Int J Thermophys
DOI 10.1007/s10765-015-1863-x

Critical Review of Industrial Techniques for Thermal-Conductivity Measurements of Thermal Insulation Materials

Ulf Hammerschmidt · Jacques Hameury · Radek Strnad · Emese Turzó-Andras · Jiyu Wu

Received: 7 October 2014 / Accepted: 2 March 2015

Abstract This paper presents a critical review of current industrial techniques and instruments to measure the thermal conductivity of thermal insulation materials, especially those insulations that can operate at temperatures above 250 °C and up to 800 °C. These materials generally are of a porous nature. The measuring instruments dealt with here are selected based on their maximum working temperature that should be higher than at least 250 °C. These instruments are special types of the guarded hot-plate apparatus, the guarded heat-flow meter, the transient hot-wire and hot-plane instruments as well as the laser/xenon flash devices. All technical characteristics listed are quoted from the generally accessible information of the relevant manufacturers. The paper includes rankings of the instruments according to their standard retail price, the maximum sample size, and maximum working temperature, as well as the minimum in their measurement range.

U. Hammerschmidt (✉)
Physikalisch-Technische Bundesanstalt, Braunschweig, Germany
e-mail: ulf.hammerschmidt@ptb.de

J. Hameury
Laboratoire National de Métrologie et d'Essais, Paris, France

R. Strnad
Český Metrologický Institut Brno, Brno, Czech Republic

E. Turzó-Andras
Magyar Kereskedelmi Engedélyezési Hivatal, Budapest, Hungary

J. Wu
National Physical Laboratory, Teddington, Middlesex, UK

Published online: 23 April 2015

Keywords Guarded heat-flow meter · Guarded hot-plate apparatus · High temperature · Laser-flash instrument · Measuring device · Thermal conductivity · Thermal insulation material · Transient hot wire · Transient plane source

1 Introduction

Due to continuously increasing requirements on the efficiency of power plants, their working temperatures are designed ever higher. With higher working temperatures, however, heat losses of new or upgraded plants can easily increase in a way that efficiency gains likewise erode. High-temperature thermal insulation materials (HT-TIMs) with their low thermal conductivity and high heat resistance can effectively cope with this problem. That is the reason that industry develops new functional HT-composites as HT-TIMs with great effort. But, so far, the metrology of the relevant thermal transport properties cannot keep pace with the development of new products.

The measurement of TIMs at temperatures up to some 100 °C, e.g., for building purposes, is a daily routine for relevant testing facilities. However, these laboratories face formidable challenges when HT-TIMs for industrial applications are to be analyzed; only a few types of measuring instruments for temperatures above 250 °C are commercially available. That is why, at present, nearly all such instruments are made in-house (see Sect. 3.1.1) and, therefore, are distinct from each other. Most regrettably, the existing various instruments have shown a significant level of scatter, sometimes even more than 100 % [1,2]. The departure of a key material property in an engineering design can be ruinous; it can even be catastrophic when a HT-TIM fails as an aerospace component or as the thermal protection material of a structural fire safety system.

The present paper attempts to critically review industrial techniques for thermal conductivity measurements of HT-TIMs. The upper limit of the operating temperature of the measuring techniques considered here is drawn at 800 °C because the majority of the CEN (European Committee for Standardization) product standards for industrial and/or technical thermal insulation state that the product operating temperatures are within 800 °C. The maximum thermal conductivity of the HT-TIMs at this (application) temperature should be around 0.2 $\mathrm{W{\cdot}m^{-1}{\cdot}K^{-1}}$.

The objective of this paper is fairly challenging because the relevant industry is very small and inside information is hard to be gained.

2 Theory

2.1 Energy Conversion: Efficiency and Thermal Conductivity

The first law of thermodynamics states that the total amount of energy is conserved regardless of converting energy from one of its various forms to another. For instance, combusting oil, gas, or coal in a heat engine turns chemical energy, via thermal (heat: Q) and kinetic energy (work: W), finally to electrical energy.

The second law of thermodynamics states that, whenever energy is converted, a certain amount of it is lost to 'inefficiency.' The measure for this inefficiency is entropy, $\Delta S = Q/T$ (T denotes temperature). For an isolated system, entropy can only rise.

The *least* inefficient conversion process follows the imaginary Carnot cycle. This process is considered to operate between a hot reservoir at T_H and a cold one at T_C. Its *efficiency*, $\eta = W/Q = (1 - T_C/T_H)$, is the best possible. Therefore, (real) heat engines with their cold reservoir generally at ambient temperature (300 K) operate more efficiently at a higher upper temperature T_H. That is why working temperatures of future heat engines are designed ever higher; existing ones are markedly upgraded. However, the higher is T_H, the larger potential heat losses of the warm reservoir to the cold one (ambient) occur due to stray heat flows. Whenever heat is transferred between a hot and a cold reservoir, e.g., via a heat engine or merely via conduction, convection, and/or radiation, entropy is produced alongside. To keep energy efficient, the production of entropy has to be minimized, e.g., by thermal protection.

Due to their low thermal conductivity, thermal insulation materials help effectively slowing down entropy production through heating and cooling of buildings for many years. These materials, however, are generally limited in their service temperature. They cannot be used for heat engines of high efficiencies, η, such as, e.g., modern power plants and gas turbines with their high working temperatures T_H and accordingly low entropy production. Here, as well as for other high-temperature (HT) applications such as, e.g., long-distance heating ducts or furnaces of process plants, special HT thermal insulations are needed because of their high-temperature resistance.

Fundamentally, the lower the thermal conductivity of a TIM and the higher its service temperature, its entropy performance is better.

2.2 Basic Characteristics of Thermal Protection Materials

Independent of temperature, a fictionally ideal TIM should be able to completely insulate heat regardless of the relevant thermal transport mechanism(s): conduction, convection, and radiation. This implies that (1) thermal conductivity ($\lambda \to 0$), (2) heat transfer coefficient ($\alpha \to 0$), and (3) transmittance ($\tau \to 0$) should vanish. Over the entire service time, a TIM should be dimensionally, structurally, and chemically stable without any aging in these respects. A HT-TIM additionally has to be non-flammable and able to withstand high operating temperatures.

In practice, the above demands are unsurpassed and can be met by a porous material. The matrix should be of a low conducting solid that can withstand high temperatures and will, as good as possible, meet the other above mechanical requirements. The pores should be permanently evacuated or contain a low conducting (inert) gas [3–5]. They should be small enough to impede the onset of convection and be closed in order to prevent the TIMs from absorbing moisture. At present, the best compromise of all these requirements is given by advanced fibrous, foam, and aerogel-based (porous) composites, multilayered materials and/or powder insulations. Also, calcium silicate, known from passive fire protection boards, is a potential candidate material for HT-TIMs.

2.3 Basic Features of Thermal Conductivity Instruments for TIMs

According to the above-mentioned second law of thermodynamics, the local production of entropy per unit volume by thermal conduction, $\mathrm{d}S_{TC}^{(V)}/\mathrm{d}t = \dot{S}_{TC}^{(V)}$, is directly

proportional to the local temperature gradient, $\nabla T\,(\vec{r})$. For a one-dimensional case, $|\nabla T\,(\vec{r})| = \partial T/\partial z$,

$$\dot{S}_{\mathrm{TC}}^{(V)} = -\frac{1}{T^2} q_z \frac{\partial T}{\partial z} \tag{1}$$

is valid. Here, the density of the heat-flow rate, Φ, per unit area A is denoted as q. According to Fourier's phenomenological (first) law, the latter quantity, q, itself is proportional to the local temperature gradient of a homogeneous, isotropic medium and thermally inert:

$$q = -\lambda \frac{\partial T}{\partial z}. \tag{2}$$

The thermal conductivity (TC), λ, actually is a constant of proportionality and, as such, independent of temperature and position. Substituting Eq. 2 into Eq. 1 furnishes a quadratic dependence of the entropy production from the local temperature gradient:

$$\dot{S}_{\mathrm{TC}}^{(V)} = \frac{\lambda}{T^2} \left(\frac{\partial T}{\partial z} \right)^2. \tag{3}$$

Thus, for a given heat conduction situation, e.g., in a power plant, the production of entropy can be minimized by minimizing the material parameter λ.

Equation 2 is a special case (Poisson equation), $T = T(\vec{r}, t = 0)$, of Fourier's more general (second) law, defining the thermal diffusivity (TD) a. From the first law of thermodynamics, $\dot{Q}^{(V)} + \partial q/\partial z = 0$, it follows with Eq. 2 that

$$\dot{Q}^{(V)} = \frac{\partial Q^{(V)}}{\partial T} \frac{\partial T}{\partial t} \quad = \quad -\frac{\partial}{\partial z} q = -\frac{\partial}{\partial z} \left(-\lambda \frac{\partial T}{\partial z} \right). \tag{4}$$

With $\partial Q^{(V)}/\partial T = \rho c_p$, where ρ and c_p denote the density and specific heat capacity, respectively, and for $\lambda = \text{const.}$, one finally gets

$$\rho c_p \dot{T} = \lambda \frac{\partial^2 T}{\partial z^2} \quad \Rightarrow \quad \dot{T} = a \frac{\partial^2 T}{\partial z^2}. \tag{5}$$

Obviously, the second thermal transport property, TD, is related to TC by $a = \lambda/(\rho c_p)$, i.e., the TD can be considered as the ratio of energy conducted to the energy stored per unit volume.

The basic principles to measure TC and TD can now directly be derived from both Fourier laws: the first law only applies for TC from time-invariant, i.e., steady-state measurements. The second one enables the simultaneous determination of both transport properties from time dependent, i.e., transient runs.

To put Eq. 2 into practice, first, a heat source and a heat sink are required to generate a heat flow. In order to impose a flow of a known rate, Φ, through the specimen of cross-section area, A, generally, a resistance heater of constant electrical power is used. Here, it is assumed that the electrical power, P, is completely converted to the heat-flow rate, $P = RI^2 = \Phi$. Secondly, to determine the resulting (macroscopic) temperature gradient, $\partial T/\partial z = \Delta T/\Delta z$, across the specimen of thickness Δz, at least

two thermometers are needed, $\Delta T = T(z_2) - T(z_1)$. One or more guard heaters prevent the heat source and/or the lateral faces of the specimen from stray heat flows to ambient and, by this means, ensure a one-dimensional homogeneous heat flow Φ.

According to Eq. 2, the working equation for an ideal steady-state instrument during temperature equilibrium reads

$$\lambda = \frac{P}{A}\left(\frac{T(z_1) - T(z_2)}{z_2 - z_1}\right)^{-1}. \tag{6}$$

To put Eq. 5 into practice, generally, the heat source of known output power, $P = \Phi$, is embedded inside the specimen. Hereby, heat losses are minimized and the specimen itself can act as the sink. To bring the material under test to the correct working temperature, a thermostated bath or a furnace is used. Generally, the heat source additionally serves as a (resistance) thermometer to measure the temperature history. A run may be performed for as long as the imposed heat flow needs to penetrate the specimen.

Ideal transient instruments operate according to one of a few existing particular solutions to Eq. 5 (see Sect. 3.2). Any solution basically depends on the geometrical shape of the instrument's heat source (point, line, plane). Generally, transient instruments simultaneously measure TC and TD.

As mentioned above, usually, the basic material of a HT-TIM is of a porous type. Though the thermal conductivities of the matrix and the pore gas are as small as possible and the pores are not large enough for convection, radiative heat flow cannot be totally excluded. This is especially true at high temperatures because of Stefan–Boltzmann's T^4 law. Consequently, the 'as measured' transport property no longer is "thermal conductivity" but something like an "overall heat transmission coefficient" or an "effective thermal conductivity" [6]. Another major departure from the true quantity value, λ, easily occurs from the unavoidable thermal contact resistances on both sides of the specimen to the heat source and sink, respectively [7].

An ideal TC instrument for TIMs actually measures the thermal conductivity and not an "effective thermal conductivity" as defined in, e.g., [6]. It operates at temperatures up to at least 800 °C, and covers a measurement range from about 20 $\mathrm{mW{\cdot}m^{-1}{\cdot}K^{-1}}$ to about 1000 $\mathrm{mW{\cdot}m^{-1}{\cdot}K^{-1}}$. Its standard measurement uncertainty according to the GUM [8,9] should not exceed 5 %. Any other systematic errors vanish by complete correction or compensation.

From the viewpoint of customers who use TC data, it is important that an ideal instrument can measure with a minimum uncertainty. The indicated uncertainty should, in any case, be rigorously assessed according to the GUM [8,9], simply to make the result of a measurement traceable and comparable. This implies that, for customers, the major relevance of uncertainty is its role as a measure of the quality of a measurement.

Generally, customers prefer those results for their analyzed products that are obtained at such measuring conditions, e.g., with respect to the temperature gradient(s), that come close to the circumstances of the intended industrial application of a thermal insulation material.

From the viewpoint of measurement engineers, an ideal TC instrument should not only provide a minimum uncertainty but also be quick and easy to operate.

In practice, there are five different types of TC instruments to come into consideration for TIMs at higher temperatures; two of them operate in the steady-state mode and the other three in the transient mode. The steady-state instruments are of guarded hot-plate (GHP) and heat-flow meter (HFM) types. The transient instruments are of plane source, hot-wire methods or of non-contact laser/xenon flash techniques. A detailed survey on all these techniques and the related instruments is given in the literature [10–12]. Here, just a brief overview will be presented.

3 Measuring Instrument Types

3.1 Steady-State Instruments

Steady-state instruments measure the thermal conductivity in just one Cartesian direction. The general assumption behind these techniques is that a one-directional and uniform stationary heat flow is established between the hot and cold surfaces of a TIM specimen. By this means, anisotropically conducting materials, such as e.g., fibrous composites, can be analyzed by individual runs on three adequately cut specimens. Typically, the sample size of steady-state instruments is much larger when compared with transient instruments. Therefore, materials of poorer homogeneity can also be analyzed provided their sample thickness is at least about ten times the size of the largest inhomogeneity [13,14]. In contrast to contact transient methods, the overall TC of multi-layered specimens can be determined.

It is a great challenge in engineering steady-state instruments in order to measure the low TC of a TIM at high temperatures. However, evaluating the result of a steady-state measurement and the related uncertainty is less complicated than for transient methods.

For the most part, steady-state instruments suitable for HT-TIMs are the guarded hot-plate apparatus and the heat-flow meter.

3.1.1 Guarded Hot Plate (GHP)

For many years, the guarded hot-plate (GHP) apparatus is the "work horse" for measuring the thermal conductivity of low conducting materials. In its two-specimen type, it consists of a heat source and two sinks. Sandwiched between them are two specimens of almost identical properties. The heat source is surrounded by a guard heater that helps to prevent the source from lateral heat losses. The GHP-working equation can directly be derived from Eq. 2.

The major advantage of a GHP is its sophisticated technique and straight forward evaluation of results and the uncertainty analysis. The working equation is simple, and the measurement uncertainty is generally comparably small. Drawbacks are the very long run times and the relatively large temperature difference across the sample(s) of typically 30 K to 70 K that has to be established and maintained constant during hours.

A rough estimate of the number of existing HT-GHP instruments would be close to 20 devices. Tables 1 and 2 list HT-GHPs at national metrology institutes on one hand and at universities, institutes and companies on the other.

Table 1 Known National Metrology Institutes equipped with HT-GHP(s)

National Metrology Institutes	Number of HT-GHPs	Maker
Czech Metrology Institute, Czech Republic	1	Planned: in-house made
Laboratoire National de Métrologie et d'Essais, France	2	1 In-house made, 1 Netzsch-made
National Institute of Standards and Technology, USA	1	In-house made
National Physical Laboratory, United Kingdom	1	In-house made
Korea Research Institute of Standards and Science (*KRISS*), Republic of Korea	1	*Planned*: custom-made by a company
The Hungarian Trade Licensing Office (*MKEH*), Hungary	1	*Planned*: in-house made

Table 2 Known Universities, Institutes, and Companies equipped with HT-GHP(s)

University, Institute, Company	Number of HT-GHPs	Maker
TU Freiberg, Germany	1	In-house made
Univ. Stuttgart, IKE, Germany	1	In-house made
Danish Technological Institute, Denmark	2	Dynatech-made
European Fire and Conductivity Laboratory, (Rockwool Int.), Denmark	1	FIW-made
Forschungsinstitut für Wärmeschutz e.V. (FIW), Germany	4	In-house made
CRIR (Isover Saint Gobain), France	1	In-house made
Netzsch Gerätebau GmbH, Germany	1	Netzsch-made
Owens Corning	1	
Zentrum für Angewandte Energieforschung, Germany	1	In-house made

3.1.2 Heat-Flow Meter (HFM)

Heat-flow meters (HFMs) to be used for TIMs are of the axial (heat-flow) type [10,11]. That means the specimen is generally sandwiched between the heat source on top of the stack and the sink at the bottom. Between sample and sink, there is a heat-flow transducer (flux gage) to measure the density of the heat-flow rate. Generally, HFMs are guarded (GHFM), i.e., the stack is surrounded by a guard furnace. The flux gage can either be a calibrated sensor or a reference sample of known thermal conductivity. The working equation can also be derived directly from Eq. 2.

Advantages and disadvantages are similar to those of GHPs. In contrast to GHPs, HFMs are not absolute measuring instruments. They (directly) compare the measurand with a quantity of the same kind having a known value. Especially in this regard, it is disadvantageous of HFMs, e.g., for their use on TIMs, that there are no reliable heat transducers for high temperatures.

3.2 Transient Instruments

As has been mentioned above, to measure the TC at least requires four different functional units: heat source, heat sink, thermometer, and sample. Most transient instruments, instead, do need just two units, a heat source and the sample. The Joule heat source, embedded between two identical sample halves, simultaneously acts as a resistance thermometer to measure the temperature history of the sample. The sample concurrently behaves as a heat sink. These two bi-functional combinations not only allow very quick runs but they also effectively facilitate the design and construction of diverse types of transient instruments. Such an arrangement, however, limits the maximum duration of a run up to the time at which the sample starts to lose heat to the environment. Moreover, in their role as heat sinks, the samples have to be homogeneous.

The methods for transient TC instruments come in two different classes, contact and non-contact. While the latter class comprises the optical laser/xenon flash techniques, the electrical contact methods are almost as manifold as there are suitable solutions to the above-mentioned differential equation, Eq. 5. So far, nearly all of these solutions are based on one of four different heat source geometries, point, line, strip, or plane. For instance, the line heat source is normally realized by a very thin metal (platinum, molybdenum) wire that is stretched out between the solid sample halves. During a run, the wire is electrically heated. Its voltage drop versus time is the measure for the temperature rise required to determine the thermal transport properties, TC and TD. In most of the other cases, the heater/thermometer combination is realized by an as-thin-as-possible foil sensor. Here, a printed circuit made from nickel or platinum is glued in between two plastic foils.

The laser or xenon flash (LFA/XFA) technique uses one of the radiation sources mentioned in place of the Joule heater and an IR-detector or a thermocouple as the temperature sensor. LFA/XFA techniques are able to measure the TD only. The TC is calculated through an equation, $a = \lambda \rho cp$, with a further knowledge of the volumetric specific heat (ρc_p).

3.2.1 Transient Hot Wire (THW)

The THW technique is state of the art in measuring the thermal transport properties of fluids. For solids, the embedding of the wire(s) between the two sample halves can be problematical with respect to the emerging thermal contact resistance, especially in the case of rigid samples. A THW instrument generally comes in one of three experimental modifications, the resistance (or hot-wire probe) method using one wire, or the cross-wire and the parallel-wire techniques using both of two wires [15].

For solids, most often used are those instruments that consist of two wires. The wires are different in their functions, a heater wire and a temperature sensor wire. These come in two different arrangements [10,11,15]: in their cross-wire arrangement, the heater wire is centrally crossed by the sensor wire. They both work together as a thermocouple. In the parallel setup, the central heater wire and the temperature sensing wire are in parallel.

The working equation of the THW technique is given by

$$\Delta T = \frac{\Phi}{4\pi L\lambda} \ln\left(\frac{4at}{Cr^2}\right) \qquad C = \exp(\gamma) \tag{7}$$

where $\gamma = 0.5772\ldots$ is Euler's constant; L and r denote the length and radius of the wire, respectively. Available correction terms to Eq. 7 are presented and discussed in detail in, e.g., [16] and in relevant standards (Table 3).

The major advantage of the THW-technique is its simple setup, the high operating temperature, and the very small measuring time of just some minutes.

3.2.2 Transient Plane Source (TPS)

The key part of the transient plane-source technique is its Hotdisk® sensor [17]. The printed circuit that is sandwiched between two foils (Kapton®, Teflon®, Mica) is made as a nickel spiral. For working temperatures above 250 °C, the Mica-cladded sensor has to be applied.

In contrast to the THW method, the complex working equation of the TPS technique cannot be linearized. Therefore, the specimen's thermal transport properties, TC and TD, have to be evaluated from the monitored temperature history by an iteration process.

3.2.3 Laser/Xenon Flash (LFA/XFA)

The laser or xenon flash technique is by far the method most often used to determine the TD (directly) and TC (indirectly) at high temperatures.

A flash of a laser or a xenon lamp is irradiated on the front surface of a small disk-shaped sample where it is converted to heat by absorption. On the rear side of the sample, the resulting temperature rise in time is detected, by a thermocouple or a pyrometer. The monitored temperature history is evaluated for the characteristic time, $t_{1/2}$, of a 50 % rise. The working equation for TD is given by

$$a = \frac{-\ln(1/4)}{\pi^2} \frac{L^2}{t_{1/2}}, \tag{8}$$

where L denotes the specimen thickness. There are many evaluation software packages commercially available by manufacturers of laser-flash devices (Table 4). Correction terms to Eq. 8 are given in [18] as well as in the related standards listed in Table 3 and, e.g., [19].

The major advantage of the method is its very broad working temperature range and its ease of use. A disadvantage is the limitation to very small and homogeneous samples.

3.3 Overview

Summing up the information given in the above sections on individual TC measurement methods, it can be noted that there is no such transient or steady-state technique

that is of a universal character, i.e., that can be applied to any thermal transport property measurement problem. For low conducting materials such as TIMs, GHP and HFM seem to be the most advantageous techniques. Both these methods have proven themselves reliable over many years, especially, in the material development and quality control fields of activity.

In the past, GHPs and HFMs have seen a couple of successfully performed international round-robin tests and intercomparisons to verify their reliability and validate their individually assessed measurement uncertainties at near ambient temperatures. However, interlaboratory comparisons within Europe and North America during the past couple of decades have shown significant levels of scatter when used at high temperatures. These intercomparisons are critically reviewed in [20]. Although the high temperature GHPs used in these intercomparisons are of different sizes and configurations, they conformed to either their national standards or the international standard ISO 8302:1991 [21]. Unfortunately, this means that for the use of these two related techniques at high temperatures, e.g., for TIMs, a very large amount of effort and development costs are needed to construct the appropriate instrument. Obviously, producers shy away from the high expenditures and the technical efforts involved in introducing a HT-GHP or HT-HFM. This might be especially the fact, as long as there is no adequate international standard for these instrument types.

In Europe, there is an estimated number of about 18 GHP instruments for the use at high temperatures. Almost all these apparatuses are self-built, the vast majority by universities and material testing laboratories.

4 International Standards for TC and TD Measurement Techniques

Much of the scientific and engineering work on the thermal transport property measurement techniques, especially for TIMs for building products and components, is summarized in a collection of adequate individual standards (see Table 3). The inter-

Table 3 Relevant international standards for measurement techniques/instruments for thermal conductivity and thermal diffusivity [21–36]

GHP	HFM	THW	TPS	LFA/XFA
ISO 8302	ISO 8301	ISO 8894-1	ISO 22007-2	
		ISO 8894-2		
EN 1946-2	EN 1946-3	EN 993-15		EN 821-2
EN 12664	EN 12667			
EN 12667	EN 12939			
EN 12939				
ASTM C177	ASTM C518	ASTM C1113		ASTM E1461
JIS A 1412-1	JIS A 1412-2			

EN: European Standard (CEN: European Committee for Standardization)
ISO: International Organization for Standardization
ASTM: American Society for Testing and Materials
JIS: Japan Industrial Standard (JSA: Japanese Standards Association)

national activities in this field are effectively propelled and well organized by standards organizations like the International Organization for Standardization (ISO), the European Committee for Standardization (EN), the American Society for Testing and Materials (ASTM), and the Japanese Standards Association (JSA). There are standards for the relevant instrument types, e.g., ISO 8302, for the measurement procedures, e.g., EN 12664 as well as for the different classes of materials (products), e.g., ISO 22007-2. Unfortunately, not all of the related standards from different organizations are compatible to each other.

However, currently there is no adequate measurement standard for the thermal conductivity of TIMs at high temperatures.

5 Commercial Instruments

Most likely, the vast majority of thermal-conductivity measurements on solids worldwide are performed in a range from 0.01 $W \cdot m^{-1} \cdot K^{-1}$ to about 7 $W \cdot m^{-1} \cdot K^{-1}$ and at temperatures between 10 °C and 70 °C. This is particularly true for building products and components as the most frequent specimens and, less relevant, for plastics. It is therefore not surprising that instrument manufacturers build their devices predominantly for this segment of the market. An additional benefit for the related industry lies in the fact that in this field, the related international standards are high in number, detailed, and well-elaborated (see Table 3).

Table 4 provides an overview of TC and TD measuring instruments for service temperatures $\vartheta \geq 250$ °C along with their major features according to the offer lists of their producers. These lists are generally available through manufacturer's web sites and their publications.

With respect to TIMs and the need to analyze these devices especially at high working temperatures, it is immediately striking that there is no instrument that would completely fit the requirements by potential customers.

From the manufacturer's published information, the Hotdisk TPS series instrument is the one that comes closest to the above-mentioned technical demands. Regrettably, to the best of our knowledge, there is no evaluation of this instrument at HT conditions available, e.g., from an international round robin and/or an intercomparison including other instrument types.

Next in line is the Netzsch *TCT 426* that has an even higher maximum working temperature but is limited in its measurement range. According to the manufacturer, the lower limit is at 50 $mW \cdot m^{-1} \cdot K^{-1}$.

All listed types of LF and XF instruments 'easily' satisfy the temperature requirements. However, these apparatuses, apparently, do not measure sufficiently precise below 100 $mW \cdot m^{-1} \cdot K^{-1}$. Their major drawback concerning (poorly homogeneous) TIMs is the very small sample size. There is a very informative publication by Ebert and Hemberger [1] on the results of an intercomparison of TC measurements on a calcium silicate insulation material for temperatures up to 1100 K. These authors negatively comment on the results of the participating LFAs.

The GHP type offering the highest working temperature is the Taurus *TLP 500HT*. Its 500 °C maximum is at least twice the value of the other listed GHP instruments,

Table 4 Commercially available measurement instruments for thermal conductivity and thermal diffusivity at elevated temperatures ($\vartheta \geq 250$ °C)

Manufacturer type of instrument	Technique	Meas. range ($W \cdot m^{-1} \cdot K^{-1}$)	Max. temp. (°C)	Accuracy/ uncertainty	Sample dim. (number)
Hotdisk AB					
TPS series/Mica sensor	TPS	0.005–1800	1000	<5 %	Min. Ø13 × 3 mm^2
Laser Comp					
FOX 300 HT	HFM	0.1–10	250	>1 % (40 °C)	≤ Ø51 mm
GHP 600	GHP	0.1–10	250		
Linseis Messgeräte GmbH					Both instr.:
XFA 500	XFA	0.1–2000	500	nn	(1) ≤Ø25.4 × 6 mm^2
LFA 1000	LFA	0.1–2000	1600	nn	(2) 10 × 10 × 6 mm^3
Netzsch Gerätebau GmbH					
Titan 456	GHP	0.005–20	250	<2 %	300 × 300× ≤ 100 mm^3
LFA 427/457	LFA	0.1–2000	≤2800	nn	all LFA/XFAs
LFA 447/467	XFA	0.1–2000	≤500	nn	(1) ≤Ø12.7 × 6 mm^2
					(2) 10 × 10 × 6 mm^3
TCT 426	THW	<2	1250 (1500)	nn	250 × 125 × 75 mm^3
Taurus					250 × 250 mm^2
TLP 500 HT	GHP	0.01–0.5	400 (500)	nn	500 × 500 mm^2
TA Instruments					
DTC 300	HFM	0.1–40	300	3 % to 8 %	Ø50 × 25.4 mm^2
DXF and *DLF* series	LFA	0.1–2000	≤2800	5 %	≤Ø25.4 × 6 mm^2
Ulvac Riko					
GH series	HFM	0.1–15	280	nn	Ø50 × 20 mm^2
TC 9000	LFA	nn	1500	5 % (TD)	Ø10 × 3 mm^2

All features as specified by the instrument manufacturers' websites [37–43], "nn": no information available, "TD": thermal diffusivity

LaserComp *GHP 600* and Netzsch *Titan 456*. The published lower end of the measuring range of the *GHP 600*, already at 100 $mW \cdot m^{-1} \cdot K^{-1}$, is somewhat surprising.

All HFM instruments considered here do not start in a measurement range before 100 $mW \cdot m^{-1} \cdot K^{-1}$; the maximum temperature is at 300 °C (TA Instruments *DTC 300*).

A more general analysis of potential departures of measurement results from transient instruments compared to those of steady-state instruments is given by [18].

5.1 Special Selection Orders

According to the open information of the producers, it was attempted to sequence the commercial instrument types in the respective descending orders according to

(1) their standard retail price:
LFA–GHP–THW–XFA–TPS–HFM,
(2) their max. sample sizes:
GHP–THW–TPS–HFM–LFA/XFA,
(3) their max. working temperatures:
LFA/XFA–THW–TPS–GHP–HFM and,
(4) their respective minimum in measurement range:
GHP–TPS–(HFM, LFA/XFA)–THW.

Unfortunately, there are no solid data on the individual uncertainty budgets of the above-mentioned instruments. This is not particularly surprising because of the facts that (1) the assessment of uncertainty, though standardized by the GUM [8,9], does not always stand up to critical analysis and (2) it is an extremely good selling point. It is well known that any uncertainty assessment can only be as good as its experimental verification. International intercomparisons and/or round-robin tests would help a lot. Furthermore, certified reference materials would allow direct experimental validations of individual uncertainty budgets. For high service temperatures, these materials, however, still have to be qualified.

6 Conclusion

The key property for the above vital applications of TIMs is their 'thermal conductivity,' i.e., their *as-low-as-possible* thermal conductivity reliably experimentally verified at high temperatures. The better known is this transport property, the more effective are the development of new TIM types, the design of thermal insulations and fire protection structures, as well as the quality control in TIM production.

Unfortunately, there is no instrument commercially available that fully satisfies the demands of a TIM's HT thermal-conductivity measurement. So far, the vast majority of adequate instruments are self-made. In order to critically assess the abilities and disabilities of existing instruments, international round robins and intercomparisons as well as good practice guides and related standards are urgently needed.

Acknowledgments This work was funded through the European Metrology Research Programme (EMRP) Project SIB 52 Thermo. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

References

1. H.P. Ebert, F. Hemberger, Int. J. Thermophys. **50**, 1838 (2011)
2. Calcarb Rigid Carbon Thermal Insulation, p. 6, http://www.mersen.com/uploads/tx_mersen/3-CALCARB-rigid-carbon-thermal-insulation-mersen_03.pdf. Accessed Mar 2015
3. J. Fricke, U. Heinemann, H.P. Ebert, Vacuum **82**, 680 (2008)
4. R. Baetens, B.P. Jelle, J.V. Thue, M.J. Tenpierik, S. Grynning, S. Uvsløk, A. Gustavsen, Energy Build. **42**, 147 (2010)
5. K. Araki, D. Kamoto, S. Matsuoka, J. Mater. Process. Technol. **209**, 271 (2009)
6. ISO 7345, Thermal Insulation—Physical Quantities and Definitions (International Organization for Standardization-ISO, Geneva, 1996)
7. C.V. Madhusudana, *Thermal Contact Conductance* (Springer, New York, 1996)

8. ISO, Guide to the Expression of Uncertainty in Measurement (International Organization for Standardization-ISO, Geneva, 2008)
9. ISO/IEC Guide 98–3:2008, Uncertainty of Measurement—Part 3: Guide to the Expression of Uncertainty in Measurement (GUM:1995) (International Organization for Standardization-ISO, Geneva, 2008)
10. K.D. Maglic, A. Cezairliyan, V.E. Peletsky (eds.), *Compendium of Thermophysical-Property-Measurement Methods*, vol. 1 (Plenum Press, New York, 1984)
11. O. Madelung (ed.), *Landolt-Börnstein, Units and Fundamental Constants in Physics and Chemistry, Subvolume a: Units in Physics and Chemistry* (Springer, Berlin, 1991)
12. T. Ohmura, in Proceedings of ASME/JSME 2007 Thermal Engineering Heat Transfer Summer Conference, vol. 3 (Vancouver, British Columbia, 2007), pp. 455–463
13. EN 12664, Thermal Performance of Building Materials and Products—Determination of Thermal Resistance by Means of Guarded Hot Plate and Heat Flow Meter Methods—Dry and Moist Products of Medium and Low Resistance (Beuth Verlag GmbH, Berlin, 2001)
14. EN 12667, Thermal Performance of Building Materials and Products—Determination of Thermal Resistance by Means of Guarded Hot Plate and Heat Flow Meter Methods—Products of High and Medium Thermal Resistance (Beuth Verlag GmbH, Berlin, 2001)
15. "Hot Wire Methods for the Thermal Conductivity Measurement," http://www.tpl.fpv.ukf.sk/engl_vers/hot_wire.htm
16. J. Kestin, W.A. Wakeham, C.Y. Ho (eds.), *CINDAS Data Series on Material Properties*, vol. 1–1 (Hemisphere Publishing Corp, New York, 1990)
17. S.E. Gustafsson, Rev. Sci. Instrum. **6**, 797 (1991)
18. T. Baba, A. Ono, Meas. Sci. Technol. **12**, 2046 (2001)
19. ISO 18755:2005, "Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Determination of Thermal Diffusivity of Monolithic Ceramics by Laser Flash Method" (International Organization for Standardization-ISO, Geneva, 2005)
20. T.O.K. Martin, D.R. Salmon, A.C. Taylor, in *Thermal Conductivity 24, Proceedings of the 24*th *International Thermal Conductivity Conference, 1997*, ed. by P.S. Gaal, D.E. Apostolescu (Technomic Publishing Co., Lancaster, 1999), pp. 161–172
21. ISO 8302:1991, Thermal Insulation—Determination of Steady-State Thermal Resistance and Related Properties—Guarded Hot Plate Apparatus (International Organization for Standardization-ISO, Geneva, 1991)
22. ISO 8301:1991, Thermal Insulation—Determination of Steady-State Thermal Resistance and Related Properties—Heat Flow Meter Apparatus (International Organization for Standardization-ISO, Geneva, 1991)
23. ISO 8894–1:2010, Refractory Materials—Determination of Thermal Conductivity—Part 1: Hot-Wire Methods (Cross-Array and Resistance Thermometer) (International Organization for Standardization-ISO, Geneva, 2010)
24. ISO 8894–2:2007, Refractory Materials—Determination of Thermal Conductivity—Part 2: Hot-Wire Method (Parallel) (International Organization for Standardization-ISO, Geneva, 2007)
25. ISO 22007–2:2008, Plastics—Determination of Thermal Conductivity and Thermal Diffusivity—Part 2: Transient Plane Heat Source (Hot Disc) Method (International Organization for Standardization-ISO, Geneva, 2008)
26. EN 821–2:1997–11-15, Advanced Technical Ceramics—Monolithic Ceramics—Thermo-Physical Properties. Determination of Thermal Diffusivity by the Laser Flash (or Heat Pulse) Method (Beuth Verlag GmbH, Berlin, 1997)
27. EN 993–15:2005–07, Methods of Test for Dense Shaped Refractory Products—Determination of Thermal Conductivity by the Hot-Wire (Parallel) Method (Beuth Verlag GmbH, Berlin, 2005)
28. EN 1946–2:1999, Thermal Performance of Building Products and Components—Specific Criteria for the Assessment of Laboratories Measuring Heat Transfer Properties—Part 2: Measurements by the Guarded Hot Plate Method (Beuth Verlag GmbH, Berlin, 1999)
29. EN 1946–3:1999–04, Thermal Performance of Building Products and Components—Specific Criteria for the Assessment of Laboratories Measuring Heat Transfer Properties—Part 3: Measurements by the Guarded Heat Flow Meter Method (Beuth Verlag GmbH, Berlin, 1999)
30. EN 12939:2001–02, Thermal Performance of Building Materials and Products—Determination of Thermal Resistance by Means of Guarded Hot Plate and Heat Flow Meter Methods—Thick Products of High and Medium Thermal Resistance (Beuth Verlag GmbH, Berlin, 2001)

31. ASTM C177–13, Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded Hot Plate (American Society for Testing and Materials, West Conshohocken, PA, 2013)
32. ASTM C518–10, Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus (American Society for Testing and Materials, West Conshohocken, PA, 2010)
33. ASTM C1113/C1113M-09(2013), Standard Test Method for Thermal Conductivity of Refractories by Hot Wire (Platinum Resistance Thermometer Technique) (American Society for Testing and Materials, West Conshohocken, PA, 2013)
34. ASTM E1461–13, Standard Test Method for Thermal Diffusivity by the Flash Method (American Society for Testing and Materials, West Conshohocken, PA, 2013)
35. JIS A1412–1, Test Method for Thermal Resistance and Related Properties of Thermal Insulations—Guarded Hot Plate Apparatus (SAI Global, München, 1999)
36. JIS A1412–2, Test Method for Thermal Resistance and Related Properties of Thermal Insulations—Heat Flow Meter Apparatus (SAI Global, München, 1999)
37. A.B. Hotdisk (Gothenburg) (2015), http://www.hotdiskinstruments.com
38. LaserComp Inc. (Saugus, MA) (2015), http://www.lasercomp.com
39. Linseis Messgeräte GmbH (Selb) (2015), http://www.linseis.com
40. Netzsch-Gerätebau GmbH (Selb) (2015), http://www.netzsch-thermal-analysis.com
41. Taurus Instruments GmbH (Weimar) (2015), http://www.taurus-instruments.de
42. TA Instruments (New Castle, DE) (2015), http://www.tainstruments.com
43. ULVAC-RIKO (Yokohama) (2015), http://www.ulvav.co.jp