good conductors where a very high heating capability would be required with serious adjustment problems. This holds in particular for cases of small sample radius. With the heat-up speed as listed in Table 1, the sample's surface temperature increases until the end of the settling time by $\Delta T_{\text{settling}}=0.348$ K, an amount which surprisingly is independent of all the geometrical and thermophysical parameters – and: it is completely insignificant. With bad conducting materials the long settling times can be reduced by taking a smaller sample radius and a higher heat-up speed.

Systematic errors and how to avoid them

Any deviation from the desired ideal adiabatic conditions cause systematic errors, which are superposed by uncertainties of the temperature measurements.

Axial heat-loss effects on the temperature field and their compensation

Because of the axial heat losses \dot{Q}_{ax} from the front surfaces at $z=\pm L$ the derived Eqs (4)–(6) are not valid exactly but they must be corrected. This correction is

realized by additional measurements in the steady state with $\dot{T}=0$ for compensation of heat loss effects on the heat flow and temperature differences in these equations.

The total heat flow rate $\dot{Q}=-\lambda 2\pi R(dT/dr)_{r=R} = C\dot{T}+\dot{Q}_{\text{ax}}$ penetrating radially into the sample causes a temperature gradient consisting of two parts according to $(dT/dr)_{r=R}=(dT/dr)_{\text{sample}}+(dT/dr)_{\text{losses}}$ which are needed for heating up the sample and for covering the axial losses respectively. The temperature difference associated with the latter one can easily be compensated from the static measurement as the heat losses are identical for both of the processes depending only on the respective sample surface temperatures which have to be the same. The true temperature difference may be calculated from the measured one as:

$$\begin{aligned} (T_{r=r_a} - T_{r=0})_{\text{corrected}} &= \\ &= (T_{r=r_a} - T_{r=0})_{\dot{T}>0} - (T_{r=r_a} - T_{r=0})_{\dot{T}=0} \end{aligned} \tag{11}$$

In an analogous manner the heat flow due to the heat losses can be compensated, too.

Preceding to the actual measurement with the sample the internal measuring device without the sample will be subjected to the same heating process. The losses are accurately identical in both cases, if the temperature distributions at the measuring device surface agree. To ensure this, the measuring device with and without the sample is enclosed in a container with local and temporal temperature distributions which are perfectly identical for both of the measuring procedures.

In accordance with eq. (6)

$$\dot{Q} = mc\dot{T}$$

the following expressions are obtained for the temperature dependent heat losses with and without the sample respectively

$$\left. \begin{aligned} \dot{Q}_{\text{with}} &= \dot{Q}_{\text{losses}}(T) + \dot{T}[(mc)_{\text{measuring device}} + (mc)_{\text{sample}}] \\ \dot{Q}_{\text{without}} &= \dot{Q}_{\text{losses}}(T) + \dot{T}(mc)_{\text{measuring device}} \end{aligned} \right\} \tag{12}$$

yielding by subtraction

$$(mc)_{\text{sample}} \dot{T} = \dot{Q}_{\text{with}} - \dot{Q}_{\text{without}}$$

and from this, Eqs (4) to (6) take the following respective formulations:

$$\left. \begin{aligned} \lambda &= \frac{\dot{Q}_{with} - \dot{Q}_{without}}{8\pi L(T_{r=r_a} - T_{r=0})_{corrected}} \left(\frac{r_a}{R}\right)^2 \\ a &= \frac{r_a^2 \dot{T}}{4(T_{r=r_a} - T_{r=0})_{corrected}} \\ c &= \frac{\dot{Q}_{with} - \dot{Q}_{without}}{m\dot{T}} \end{aligned} \right\} (13)-(15)$$

Contact resistance and asymmetry of temperature measurements

The cylindrical sample contains two axial drillings for the thermocouples in a defined radial distance. Besides this, in course of the present quasi-steady heating process a further error can occur due to a possible asymmetry of the two temperature-measuring locations. Depending on the heat-up speed \dot{T} the following heat flow rates have to be transferred from the inner borehole walls to the respective thermocouples

$$C_a \dot{T} = \frac{T_{wa} - T_a}{R_a} \quad \text{and} \quad C_i \dot{T} = \frac{T_{wi} - T_i}{R_i}$$

with C_i and C_a as the heat capacities, (T_{wi}, T_{wa}) and (T_i, T_a) the temperatures of the borehole walls and the thermocouples respectively and (R_i, R_a) the heat-conduction resistances lying in between. The difference between these two heat flow rates yields

$$\underbrace{T_a - T_i}_{\Delta T} - \underbrace{(T_{wa} - T_{wi})}_{\Delta T_w} = \dot{T} C_i R_i (1 - V_C V_R)$$

where $V_C = C_a/C_i$ and $V_R = R_a/R_i$ are the respective heat capacity and resistance ratios, and furthermore $\Delta T = T_a - T_i$ represents the temperature difference as measured with the thermocouple whereas $\Delta T_w = T_{wa} - T_{wi}$ is the true one at the borehole locations.

For steady-state problems, i.e. for $\dot{T} = 0$, the deviation between these temperature differences equals zero and does not depend on V_C and V_R . For $\dot{T} \neq 0$, however, deviations from the true temperature difference arise in cases of asymmetry in C and R , i.e. for $V_C \neq 1$ and $V_R \neq 1$ respectively. For measurements with, e.g., type B thermocouples (1 mm in diameter) embedded with sinter corundum ceramics in the 1.2 mm bore holes, applied to molybdenum and iron at $\dot{T} = 5 \text{ K min}^{-1}$ these deviations amount to

$$\left(\frac{\Delta T - \Delta T_w}{\Delta T_w}\right)_{\text{copper}} = 33\% \quad \text{and}$$

$$\left(\frac{\Delta T - \Delta T_w}{\Delta T_w}\right)_{\text{iron}} = 5.6\%$$

if $V_C = V_R = 2\%$ is assumed. This shows that the uncertainty may become very large with well conducting materials.

For achievement of a reasonable measuring accuracy a sound contact between the temperature sensor and the respective borehole wall is imperative. This can, e.g., be provided in the considered temperature range by ceramic powders which additionally fix the position of the thermocouples in the best possible way.

Experimental set-up

The request for adiabatic conditions in axial direction is additionally supported by selecting the aspect ratio $2L/R$ as large as possible and also by multiple subdivision of the sample perpendicularly to the cylinder axis with the gaps kept open by ceramic spacers. The principle of the internal heating device is shown in Fig. 2.

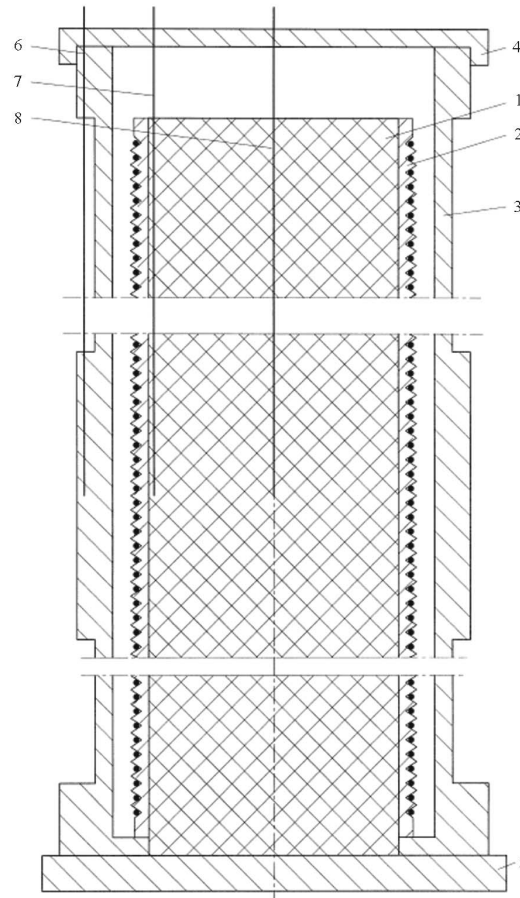


Fig. 2 Scheme of the internal heating system

A cylindrical sample (pos. 1) is used with length and outer diameter as 400 and 36 mm, respectively.

The sample is enclosed inside the internal heating tube (pos. 2) consisting of highly pure corundum (wall thickness 2 mm) with a milled in thread at its outer surface which contains the heating wire (molybdenum) in such a way that possible contacts with the surroundings are prevented. This device serves for the time-proportional adiabatic heating of the sample in accordance with the chosen heating program of the outer furnace (outer heating tube, pos. 2 in Fig. 1). The sample is enclosed by an additional molybdenum tube (balancing jacket, pos. 3 in Fig. 2, wall thickness 2 mm) with top and bottom plates (pos. 4 and 5, respectively). By this the internal heating system is completely covered by a well conducting envelope which ensures identical outside temperature distributions in course of experiments with and without the sample. A borehole is axially drilled into the molybdenum tube containing a thermocouple (pos. 6 in Fig. 2; corresponding to T_C in Fig. 1) used for the control of the internal heating tube (pos. 2 in Fig. 2). The radial gap between the molybdenum tube (pos. 3) and the internal heating tube (pos. 2) amounts to only 1 mm. The internal measuring device can easily be removed from the outer furnace in axial direction.

The furnace (pos. 2 in Fig. 1) is subdivided into three sections consisting of molybdenum wire mounted on the ceramic outer heating tube. The resulting furnace chamber is evacuated and flooded by argon.

Depending upon the temperature level, the measurements are taken with coated thermocouples of type K and B, respectively (diameter 0.65 mm) which are positioned in 0.7 mm drillings at the $z=0$ level, i.e. in the axial center of the sample (pos. 7 and 8 in Fig. 2).

Results and discussion

Steady-state adiabatic control

Heat losses have been checked for by steady-state measurements at various temperatures up to 1700 K. For $\dot{T}=0 \text{ K s}^{-1}$ Eq. (12) brings a balance of the radial heat flow to the sample and the axial losses

$$\dot{Q}_{\text{with}} = \dot{Q}_{\text{without}} = \dot{Q}_{\text{losses}}(T)$$

which proofed to keep below the lower limit of measureability in all the cases considered. By this the demand for adiabatic conditions is ensured for the quasi-steady measurements.

Specific heat, transformation temperature and enthalpy measurements of titanium

For the present test measurements titanium with a purity of 99.861% has been used which may have two different modifications, α - and β -titanium established below and above the transformation point with hexagonal and cubic structure respectively.

For the transformation temperature measurements there is a substantial advantage of the simultaneous method presented here. According to the theory of Poessnecker [27] the true transformation temperature T_U of first-order physical reactions is obtained independently of heat-transfer characteristics, adiabatic tangent losses and heat-up speeds as the intersection between the

$$\frac{dc_p}{dT} = \frac{A}{m\dot{T}} (\alpha_{w1}^{-1} + \alpha_{w2}^{-1})^{-1}$$

through the inflection point of the rising flank of the transition and the temperature axis in the c_p vs. T diagram (Fig. 3). A is the cross-section area of the heat flow penetrating into the sample. In case of infinitely high heat transfer coefficients α_{w1} and α_{w2} from the heating element to the envelope and from the envelope to the sample respectively, the dotted vertical line (in Fig. 3) is obtained as the virtual course of the curve.

The representation of the α - β transition of titanium is not handled uniformly in the literature. The plot of measured specific heat data shows a more or less pronounced peak with increasing and decreasing flanks (curve 1 in Fig. 3). Its shape and peak value depend on heat transfer details of the respective measuring device. Such plots are often found in literature, however, most of the authors prefer the depiction as sketched in Fig. 3 (and later in Fig. 4, too) with the idealized infinitely high heat transfer coefficients (dotted lines in Fig. 3).

In contrary, the very frequently used DSC methods as alternative scanning procedures only permit the determination of the onset temperature T_{on} which depends on both the heat-up speed and the heat transfer conditions.

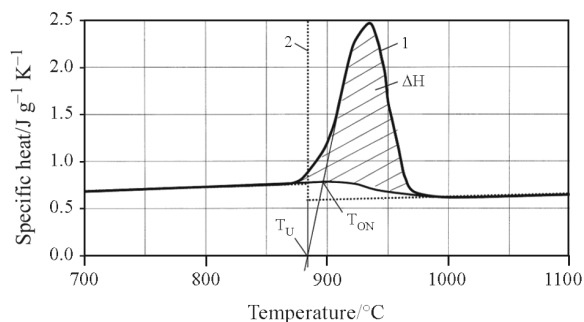


Fig. 3 Specific heat with the transition between modifications (titanium)

From the present measurements the transformation temperature (Fig. 3) has been evaluated as $T_{\alpha} = 1155$ K (882°C) which is found to fit well into the lower range of data from electrical resistance and specific heat measurements respectively reported in literature:

- 1155...1161 K McQuillan and McQuillan [28]
- 1155 K Hultgren *et al.* [29]
- 1165 K Cezairliyan and Miller [30]
- 1166 K Cezairliyan and Miller [31]
- 1150...1160 K Belskayja [32]
- 1155±2 K Boyer [33]
- 1164±1 and 1160±1 K Maglič and Pavčić [34]

The enthalpy increase connected with the α - β transition is obtained by numerical integration of the area between the measured specific heat and the sigmoidal connecting curve between α and β ranges (Fig. 3) and it has been evaluated to be 81.1 J g⁻¹. The agreement with 85 J g⁻¹ as the only data found in literature which has been estimated by Boyer [33] for titanium with the purity of 99.837% is rather well.

In the open literature there are some reports about previous specific heat measurements of titanium. The most recent compilation comes from Maglič and Pavčić [34], which face own measurements with literature data, and furthermore they evaluate and assess the measuring procedures used in the various experiments (drop technique, modulation technique, adiabatic calorimetry, microsecond-resolution pulse heating technique).

For α -titanium (Fig. 4) the present results are found to agree very well with the experimental data of Kothen [35] within (+3/-1%), whereas those of Maglič and Pavčić [34] and Peletski *et al.* [36] are somewhat above (+7/0) and below the own ones (0/-4%), the deviation of the present results from the compilations of JANAF [37] (1985) and [38] are found to be within (+2/-5%) and (+1/-6%) respectively.

For β -titanium excellent agreement is obtained with most of the literature data above 1300 K

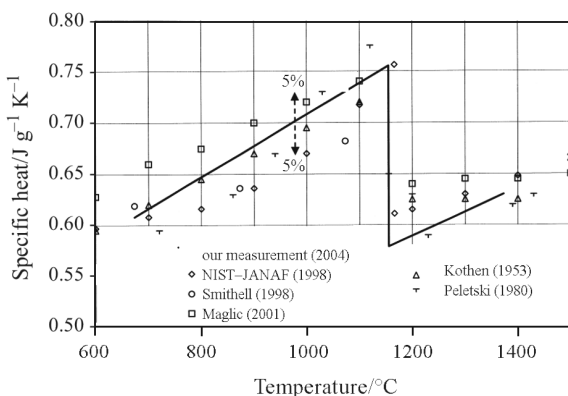


Fig. 4 Specific heat of titanium

(+1/-2%). The same holds for Peletski's results [36] in the entire temperature range whereas those of Kothen [35] and Maglič and Pavčić [34] are typically somewhat higher (up to 5 or 6%). This is also valid for the compilation of JANAF [38].

Within the range of phase conversions (first-order transformations) the specific heat cannot be evaluated and it can only be obtained therefore by a steady interpolation from α and β ranges respectively as the lower sigmoidal curve in Fig. 3.

Specific heat measurements of molybdenum

The specific heat of molybdenum (purity 99.888%) measured between 700 and 1600 K agrees excellent with literature data (Fig. 5). The comparison with the results of, e.g., Matsumoto and Ono [39] shows maximum deviations of (0/-1%) for 1300<T<1600 K, the comparison with Shinzato and Baba [8] yields (+2/-1%) for 700<T<1100 K and with Cezairliyan [40] (+1/-1%) for 1500<T<1600 K.

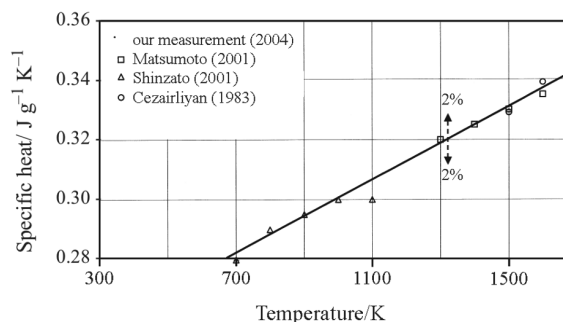


Fig. 5 Specific heat of molybdenum

Simultaneous measurements of specific heat, thermal conductivity and thermal diffusivity of Al₂O₃

Because of the relatively low thermal conductivity of Al₂O₃ the restrictions for the temperature difference measurements as discussed above here only exist in a strongly reduced manner so that respective measurements appear to be quite appropriate. A sintered Al₂O₃ sample (relative density 99.7% related to zero porosity) has been used and the measurements are evaluated according to formulas (13)–(15).

Specific heat

Figure 6 shows the results obtained for the specific heat (solid line) compared with two sets of literature data (both for relative density of 100%). Deviations from the standard reference data reported by Ewing *et al.* [41] from NBS are within 2 or 3%.

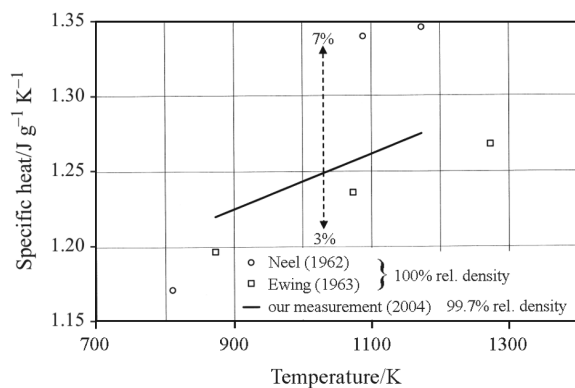


Fig. 6 Specific heat of Al_2O_3

Thermal conductivity and thermal diffusivity

The present measurements are found in the middle of other ones with similar relative densities (Fig. 7) ranging between 99.5 and 99.8%. However, pure crystal conductivities (Lee, [42]) are found far above. As to be seen, sintered materials yield very strong relative density and porosity effects on the thermal conductivity. The same holds for the thermal diffusivity measurements, Fig. 8, where comparison of own measurements with other ones without exact features of the samples appears problematic.

Conclusions

An instrument has been developed and constructed for the simultaneous measurement of specific heat,

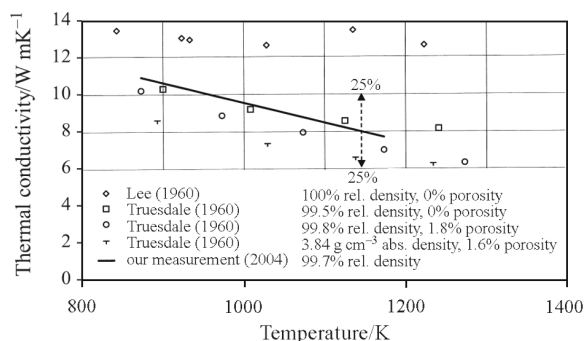


Fig. 7 Thermal conductivity of Al_2O_3

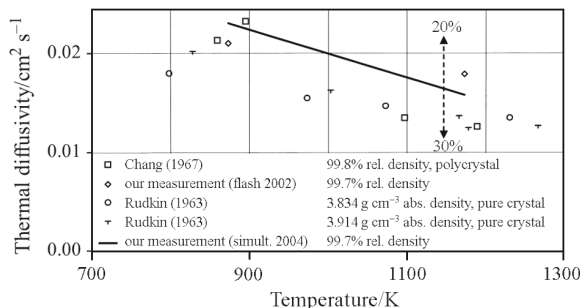


Fig. 8 Thermal diffusivity of Al_2O_3

thermal conductivity and thermal diffusivity for bad conducting materials without knowledge of the density in the solid state up to 2000 K.

For successful implementation of the measurement principle it is absolutely necessary that the measuring device containing the cylindrical sample possesses its own mechanism for radial heating of the sample being adiabatic in outward direction. This is ensured by a set of measures.

Exemplarily results measured with titanium and molybdenum at temperatures up to 1700 K are presented obtained by the quasi-steady procedure for specific heat, phase transition temperature and enthalpy. The agreement with data from the literature proves to be very good. Specific heat, thermal conductivity and thermal diffusivity are measured simultaneously for Al_2O_3 .

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