



Investigation and Optimization of the Hot Disk Method for Thermal Conductivity Measurements up to 750 °C

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Abstract

The Hot Disk method is a transient measurement method for the determination of thermal properties like the thermal conductivity, which is characterized by advantages such as a short measurement time or a low effort for the sample preparation. However, some difficulties related to measurements at elevated temperatures, which could be attributed to inaccuracies of the Temperature Coefficients of Resistance (TCRs), have been pointed out in the past. This paper presents a detailed investigation of the Hot Disk method for the determination of the thermal conductivity and contributes to a further improvement of its measurement accuracy. Subsequent to an extensive literature review of available reference materials for the thermal conductivity, measurements up to 750 °C were carried out with a Hot Disk TPS 2500 S with various Kapton and Mica sensors using three reference materials (Silcal 1100, Pyroceram 9606, Inconel 600). While room-temperature measurements confirmed the suitability of the reference samples as well as the independence of the measured thermal conductivity from the sensor, temperature-dependent measurements allowed the verification of the accuracy of the given TCRs. A set of optimized TCRs is proposed, with which the thermal conductivity of all three reference materials could be determined with an accuracy of 2 %. Furthermore, the measurement uncertainty of ± 5 % specified by the manufacturer could be confirmed. Hence, with the newly suggested TCRs, the Hot Disk method enables the determination of the thermal properties of a variety of materials even at high temperatures with high accuracy.

Keywords Hot disk · Reference materials · Temperature coefficient of resistance · Thermal conductivity measurement · Transient plane source method

List of Symbols

a Thermal diffusivity ($\text{m}^2 \cdot \text{s}^{-1}$)
 c_p Specific heat capacity ($\text{J} \cdot (\text{kg} \cdot \text{K})^{-1}$, $\text{J} \cdot (\text{m}^3 \cdot \text{K})^{-1}$)

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$D(\tau)$	Shape function (–)
P_0	Input power (W)
r	Radius (of the sensor) (m)
R	Resistance (Ω)
R_0	Reference resistance (Ω)
T	Temperature (K)
t	Time (s)
T_0	Reference temperature (K)
t_c	Corrected time (s)
T_r	Dimensionless temperature (–)
Δp	Probing depth (m)
ΔT	Temperature increase (K)
ΔT_i	Temperature increase across insulation layer (K)
ΔT_s	Temperature increase at the sample surface (K)
θ	Temperature ($^{\circ}\text{C}$)
Θ	Characteristic time (s)
λ	Thermal conductivity ($\text{W}\cdot(\text{m}\cdot\text{K}^{-1})$)
ρ	Density ($\text{kg}\cdot\text{m}^{-3}$)
τ	Dimensionless time (–)
τ_c	Corrected dimensionless time (–)

1 Introduction

Reliable, thermophysical material data are required for most procedures involving the design, layout, and optimization of heat transfer processes, with the thermal conductivity being one of the most important quantities. In general, there are a great variety of experimental methods for determining the thermal conductivity that can be classified into steady-state methods like the Heat Flow Meter or the Guarded-Hot-Plate method and transient methods like the 3ω principle, the Hot Wire method or Transient Plane Source (TPS) technique [1–3]. While simple analytical equations can usually be used for the evaluation of steady-state methods, transient methods require the adaptation of suitable mathematical-physical models to the measured temporal temperature course. Thus, evaluation and physical models are more complicated which might reduce the accuracy of these methods. However, the main advantages of transient methods are the smaller required sample sizes and the strongly reduced measurement time since no thermal equilibrium has to be reached [1–3].

The Hot Disk method, based on the TPS technique, is a very flexible and multifunctional transient method for measuring the thermal properties like the thermal conductivity of different materials including solids, liquids, and porous media, even at elevated temperatures. However, the application of this method at high temperatures is only scarcely investigated and some challenges in performing high-temperature measurements were demonstrated. Since the temperature increase at the sensor is determined via the change of its electrical resistance (further details in Chap. 2), precise knowledge of the temperature coefficient of resistance (TCR)

at the respective measurement temperature is necessary for reliable measurement results. At elevated temperatures, Goetze *et al.* [4] observed deviations in the received results up to 35 % compared to reference values attributing this to inaccurately determined TCRs. A suggested calibration and adaption of the TCRs led to significant improvements.

The general aim of this study is to further investigate and improve the reliability and accuracy of the Hot Disk method for thermal conductivity measurements at temperatures up to 750 °C with a focus set on the temperature-dependent TCRs. First, a literature review provides an overview of potential reference materials for the thermal conductivity that can be used to evaluate and calibrate the Hot Disk measurement system. Secondly, with several selected materials of different thermal conductivities, extensive measurements between room temperature and 750 °C were performed with a Hot Disk TPS 2500 S from Hot Disk AB (Sweden). With the room-temperature measurements, the suitability of the reference materials as well as the independence of the measurement results from the used sensor should be investigated. Based on the temperature-dependent measurements, the correctness of the TCRs proposed by Goetze *et al.* [4] in comparison to the original TCRs given by the manufacturer is analyzed and a further revision is suggested.

2 Fundamentals of the Hot Disk Method

As already mentioned, the Hot Disk method shows great flexibility and offers several advantages in contrast to other measuring methods, making it useful for numerous applications. Besides the fact that nearly all types of materials (solid, liquid, pastes, and porous media like powders and granular materials) can be measured [1–3, 5, 6], a main benefit is that due to the measurement setup, the sample form, and dimension are very flexible [2, 7]. Furthermore, the effort for the specimen preparation is very low, since only two samples with a flat, smooth surface are required for the measurement. Thus, in most cases, samples do not necessarily have to be destroyed for the measurements [6]. In addition to the standard isotropic measurement mode, the thermal conductivity of anisotropic materials in axial and radial directions can be determined under certain conditions [8]. Moreover, modules for the determination of the thermophysical properties of thin films and slab materials exists, which further extends the range of materials that can be investigated [8].

Due to the transient measurement principle, the thermal conductivity and the thermal diffusivity can be determined simultaneously [2, 3, 7] enabling also the calculation of the volumetric heat capacity. Here, the range in which thermal properties can be determined is very wide [2, 5]. When using the Hot Disk TPS 2500 S from Hot Disk AB, the thermal conductivity can be measured from $0.005 \text{ W}\cdot(\text{mK}^{-1})$ to $500 \text{ W}\cdot(\text{mK}^{-1})$ [6] (with a highest measured thermal conductivity of $1800 \text{ W}\cdot(\text{mK}^{-1})$ [8]), the thermal diffusivity from $0.1 \text{ mm}^2\cdot\text{s}^{-1}$ to $1200 \text{ mm}^2\cdot\text{s}^{-1}$, and the volumetric heat capacity up to $5 \text{ MJ}\cdot(\text{m}^3\text{K}^{-1})$ [9]. According to the manufacturer, the thermal properties can be investigated from strongly cryogenic temperatures to very high temperatures (approx. 1273 K) [8, 9].

He 2005 [6] also emphasizes the good accuracy of the method. As stated by the manufacturer, the thermal conductivity can be determined with an accuracy of $\pm 5\%$ [8–10]. Due to the complex mathematical model, it is not possible to calculate the measurement uncertainty, e.g., by using the GUM standard (propagation of uncertainties of contributing factors). The mentioned measurement accuracy of 5% was determined by Log and Gustafsson [10] by performing experiments at room temperature with several standard materials in a wide range of thermal conductivities ($0.02\text{--}200\text{ W}\cdot(\text{mK}^{-1})$) [10]. Furthermore, a general reproducibility of $\pm 2\%$ for the thermal conductivity (typically better than $\pm 1\%$ [9]), $\pm 5\%$ for the thermal diffusivity, and $\pm 7\%$ for the specific heat per unit volume is given [8].

Initially, the Hot Disk method was proposed by Gustafsson [11]. The applied sensor consists of a thin bifilar nickel spiral with a thickness of $\approx 10\ \mu\text{m}$ insulated with a layer of Kapton (total thickness of sensor: $60\text{--}80\ \mu\text{m}$), Mica (total thickness of sensor: $250\ \mu\text{m}$) or Teflon and has to be placed between two identical samples with at least one smooth surface each [8]. Principally, this sensor acts as a heater and temperature sensor (in form of a resistance thermometer) at the same time [6, 8, 10–12]. Depending on the size of the sample and its thermal properties, different sensor sizes with diameters ranging between $1\ \text{mm}$ and $59\ \text{mm}$ are available [8]. According to the manufacturer [8], Kapton sensors can be used for temperatures up to $300\ ^\circ\text{C}$, and Mica sensors for temperatures up to $1000\ ^\circ\text{C}$. However, it is not recommended to use Mica sensors at temperatures lower than $300\ ^\circ\text{C}$ since the higher insulation thickness reduces the measurement accuracy.

During the measurement time defined by the user a constant electric current with an input power P_0 is passed through the sensor generating a transient temperature field in the samples [6, 10–12]. Simultaneously the temperature change is recorded by measuring 200 resistance values [6, 8, 11, 12]. This recording of the resistance is performed by measuring the current and voltage variation using an electrical bridge [10, 12]. The time-dependent resistance $R(t)$ at the sensor during the electrical heating is described by [6, 8, 10–12]:

$$R(t) = R_0[1 + TCR \cdot \Delta T(\tau)] \quad (1)$$

where R_0 is the reference resistance before the temperature increase (at the initial sensor temperature T_0) and TCR is the temperature coefficient of resistance (in $1/\text{K}$), which describes the relative change of the resistance of the nickel spiral per Kelvin temperature change. $\Delta T(\tau)$ is the mean temperature increase of the sensor not recorded over the normal time t (from the beginning of the measurement), but over the dimensionless parameter $\tau = \left(\frac{t}{\Theta}\right)^{\frac{1}{2}}$ to account for the influences of the sensor geometry and material behavior. The characteristic time Θ is calculated by $\Theta = \frac{r^2}{a}$, where r is the radius of the sensor and a is the thermal diffusivity of the sample [6, 8, 10–12].

By measuring the resistance change $R(t)$ the temperature increase $\Delta T(\tau)$ at the sensor can be deduced. This strongly depends on the thermal properties of the material which can be identified by comparing recorded values of $\Delta T(\tau)$ with an idealized model for the heat conduction coming from the Hot Disk sensor [6, 12]. An approximate solution for the theoretical average temperature increases at the

sensor which is compared with a concentric and equally spaced ring source can be calculated by solving the transient heat conduction equation [6, 8, 10–12]:

$$\Delta T(\tau) = P_0 \left(\pi^{\frac{3}{2}} r \lambda \right)^{-1} D(\tau) \quad (2)$$

with the thermal conductivity of the material λ , the input power to the sensor P_0 and the dimensionless time function $D(\tau)$ which also includes the modified Bessel function. A detailed derivation of Eq. 2 is given by He [6] or Gustafsson [11].

Theoretically $\Delta T(\tau)$ consists of a small temperature drop across the insulation layer $\Delta T_i(\tau)$ and the temperature increase of the sample surface $\Delta T_s(\tau)$. However, $\Delta T_i(\tau)$ becomes constant after a short initialization period and can be excluded in the calculations [8, 10, 12]. Furthermore, a time correction is applied to correct the time delay between hardware and software by using τ_c instead of τ [12].

In an iteration or optimization process the time correction t_c and the thermal diffusivity a (and hence the dimensionless time Θ) are varied so that Eq. 2 is a linear relationship between $\Delta T(\tau)$ and $D(\tau)$, and the thermal conductivity can be received from the slope of the correlation [6, 8, 10, 12]. With the relation $\lambda = a\rho c_p$ the volumetric heat capacity can also be calculated [12].

From the received thermal diffusivity and the measurement time, the finally achieved probing or penetration depth of the heat flow into the specimen can be calculated by [8, 11, 12]: $\Delta p = 2(at)^{\frac{1}{2}}$. This should not exceed the distance between each point of the sensor and the outer boundaries to ensure an unhindered propagation of the heat flux. Since the probing depth should be in the range between the sensor radius and the sensor diameter, the sample must have at least a height of the sensor radius and a diameter of two times the sensor diameter [8].

As already mentioned, for the solution of Eq. 1 the knowledge of the temperature-dependent temperature coefficients of resistance (TCRs) of the nickel spiral at the respective measurement temperatures is required. The material nickel is selected because of its relatively large TCRs for a wide range of temperatures [8]. However, a special feature with nickel is the Curie point at about 358 °C, at which the magnetic conversion takes place and many properties of the material fundamentally change, including the TCR. In addition to a strong decrease in the TCR at this point, the temperature dependence of the TCR is not very pronounced anymore at higher temperatures (see Chap. 5.3, Fig. 4), which could both impair the evaluation of the thermal conductivity measurements.

Since the TCR has a nearly direct influence on the resulting calculated thermal conductivity, its exact knowledge is essential. In general, there are two different options for the determination of the TCR [8]: by recording the temperature-dependent sensor resistance or by comparing the measured thermal conductivity with a known reference value. Values for the TCR, which are determined by the manufacturer applying these methods at certain temperatures, are tabulated in the software; the TCR at the actual measurement temperature is then calculated by linear interpolation. However, as pointed out by Goetze *et al.* [4], the accuracy of

the given values might be limited; therefore, a redefinition of the TCRs has been sought.

3 Literature Research: Reference Materials

For the calibration and verification of the accuracy of a measurement system, appropriate reference materials are required. In the case of the Hot Disk method, these should especially serve to check the correctness of the TCRs to reliably determine the temperature-dependent thermal conductivity. Official institutes providing reference standards are for example the National Institute of Standards and Technology (NIST, USA), the National Physical Laboratory (NPL, UK) or the Institute for Reference Materials and Measurement (IRMM, Europe, before BCR: Community Bureau of Reference). The most important criteria for a reference material are homogeneity, isotropy, and stability so that properties are reproducible over several measurements without modification or altering of the material.

In Table 1 an overview of available reference materials for the thermal conductivity is given. The materials are either already certified by an above-mentioned institute or investigated intensively in an intercomparison, and accordingly taken into consideration as a reference material. Furthermore, for some of the materials, other researchers have recommended additional reference data, mostly extending the certified temperature range.

Besides the tabulated materials, several further suggestions for potential reference standards can be found in the literature. For example, Wu *et al.* [30] presented a promising high-temperature (150 °C–650 °C) thermal conductivity reference material, namely a high-density calcium silicate (HDCaSi-N). Its long-term stability remained under investigation and the results of an intercomparison should be presented in the future. Furthermore, during research Platinum [31] or the Polyimide Vespel SPI [32] were also found as potential reference materials. In addition, Jannot *et al.* [33] used LUX500 & 800 (calcium silicate board) as well as Quartzel® (low-density fibrous felts) for testing (calibration and validation of the results) the newly developed Comparative FluxMetric method to measure the apparent thermal conductivity of insulation materials up to 600 °C.

In summary, a wide range of reference materials of different types (plastics, glasses, ceramics, metals) is available in the temperature range below 100 °C. However, with an increasing application temperature up to 1000 °C, only few ceramics or metals feature sufficient high-temperature stability. Furthermore, the intercomparisons have shown that with increasing temperature the measurement uncertainty often increases, which could preclude the certification as an official reference material [20, 26, 29]. A reason for the increased uncertainty might be that at high temperatures only a few measurement methods, like the Laser-Flash method, are applicable. However, as an indirect measurement method, the measurement uncertainties of the thermal diffusivity, the heat capacity, and the density add up reducing the total measurement accuracy of the thermal conductivity [29]. Thus, the research revealed the still existing need for suitable reference materials for thermophysical properties like the thermal conductivity at high temperatures as well as for suited measurement

Table 1 Overview of certified and potential reference materials for the thermal conductivity including their temperature and thermal conductivity range

Reference material	Temp. range in °C	Temp. range in K	Thermal conductivity in $W \cdot (mK^{-1})$	Certification/ Reference
GLASS				
Pyrex 7740 (Borosilicate glass)	- 75 to 195	198–468	0.954–1.326	IRMM: BCR-039 [13, 14]
	- 241 to 469	32–742	0.190–1.728	Recommended values [15]
BK7 (Borosilicate Crown Glass)	- 95 to 500	178–773	0.93–2.33	Intercomparison [16]
	- 194 to 500	79–773	0.395–1.459	Recommended values [15]
Resin-bonded glass fiber board	- 10 to 50	263–323	0.028–0.035	IRMM: IRMM-440 [14, 17]
Glass fiber board	7–87	280–360	0.030–0.040	NIST: SRM-1450e [18]
Fibrous glass blanket	- 173 to 57	100–330	0.010–0.050	NIST: SRM 1451/1452 [14, 19] (for thermal resistance, but thermal cond. data provided)
CERAMIC				
Silcal 1100	27–827	300–1100	0.085–0.173	Intercomparison [20]
Pyroceram 9606	25–752	298–1025	4.061–2.835	IRMM: BCR-724 [21]
	17–1002	290–1275	3.940–2.750	Recommended values [15]
PLASTIC				
PMMA	- 70 to 80	203–353	0.181–0.201	Intercomparison [22]
	0–80	273–353	0.188–0.198	NPL (Transfer Standard) [13]
	- 178 to 90	95–363	0.144–0.196	Recommended values [15]
Thin expanded polystyrene board	8–40	281–313	0.032–0.035	NIST: SRM-1453 [14, 23]
METAL				
AISI 304 (SS 1.4301)	100–500	373–773	15.9–22.3	NPL: 2S09 [24]
	20–273	293–546	14.32–18.87	Recommended values [15]
AISI 310 (SS 1.4841)	50–500	323–773	13.2–21.2	NPL: 2S89 [25]
	50–500	323–773	21.0–24.9	replaced by AISI 304 [24]
AISI 430 (SS 1.4016)	50–500	323–773	21.0–24.9	NPL [25]

Table 1 (continued)

Reference material	Temp. range in °C	Temp. range in K	Thermal conductivity in $W \cdot (mK^{-1})$	Certification/ Reference
X10NiCrMoTiB1515 (SS 1.4970)	20–1000	293–1273	13.49–27.07	Intercomparison [26]
Inconel 600	50–750 – 125 to 1000	323–1023 148–1273	13.3–27.2 10.961–29.689	NPL[25] Recommended values [27]
Nimonic75	100–500	373–773	13.9–21.6	NPL: 2109 [28]
Pure iron	50–500	323–773	12.8–21.1	NPL [25]
Interstitial free iron	20–1000	293–1273	82.5–28.4	Intercomparison [29]
Multi-phase iron	20–1000	293–1273	71.9–27.9	Intercomparison [29]
	20–1000	293–1273	32.8–25.6	Intercomparison [29]

methods. Even though, some appropriate reference materials presented in detail in the next chapter could be identified for the current investigation.

4 Materials and Methods

4.1 Reference Materials and Data

From Table 1 three reference materials with different thermal conductivities were selected for the planned investigations. The opaque glass–ceramic **Pyroceram 9606** is produced by Corning Inc. (USA) and was certified as a reference material for thermal conductivity and thermal diffusivity by the IRMM in 2007 with an uncertainty of 6.5 % and 6.1 % [21]. It consists of SiO₂ (56 %), Al₂O₃ (19.6 %), MgO (14.9 %), TiO₂ (8.6 %), and smaller amounts of several other oxides [15]. The temperature-dependent thermal conductivity in the range between 298 K and 1025 K was certified as [21]:

$$\lambda = 2.332 + \frac{515.1}{T} \quad (3)$$

Furthermore, Antoniadis *et al.* [15] provided a new reference correlation for the temperature range from 290 K to 1275 K for this material:

$$\lambda = 6.021828 - 3.03444657 * T_r + 1.24720006 * T_r^2 - 0.24519953 * T_r^3 + 0.01822542 * T_r^4 \text{ with } T_r = \frac{T}{273.15} \quad (4)$$

The latter resulted in a better agreement with own measurements at room temperature (see Chap. 5.1) and was therefore used for the evaluation of the temperature-dependent measurements. However, the deviations between the two reference data [15, 21] are always less than 2 % at higher temperatures.

A material with a low thermal conductivity ($<0.5 \text{ W} \cdot (\text{mK}^{-1})$) suitable for the temperature range up to 750 °C (1023 K) is **Silcal 1100**. The material was already applied by Goetze *et al.* [4] for their TCR improvement. Silcal 1100 is a commercially available, highly porous calcium silicate produced by CALSITHERM Silikatbaustoffe GmbH (Germany) and consists of 46 % to 47 % CaO and 44 % to 45 % SiO₂ [20]. Although it is not officially certified yet, the intercomparison by Ebert and Hemberger [20] provides reliable reference data. The thermal conductivity from 300 K up to 1100 K is tabulated with a relative uncertainty between 3.5 % and 7 % (increasing with temperature) and the following third-order polynomial function is given for approximation [20]:

$$\lambda = -9 * 10^{-12} * T^3 + 6 * 10^{-8} * T^2 + 4 * 10^{-5} * T + 0.0674 \quad (5)$$

In consultation with the authors, it is pointed out, that the reference equation given by Ebert and Hemberger [20] has a sign error, corrected in Eq. 5. Furthermore,

an own fit of the tabulated reference data was set up to obtain the exact coefficients of the polynomial function.

Inconel 600 is a nickel–chromium alloy and was certified by the NPL Clark and Tye [25] publishing reference values for a temperature range from 20 °C to 750 °C and setting up a correlation for an interval from 50 °C to 750 °C:

$$\lambda = 12.479 + 1.648 * 10^{-2}\theta + 3.741 * 10^{-6}\theta^2 \quad (6)$$

Since the supply of the reference material was exhausted and the previous measurement apparatus was no longer available, a new certification by the NPL Wu *et al.* [28] for a temperature range from 100 °C to 500 °C was performed. Reference values and a new correlation with an overall uncertainty of 4.8 % were presented deviating only 2 % from the previous NPL values [28]:

$$\lambda = 12.158 + 1.6638 * 10^{-2}\theta + 4.3208 * 10^{-6}\theta^2 \quad (7)$$

Additionally, measurements extending the temperature range were conducted by Blumm *et al.* [27], who determined the thermal conductivity by measuring the thermal diffusivity, the specific heat and the density from – 125 °C to 1000 °C. However, the range from 550 °C to 700 °C should be critical due to the formation of Ni₃Cr clusters. Because of the limited temperature range of the new NPL certification, own measurements will be compared with previous NPL certification values Clark and Tye [25] both at room temperature and higher temperatures. This seems to be valid since extrapolation of the newly certified values leads to nearly identical results with highest deviations of 2.6 % at 0 °C and deviations less than 1 % for temperatures > 400 °C. Furthermore, at room temperature tabulated values of Blumm *et al.* [27] were considered as reference.

4.2 Room-Temperature Measurements

The measurements in the current investigation are performed with a Hot Disk TPS 2500 S measurement system by Hot Disk AB. First, extensive measurements at room temperature (RT, approx. 23 °C) using Kapton sensors (KXXXX) should prove the suitability of the selected materials as reference materials. Furthermore, it should be demonstrated if the sensor size or connection of the sensor influences on the measurement result. For this purpose, sensors with three different diameters respecting the material, the penetration depth and the available sample size were considered: 6.4 mm (K5465), 12.8 mm (K5501), and 29.2 mm (K4922L). For Pyroceram 9606 and Inconel 600, the K5464 and K5501 sensors were used and for Silcal 1100, the K5501 and K4922L sensors (see Fig. 1). Sufficiently large samples to meet the sample size requirements (see Chap. 2) were used. At room temperature, there are two possibilities to connect the sensor to the measurement device: the sensors can have a cable connected directly (RT-C) or they can be installed in a special room-temperature sensor holder (RT-SH), see Fig. 1. If available, both options with each sensor size were tested. Furthermore, the measurement value at room temperature, received in context with the subsequent temperature-dependent measurements using a high-temperature sensor holder (HT-SH, see Fig. 2), was

Fig. 1 Kapton sensors used for the room-temperature measurements with Silcal 1100: (1)—cable connected (RT-C) K4922L ($d=29.2$ mm), (2)—K4922L ($d=29.2$ mm) connected by room-temperature sensor holder (RT-SH), (3)—cable connected (RT-C) K5501 ($d=12.8$ mm)

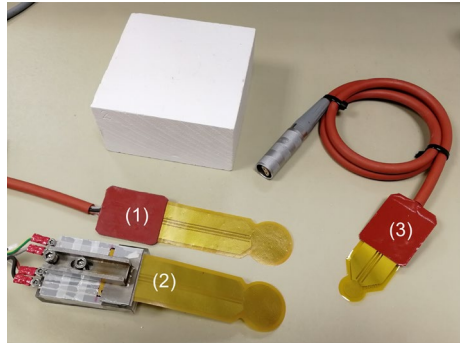


Fig. 2 Hot Disk TPS 2500S with high-temperature setup (installation for the muffle furnace): Mica sensor M4922L ($d=29.2$ mm) in the high-temperature sensor holder (HT-SH) on the lower Silcal 1100 sample



considered for comparison. An overview of the measurements performed for each material is presented in Table 2.

The room-temperature measurements were conducted at temperatures between 22.9 °C and 24.4 °C and compared to the reference values, although these were given for temperatures between 20 °C and 27 °C. This seems reasonable since all materials do not feature a strong temperature dependence in this range. For each sensor and material, adequate measurement parameters were selected to fulfill $0.33 < \tau < 1$ [8]. A time corresponding to the thermal conductivity of the material (10–40 min) was waited between the measurements to obtain again a stable temperature field as an initial condition, which was checked by the systems temperature drift measurement. For each setup of sensor and reference material, measurements were performed for all combinations of the faces of the two reference specimens.

Table 2 Summary of conducted experiments at different temperatures with the selected sensors and their connections (C—sensor with an integrated cable, RT-SH—room-temperature sensor holder, HT-SH—high-temperature sensor holder in the furnace)

Material	Temperature (range) in °C	Sensor (sensor diameter in mm)	Connection
Pyroceram 9606	RT	K5501 (12.8)	C, RT-SH
	RT	K5465 (6.4)	RT-SH
	RT-250	K5501 (12.8)	HT-SH
	200–750	M5082 (12.8)	HT-SH
Silcal 1100	RT	K4922L (29.2)	C, RT-SH
	RT	K5501 (12.8)	C, RT-SH
	RT-250	K4922L (29.2)	HT-SH
	200–750	M4922L (29.2)	HT-SH
Inconel 600	RT	K5501 (12.8)	C, RT-SH
	RT	K5465 (6.4)	RT-SH
	RT-250	K5501 (12.8)	HT-SH
	200–600	M5082 (12.8)	HT-SH

Thus, a statement about the homogeneity of the materials and the surfaces can be made based on the standard deviations.

4.3 Temperature-Dependent Measurements

In the second part of the present investigation, temperature-dependent measurements are carried out to verify the correctness and accuracy of both, the TCRs provided by the manufacturer and the values revised by Goetze *et al.* [4]. If necessary, another adjustment of the values based on the measurements with the selected reference material should be performed.

These measurements were performed in a muffle furnace with an integrated high-temperature sensor holder (HT-SH, see Fig. 2). Before heating up, the oven was always evacuated and flushed with nitrogen several times to remove oxygen which would lead to an amplified sensor destruction at higher temperatures. From room temperature up to 250 °C a Kapton sensor (KXXXXX) was used and a second series with the corresponding Mica sensor (MXXXXX) was performed from 200 °C to 750 °C (see Table 2), in each case with temperature steps of approx. 50 K. Theoretically, the manual recommends performing measurements up to 300 °C with Kapton sensors [8], as this leads to more accurate results due to the thinner insulating layer. However, own measurements have shown an increased wear on the Kapton sensors at temperatures > 250 °C, so the use of Mica sensors at temperatures lower than 300 °C is considered. Furthermore, although the Mica sensor should be able to be used up to 1000 °C [8], internal preliminary investigations have indicated high damage to the sensors at these temperatures leading to unreliable measurement results. Therefore, the maximum testing temperature was limited to 750 °C. However, depending on the duration of the measurements, even at this temperature there is severe wear on the sensors, which in some cases does not allow them to be reused.

As already stated by Goetze *et al.* [4], around the Curie temperature the TCRs show a highly non-linear behavior. For this reason, additional measurements between 350 °C and 400 °C were conducted in 10 K steps. The largest possible sensor for each sample dimension (12.8 mm—K5501/M5082 or 29.2 mm—K4922L/M4922L) and one sample face combination were selected to perform the measurements. At each temperature, five measurements were performed and averaged. The standard deviation for these measurements was mostly below 1 % and only rarely extended up to 3 %. The material Inconel 600 was measured only up to 600 °C since at higher temperatures deviations from the expected course possibly due to specific transformation or reaction processes of the material like the already mentioned formation of Ni₃Cr clusters [27] have occurred.

Since the set furnace temperature deviated by 10–15 K from the actual temperature in the sample, the temperature steps were not perfectly matched. The actual temperature used for further evaluation was determined by additional thermocouples. In the case of the soft Silcal 1100 material, a thermocouple could be placed between the two samples near the sensor. For the two other materials, the temperatures above and below the samples were measured and averaged.

5 Results

5.1 Room-Temperature Measurements

The results of the room-temperature measurements are summarized in Table 3. All values were calculated by using the TCRs provided by the manufacturer. The standard deviations of all materials and sensors are always below 1.5 %, often even much lower demonstrating the homogeneity of the materials themselves and their surfaces and confirming the suitability of the samples as reference materials. Furthermore, the independence of the exact sensor position or face combination allows the selection of just one face combination of the specimens for the temperature-dependent measurements.

Comparing the different sensors and sensor connections, the deviations to the results obtained with the cable-connected K5501 (C) sensor (reference case) are always limited to ± 3 %, which is within the range of the measurement accuracy of ± 5 % given by the manufacturer (see Chap. 2) [8–10]. There is no clear tendency for one specific sensor to result in a continuously higher or lower measured thermal conductivity. Deviations might result from the contact resistance between the material and the sensor and the sensor holder, the contact resistance between the material and the sensor or slight instabilities of the temperature field before the measurement.

The deviations from the literature reference values are in general limited to a few percent. Only in the case of Pyroceram 9606, higher deviation (5.74 %) from the official certified thermal conductivity occurs which is reduced to 1.65 % when compared to the value proposed by Antoniadis *et al.* [15]. Since the measurement results given in Table 3 are all calculated using original TCRs given by

Table 3 Thermal conductivities and calculated deviations of selected reference materials; measured by the Hot Disk TPS 2500 S with different sensors at room temperature

Sensor type/source of value	Thermal conductivity in $W \cdot (mK^{-1})$	Standard deviation in %	Deviation from K5501 (C) in %
Pyroceram 9606			
K5501 (C)	3.84	0.05	
K5501 (RT-SH)	3.88	0.11	1.04
K5465 (RT-SH)	3.90	0.48	1.56
K5501 (HT-SH)	3.86		0.52
Reference 1	4.06 [21]		5.74
Reference 2	3.90 [15]		1.65
Silcal 1100			
K5501 (C)	0.0847	0.18	
K5501 (RT-SH)	0.0848	0.57	0.12
K4922L (C)	0.0856	1.41	1.06
K4922L (RT-SH)	0.0861	1.35	1.65
K4922L (HT-SH)	0.0827		− 2.36
Reference 1	0.0846 [20]		− 0.12
Inconel 600			
K5501 (C)	12.63	0.77	
K5501 (RT-SH)	12.94	0.06	2.43
K5465 (RT-SH)	12.26	1.34	− 2.95
K5501 (HT-SH)	12.70		0.54
Reference 1	12.69 [27]		0.49
Reference 2	12.80 [25]		1.33

the manufacturer, it can be concluded that at room temperature these values are appropriate and deliver correct results independent of the sensor used for the measurements. In contrast, the suggested correction of the TCR by Goetze *et al.* [4] (0.00499 1/K instead of 0.0047 1/K) would increase the deviations to the reference values to 5–6 %.

5.2 Temperature-Dependent Measurements

The measurements in the range from approx. 20 °C to 750 °C were evaluated using both, the original TCRs given by the manufacturer (Orig) and the values proposed by Goetze *et al.* [4] (Goe). The resulting thermal conductivities as well as the selected reference values (see Chap. 4.1) are plotted in Fig. 3.

As already proved in Chapter 5.1, at room temperature a good agreement between the reference values and the measurements with the Kapton sensors using the TCRs provided by the manufacturer is achieved for all materials. With increasing temperature up to 250 °C, the reference values become slightly overpredicted by the measurements by a few percent, but the manufacturer's TCRs

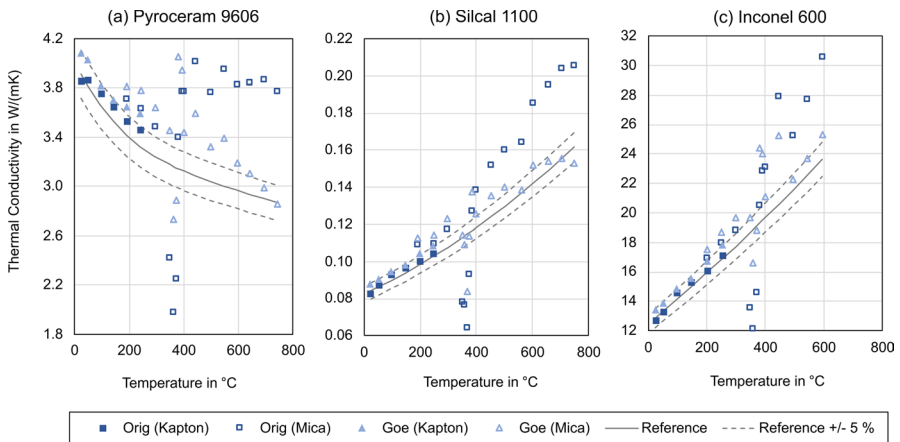


Fig. 3 Thermal conductivities of (a) Pyroceram 9606, (b) Silcal 1100, and (c) Inconel 600 from 20 °C to 750 °C: Measurement results calculated with the original TCRs given by the manufacturer (Orig) and the values proposed by Goetze *et al.* [4] (Goe), and reference curves [15, 20, 25]

are still appropriate in this range. At 200 °C and 250 °C, measurements have been conducted with both sensor types revealing that the thermal conductivities determined with the Mica sensors are 5–10 % higher than the values determined with the Kapton sensors and have thus a higher deviation from the reference. This could be because of the limited accuracy of the Mica sensor in this temperature range or an influence of the insulation layer on the measurement result.

Applying the suggested TCRs of Goetze *et al.* [4] at temperatures < 350 °C deviations to the reference values are continuously increased by up to 5 %. Around the Curie point (350–400 °C), the transformation of the materials evokes a strong, non-linear change of the TCR (see Chap. 5.3). For this reason, the manufacturer recommends not to perform measurements in the range of 300–420 °C [8]. Applying a linear interpolation to TCRs stored in the software would lead to average deviations of up to 44 % and the results would clearly deviate from the expected course. By defining a TCR at 350 °C, clearly differing from the value that would be received by linear interpolation with the original TCR, Goetze *et al.* [4] narrowed the range of strongly unreliable measurement results to 360–400 °C with deviations of always below 30 %. Above the Curie temperature, the manufacturer's TCRs lead to highly unreliable measurement results (deviations of up to 36 %), which has already been noted by Goetze *et al.* [4]. Applying their revised values, the deviations can be significantly reduced. A very good agreement with the reference data is achieved at temperatures above 600 °C. In contrast, the remaining higher deviations at 450 °C imply that the TCR resulting from linear interpolation at this temperature is still not correct and disagrees with the real behavior of nickel.

In summary, the corrected TCRs of Goetze *et al.* [4] significantly reduce the deviations of the experimentally determined thermal conductivity from the

reference around and above the Curie temperature. However, deviations still often exceed an acceptable level of $\pm 5\%$, corresponding to the measurement accuracy given by the manufacturer and are strongly changing with temperature indicating an incorrect progression of the nickel TCRs. Below the Curie temperature, the revised TCRs even lead to higher deviations for all materials than the original TCRs. Thus, a further modification of the values is suggested.

5.3 Optimization of the TCRs

In the optimization procedure, at each measured temperature the TCR was adapted so that the average deviations of all materials from their respective reference values were minimized. As discussed in Chapter 5.2, at 200 °C and 250 °C, measurements with Kapton and Mica sensors at the same temperature led to differences in the results of approx. $\pm 5\%$. To avoid the aging and delamination of the Kapton sensors because of the elevated temperatures and to guarantee a long sensor usage, it is recommended to use Kapton sensors at only up to 200 °C and Mica sensors at higher temperatures. Thus, only the respective values were considered for the optimization. All in all, it must be noted that this adjustment procedure of the TCRs also compensates for measurement errors and inaccuracies, which might not result from inaccurate TCRs, but are consistent for all materials and sensors.

The resulting optimized TCRs are summarized in Table 4. Figure 4a shows in comparison the temperature-dependent original TCRs given by the manufacturer (Orig), the ones proposed by Goetze *et al.* [4] (Goe) and the newly optimized values (Opt). In each case, the unreliable measuring range near the Curie temperature is marked by a dotted line. Figure 4b presents the percentage deviations of the newly determined TCRs from the previous ones.

As emerged previously, below 200 °C the manufacturer's values were largely confirmed, while deviations to the TCRs of Goetze *et al.* [4] amount to approx. -5% . With increasing temperature, the deviation from the original values increases to up to -25% , whereas at temperatures above 500 °C, the new TCRs differ by a maximum of approx. $\pm 5\%$ from the ones proposed by Goetze *et al.* [4]. However, significant deviations from these values, partially of up to more than -15% ,

Table 4 Optimized TCRs based on the measurements with the reference materials

T in °C	TCR	T in °C	TCR	T in °C	TCR	T in °C	TCR
20	4.765E-03	90	4.139E-03	300	3.17042E-03	450	9.3841E-04
30	4.676E-03	100	4.064E-03	350	3.36288E-03	500	8.1024E-04
40	4.569E-03	110	4.009E-03	360	3.51900E-03	550	7.4680E-04
50	4.468E-03	120	3.954E-03	370	3.23533E-03	600	6.5855E-04
60	4.385E-03	150	3.794E-03	380	1.92077E-03	650	6.2157E-04
70	4.303E-03	200	3.570E-03	390	1.46061E-03	700	5.8265E-04
80	4.221E-03	250	3.30664E-03	400	1.25905E-03	750	5.6769E-04

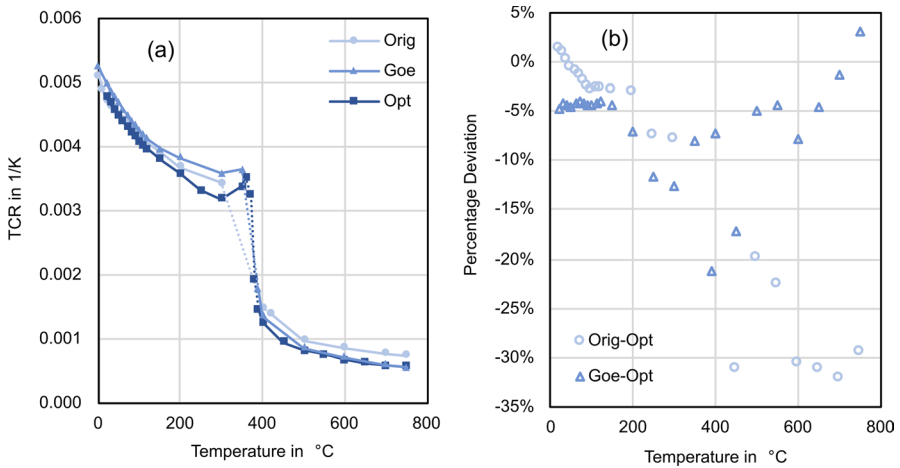


Fig. 4 (a) Temperature-dependent TCRs: Original TCRs given by the manufacturer (Orig), TCRs proposed by Goetze *et al.* [4] (Goe) and newly optimized values (Opt), (b) Percentage deviations between optimized TCRs and previous ones

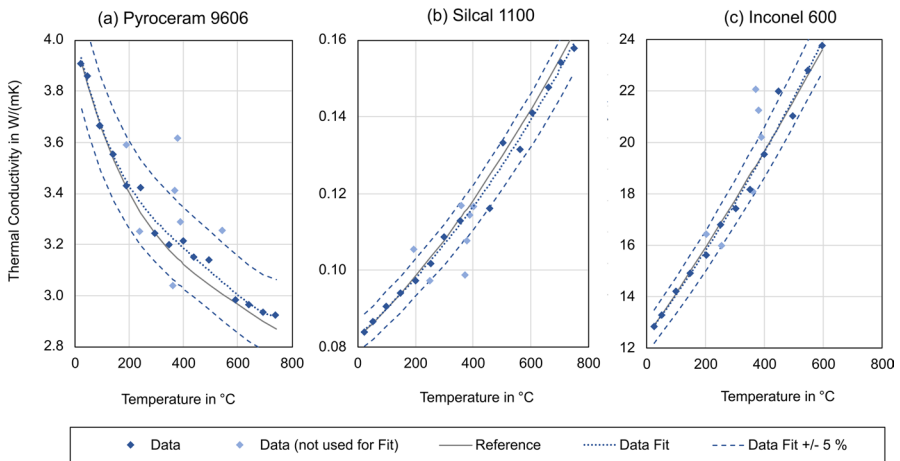


Fig. 5 Thermal conductivity of (a) Pyroceram 9606, (b) Silcal 1100, and (c) Inconel 600 from 20 °C to 750 °C: Measurement results calculated with the newly optimized TCRs, fitted curves (+/- 5 %), and reference curves [15, 20, 25]

occur from 200 °C to 450 °C. Around the Curie temperature (300–400 °C), a further refinement of the curve progression, showing the highly nonlinear behavior of the TCRs in this range, was achieved with the optimization.

In Fig. 5 the thermal conductivities of the three materials calculated with the newly optimized TCRs are plotted. A curve fitting (dotted lines in Fig. 5) with the same function type as given for the reference data (see Chap. 4.1) was performed excluding the values measured with the not recommended sensor at 200 °C and

250 °C, the values in the Curie range (360 °C to 390 °C) or isolated outliers (light blue points).

A very good agreement is achieved between the fitted and the reference curve for all the materials with deviations of always less than 2 %. Up to 200 °C, each reference curve is nearly perfectly matched. Excluding the Curie range, the single measurement points usually scatter less than ± 5 % around the fitting curve (dashed lines in Fig. 5), which corresponds to the measurement accuracy stated by the manufacturer [8]. In general, measurements with the Mica sensors exhibit a greater spreading around the fitting curve, which might be caused by the increased sensor insulation thickness and reduced flexibility of the sensors, leading to worse contact between the samples and the sensor. Furthermore, the low TCRs above 400 °C could decrease the measurement accuracy.

Deviations significantly higher than 5 % only occur at temperatures around 370 °C and 380 °C caused by the strong temperature-dependency of the TCR. A very accurate temperature measurement would be necessary but can be guaranteed with difficulty, if the sample should not be influenced or destroyed by the insertion of thermocouples. Even though the deviations could be reduced to a maximum of 16 %, it is not recommended to perform measurements in this range.

In summary, it is suggested to use the newly proposed TCRs for temperature-dependent measurements of up to 750 °C with a Hot Disk measurement system instead of the TCRs originally given by the manufacturer, as the measurement accuracy, especially at temperatures ≥ 200 °C, can be greatly increased. Deviations between the original TCRs and the newly optimized ones extend up to -25 % and even to the already revised values of Goetze *et al.* [4] deviations higher than 15 % occur. These deviations have an almost direct effect on the determined thermal conductivities. Using the newly proposed TCRs, a maximum deviation from the reference values of the investigated materials of 2 % could be achieved with an accuracy of approx. ± 5 %.

6 Summary

In this study, the Hot Disk technique for the measurement of the thermal conductivity up to 750 °C is investigated and improved, contributing to a more reliable determination of thermophysical material data at higher temperatures. In particular, the challenge of inaccurate TCRs pointed out previously by Goetze *et al.* [4] is addressed. As for the calibration and validation of all measurement methods and systems, adequate reference materials are required. A literature review provides an overview of currently available reference materials for the thermal conductivity and confirmed that while at temperatures < 100 °C numerous reference materials of different types (glass, ceramic, plastic, metal) are offered, up to 1000 °C the availability of appropriate reference materials for the thermal conductivity is still strongly limited.

For the present investigations, three materials with completely different thermal conductivities were selected, namely Inconel 600, Pyroceram 9606, and Silcal 1100. Room-temperature measurements with different Kapton sensors showed the

suitability of the reference samples as well as the independence of the measured thermal conductivity from the sensor. Temperature-dependent measurements with Kapton (RT–250 °C) and Mica (200–750 °C) sensors revealed the inappropriateness of the TCRs proposed by the manufacturer, especially at high temperatures, and also showed the necessity to further modify the TCRs revised by Goetze *et al.* [4].

With the newly optimized TCRs, a very good agreement between the measurements and the reference data was achieved. The deviation of the data fit to the reference is always lower than 2 % and the scattering of the single measurement points around the fitted curve is generally limited to ± 5 %, which corresponds to the measurement accuracy given by the manufacturer. Especially in the temperature range from 200 °C to 500 °C, significant improvements could be achieved compared to the application of previous TCRs. Hence, to enhance the measurement accuracy it is suggested to use the newly presented set of TCRs for the measurements with a Hot Disk System for temperature of up to 750 °C. However, measurements around the Curie temperature (approx. between 370 °C and 380 °C) should still be skipped. Furthermore, it is suggested to use Kapton sensors at only up to 200 °C and Mica sensors at higher temperatures up to a maximum of 750 °C.

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Declarations

Competing interests The authors have no relevant financial or non-financial interests to disclose.

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