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Intercomparison of thermal conductivity measurements on an expanded glass granulate in a wide temperature range

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ABSTRACT

For laboratories within the scheme of the voluntary surveillance system of technical insulation material in Europe (VDI/Keymark) a proficiency test is the basis of obtaining a "registered laboratory". These laboratories are allowed to conduct measurements to verify the declared thermal conductivity curve of technical insulation materials under the umbrella of the quality mark VDI/Keymark. The initial characterization work on round robin materials for the determination of thermal conductivity at higher temperatures led to the selection of an expanded glass granulate. With all the results of the round robin tests produced by five European laboratories, the expanded glass granulate has become a very important material for establishing a reliable European level of thermal conductivity at higher temperatures with a moderate uncertainty.

The German expert group "AK-Thermophysik" of the Association of Thermal Analysis (GEFTA, Gesellschaft für Thermische Analyse e.V.) further initiated a round robin test for thermal conductivity in the year 2012. With this additional use of the expanded glass granulate material, more experience with transient methods and thermal conductivity values in the extended temperature range could be obtained. The thermal conductivity of expanded glass granulates was determined from −170 °C to 530 °C with different methods and apparatuses. Within this temperature range the thermal conductivity increases from about 0.030 W m^{-1} K^{-1} to 0.160 W m^{-1} K^{-1}. The comparison with the reference curve of the VDI/Keymark indicates very good compliance within 4% for most cases.

1. Introduction

In 1980 engineers and scientists established within the VDI – The Association of German Engineers – a voluntary surveillance system for technical thermal insulation materials. In 2012, the VDI and the Keymark Group of the CEN Certification Board collaborated and developed scheme rules for a new surveillance system, termed "VDI/Keymark". An important aspect is a proficiency test for laboratories to obtain a "registered laboratory" status. Registered laboratories are allowed to conduct measurements to verify the declared thermal conductivity curve of technical insulation materials under the umbrella of the quality mark VDI/Keymark [1,2].

Because there is no certified reference material for thermal insulation products at higher temperatures available, neither in Europe nor in USA, the search for an appropriate material began in 2004. The initial characterization work on round robin materials[1] for the determination of thermal conductivity at higher temperatures led to the selection of the expanded glass granulate Liaver® with a particle size of 1 mm–2 mm and of uniform density in the frame of a round robin test.[2] This granulate is temperature resistant up to 550 °C and incompressible. The uniformity of the material created homogeneous and isotropic test specimens. With its high availability and thermophysical characteristics, comparable to common thermal insulation materials, the expanded glass

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[1] A round robin material is a sample material which allows the preparation of test specimen with defined physical properties in order to determine these properties as exactly as possible.

[2] A round robin test is an interlaboratory comparison where the thermal conductivity of a well-defined sample material is determined by different laboratories under defined conditions.

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Nomenclature

e^*	total specific extinction coefficient, $m^2\ kg^{-1}$
d	specimen thickness, m
$\vec{q}$	density of heat flow rate, $W\ m^{-2}$
T	temperature, K

Greek symbols

τ	optical thickness, 1
ρ	specimen density, $kg\ m^{-3}$
λ	thermal conductivity, $W\ m^{-1}\ K^{-1}$
ϑ	temperature, °C

granulate was qualified as a round robin material for the determination of thermal conductivity.

Five European laboratories measured the thermal conductivity of the expanded glass granulate Liaver® in the framework of a round robin test and defined a VDI/Keymark reference curve. Therefore this material provides a very important standard to establish a reliable European level of thermal conductivity measurements with a moderate uncertainty for higher temperatures. In this context it is noteworthy to say that only steady state methods for the determination of the thermal conductivity were used.

In the year 2012 the German expert group "AK-Thermophysik" of the Association of Thermal Analysis (GEFTA, Gesellschaft für Thermische Analyse e.V.) initiated a round robin test for measuring the thermal conductivity of the expanded glass granulate at temperatures ranging from −170 °C to 530 °C, using different measuring methods. By participating in this comparative test, laboratories had the opportunity to improve quality assurance and reduce the uncertainty of thermal conductivity measurements. In this round robin test different steady state and transient measurement methods were applied. The derived thermal conductivity values will be discussed in detail and compared with the VDI/Keymark reference curve.

2. The round robin material

Early studies to identify an appropriate round robin material for the determination of thermal conductivity in a wide temperature range, especially at higher temperatures, led to an expanded glass granulate, i.e. Liaver® produced by the company Liaver GmbH & CoKG, Ilmenau, Germany. According to the manufacturer, the Liaver® expanded glass granulate is produced from recycled glass by applying a patented technique. Treated broken glass is fine-ground, mixed and formed. The granulate is sintered and expanded at a temperature of 750 °C–900 °C in a rotary kiln. By this method, a light and compression-resistant mineral product is produced with defined particle sizes and loose bulk densities (cf. Fig. 1).

Table 1
Compilation of physical properties of the investigated expanded glass granulate Liaver®.

Property	Value	Source
Particle size/mm	1–2	datasheet Liaver® [3]
Particle density/kg m^{-3}	350 ± 35	datasheet Liaver®
	396 ± 10	FIW, this work
Loose bulk density/kg m^{-3}	220 ± 22	datasheet Liaver®
	224 ± 5	FIW, this work
Thermal conductivity/W m^{-1} K^{-1}	0.070 (at 10 °C)	datasheet Liaver®
Average crushing resistance/MPa	2.2–3.0	datasheet Liaver®
Maximum operating temperature/°C	750	datasheet Liaver®
Porosity of the particle	0.81	FIW, this work
Bulk density of the glass/kg m^{-3}	2074	FIW, this work
Porosity of the granulate	0.38	FIW, this work

For the round robin test an expanded glass granulate with a particle size of 1 mm–2 mm and a loose bulk density of (220 ± 22) kg m^{-3} was used. This granulate is temperature resistant up to 550 °C and incompressible. The uniformity of the material allows creating homogeneous and isotropic test specimens with different shapes, if required. Therefore, test specimens with nearly identical properties for different test methods or setups can be used. The handling of the material is difficult and therefore detailed instructions for preparation of the test specimens have to be followed. The physical properties are compiled in Table 1. Additionally derived values of the batch used for this round robin tests are provided in this table. As shown the particle and the loose bulk densities stated by the manufacturer and determined by the authors are in close agreement.

Fig. 2 shows the distribution of the cumulative frequencies of the particle size of the investigated expanded glass granulates determined at FIW München.

It is important for the selection of a proper sample material for a round robin test dedicated to the thermal conductivity that the investigated specimen volume is optically thick, i.e. radiative heat transfer can be described by a diffusion process. This is the case for values of the optical thickness $\tau \gg 1$ and derived values of the thermal conductivity are independent of the infrared optical properties of the surroundings of the specimen, e.g. the emissivity of the measuring plates of a guarded hot plate, of sample thickness and measurement geometry with respect to thermal radiation. The optical thickness τ is defined by Ref. [4]:

$$\tau = e^* \cdot \varrho \cdot d, \tag{1}$$

e^* is the total specific extinction coefficient, ρ is the specimen bulk density and d is the thickness of the investigated specimen volume. In Fig. 3 the Rosseland averaged specific extinction of the investigated expanded glass granulate Liaver® is depicted as a function of temperature. The procedure for its determination is described in detail in literature [5–7]. For the given value of the

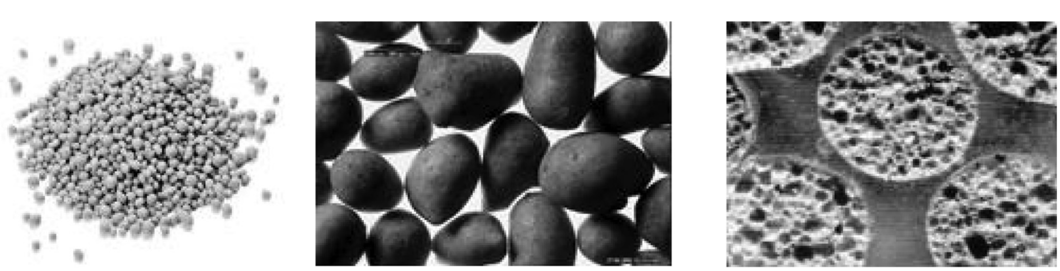

Fig. 1. Investigated expanded glass granulate Liaver®: loose bed (left), single granules with a granule size of approximately 1 mm–2 mm (middle) and cross sections through single granules (right).

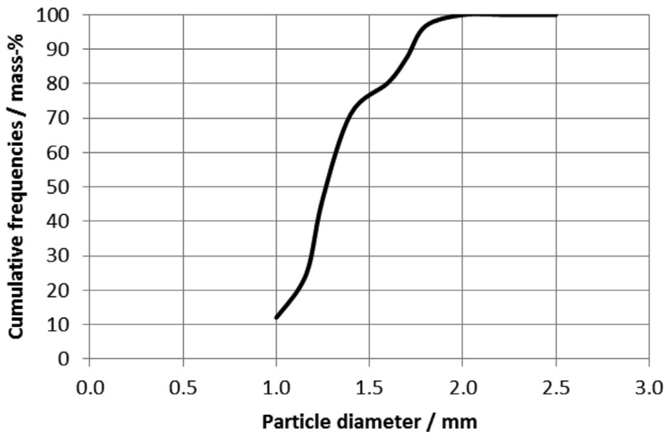

Fig. 2. Distribution of cumulative frequencies of particle diameter of the investigated expanded glass granulate Liaver®.

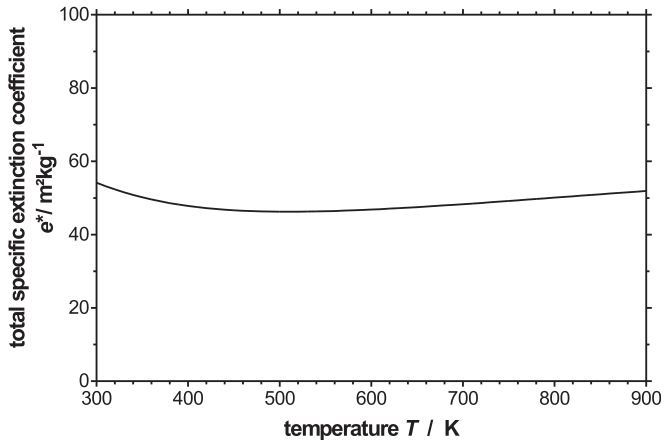

Fig. 3. Rosseland averaged total specific extinction coefficient of the investigated expanded glass granulate Liaver®.

loose bulk density of 220 kg m^{-3} of the investigated expanded glass granulate Liaver® and typical values of the specimen thickness in the range of 5 mm and above, the optical thickness of the investigated specimen is always higher than 55. Thus no significant radiation effects are expected regarding the determination of a reliable effective thermal conductivity in different measurement setups.

3. Applied methods for the determination of thermal conductivity

For the determination of thermal conductivity numerous methods exist which in general can be divided into two groups: steady state and transient methods [8,9]. For the steady state methods Fourier's law is the working equation for deriving thermal conductivity values. According to this law the density of heat flow rate under stationary conditions within a homogeneous and isotropic material is given by:

$$q = -\lambda \frac{dT}{dx}, \quad (2)$$

q: density of the heat flow rate, λ: the thermal conductivity and dT/dx: local temperature gradient. The experimental design of the measurement equipment has to fulfill the conditions of this law as accurately as possible. Thus, in principle, a stationary temperature gradient within a specimen has to be established and the density of heat flow rate and the temperature gradient has to be determined to calculate the thermal conductivity. The most widely used steady state methods are the guarded hot plate and heat flow meter methods [8,10–12]. The geometry of the specimen used for these methods are rectangular or disc-shaped flat specimen volumes with defined plan parallel specimen surfaces perpendicular to the heat flow. In the case of the steady state pipe tester method the specimen has the shape of a hollow cylinder where the temperature gradient is perpendicular to the axis of the cylinder [13]. Regarding the sphere method a spherical specimen is investigated [14]. One advantage of the mentioned steady state methods is that larger specimen volumes can be investigated. This reduces the influence of small inhomogeneities within the investigated specimens on the determined thermal conductivity values. For the determination of the VDI/Keymark reference curve only these mentioned steady state methods were applied.

Within the round robin test initiated by the German Thermophysics Working Group also transient methods for the determination of the thermal conductivity, i.e. transient hot wire, hot disk and transient hot bridge (hot strip), were used. In the case of transient methods a thermal disturbance, i.e. a temperature change, affects the temperature distribution within the investigated specimen and this thermal response is evaluated to derive a thermal conductivity value. Therefore the experimental setup is designed so that it corresponds closely to an available solution of the transient heat transfer equation. The transient hot wire method is based on the ideal line heat source model. An electrically conducting wire which is embedded within the specimen is heated up by a constant power. The resulting time dependent temperature increase of the wire is a function of the thermal conductivity of specimen material [15–18]. In the hot disk method a disk shaped thin heating element is in close thermal contact to the specimen surface. At the same time the heating element acts as temperature sensor [19,20]. In the hot strip method a thin electrically conducting strip is used as heating and sensor element [21–23]. Regarding the transient methods it should be mentioned that the thermal disturbance, e.g. as a induced temperature increase by heating a wire, disk or strip with constant power, is propagating from the heating source into the specimen material and only the thermal conductivity of this specimen volume can be determined.

4. The European level of thermal conductivity —VDI/Keymark reference curve

Voluntary quality surveillance is applicable to thermal insulation products for building equipment and industrial installations. The Quality Assurance Committee (QAC) which was set up by CEN and VDI is responsible for the development and maintenance of the scheme rules. The registered testing laboratories related to the determination of thermal conductivity are those that have demonstrated that they are sufficiently competent to examine a temperature-dependent thermal conductivity curve. The group of registered testing laboratories forms the European group of reference laboratories active in quality surveillance, called the QAC/Lab group [1].

The role of the registered laboratories is to conduct product type testing and audit testing according to European standards and the specific scheme rules for thermal insulation products for building equipment and industrial installations. To ensure transparency and fair competition on the open European market, there is a need to obtain European conformity requirements for the thermal conductivity curve of insulation products. The term 'European level of conformity requirement' is used for the evaluation of comparative testing in which the same test specimens/samples are used by both experts and registered laboratories. It is defined by showing compliance with the relevant European standards and a reference material. The results should be in agreement with the European levels of conformity requirements for the thermal conductivity curve as follows:

- ≤ 3% for a temperature range from −180 °C and 500 °C
- ≤ 5% for a temperature above 500 °C.
- To ensure consistency with the thermal conductivity at lower temperatures, the apparatus shall show compliance with the European-certified reference material IRMM 440 at a temperature interval from approx. 40 °C to 70 °C [24].

The experts of the QAC/Lab group decided to use the expanded glass granulate Liaver® to establish the VDI/Keymark reference curve of thermal conductivity related to temperature. Furthermore, this granulate became the test material for proficiency tests used as the basis for becoming a “registered laboratory”. This thermal conductivity is interpreted as the thermal transmissivity or effective thermal conductivity of the insulation material and is independent of the experimental conditions. Every measured value under different conditions, for example, a high temperature difference, had to be recalculated accordingly.

The active registered laboratories in the QAC/Lab group are LNE (France), MPA NRW (Germany), DTI (Denmark), EFiC (Denmark) and FIW München (Germany). The used apparatuses for the determination of the temperature dependent thermal conductivity, the VDI/Keymark reference curve, are shown in Table 2. The different constructed Guarded Hot Plates (A-E) work all according to the European test standard EN 12667. The test method for the Pipe Testers is the EN ISO 8497.

For the determination of the thermal conductivity values for the reference curve only steady state methods were used, i.e. guarded hot plate and heat flow meter methods as well as the pipe tester and sphere method. The steady state methods for flat specimens, i.e. guarded hot plate and heat flow method, uses small temperature differences below 40 K for the determination of the thermal conductivity. Due to the fact that for the pipe tester and sphere methods the temperature difference within the specimen is very high, the derived values for these methods have to be recalculated to meet the boundary conditions of the VDI/Keymark reference curve and a so called VDI/Keymark effective reference curve has to be calculated.

A pool of approximately 200 measured results was the basis for determining the reference curve. The measured values were improved using the Gaussian correction method to evaluate the ±3% uncertainty (criteria) of the reference curve. A graphical presentation of the temperature-dependent thermal conductivity of the VDI/Keymark reference curve as a third degree polynomial is shown in Fig. 4.

All thermal conductivity values are related to small temperature differences of the specimens (<40 K). Other conditions have been considered with appropriate recalculations. The valid temperature range of the VDI/Keymark reference curve is 0 °C to 550 °C. An assessment of measured values beyond this valid range is not possible.

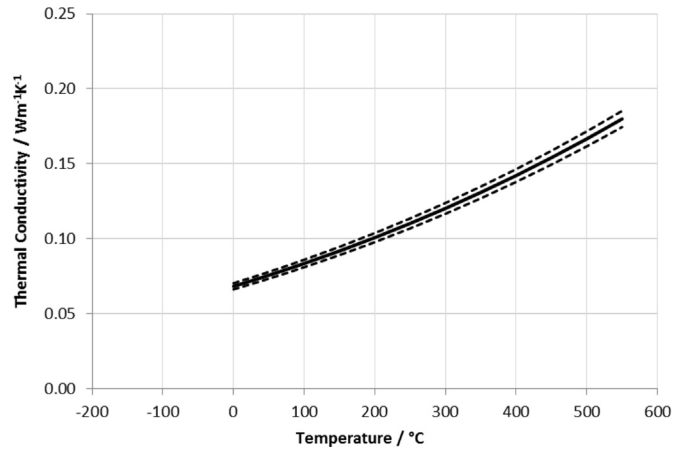

Fig. 4. Thermal conductivity VDI/Keymark reference curve of the investigated expanded glass granulate Liaver® as a function of temperature (solid line) with the uncertainty criteria of ±3% (dashed lines).

In most cases the third degree polynomial is a good enough curve fit function. However, this situation can be improved with a known heat transfer model for the expanded glass granulate. If the thermal conductivity of the glass itself and the loose fill is known, it is possible to calculate the heat transfer using a physical model with parameters corresponding to spectral characteristics with respect to the radiation transfer [25].

5. Round robin test of the German Thermophysics Working Group – participants and applied methods

In 2012 the German expert group “AK-Thermophysik” launched a round robin test for the determination of the thermal conductivity of expanded glass granulate Liaver® in a temperature range from −160 °C up to 600 °C. Following laboratories were involved:

- NETZSCH Gerätebau GmbH, Selb,
- Austrian Institute of Technology GmbH (AIT), Vienna, Austria,
- Deutsches Zentrum für Luft-und Raumfahrt e.V. (DLR), Stuttgart,
- Bavarian Center for Applied Energy Research (ZAE Bayern), Würzburg,
- Evonik Industries AG, Hanau-Wolfgang,
- Linseis Messgeräte GmbH, Selb,
- Institute of Heat Technology and Thermodynamics (IWTT), Freiberg.

Table 2
Used apparatuses of the registered laboratories of the QAC/Lab group for the determination of the VDI/Keymark reference curve.

Apparatus	Measurement principle, specimen geometry	Temperature range of the apparatus
Guarded Hot Plate A	steady state, flat	−160 °C to 700 °C
Guarded Hot Plate B	steady state, flat	50 °C to 800 °C
Guarded Hot Plate C	steady state, flat	80 °C to 550 °C
Guarded Hot Plate D	steady state, flat	50 °C to 700 °C
Guarded Hot Plate E	steady state, flat	30 °C to 800 °C
Several Pipe Testers	steady state, cylindrical	−70 °C to 350 °C[a]
Two Spheres	steady state, spherical	30 °C to 150 °C[a]

[a] Mean temperature.

The used apparatuses by the participants are compiled in Table 3.

6. Results

For the evaluation of the measured thermal conductivity values in the frame of the round robin test the VDI/Keymark curve is used as reference within the validity range between 0 °C and 550 °C. Measured values beyond this temperature range are only informative. The difference between the measured thermal conductivity values and the VDI/Keymark reference curve is expressed as a relative mean deviation.

Fig. 5 presents all measured results of the thermal conductivity of the expanded glass granulate Liaver® as well as the VDI/Keymark reference curve in relation to the temperature.

Considering only apparatus pursuant to steady state measuring methods for flat specimens like the guarded hot plate and the heat flow meter the presentation of the results are shown in Fig. 6.

The measured results of the transient methods for the determination of the thermal conductivity are shown in Fig. 7.

In Fig. 8 the derived measuring results of the „steady state, other" apparatuses (Pipe tester and sphere) are available. These steady state methods used specimen temperature differences greater than 40 K during the test. The VDI/Keymark reference curve had to be recalculated for these boundary conditions (VDI/Keymark effective reference curve). All results refer to the mean temperature.

7. Discussion

A summary of all differences between the measured values and the VDI/Keymark reference curve expressed as the relative mean deviation is summarized in Table 4. The different apparatuses are grouped accordingly. The influence of the loose bulk density of the expanded glass granulate on the thermal conductivity is not taken into account because of installing all granulate specimens in a

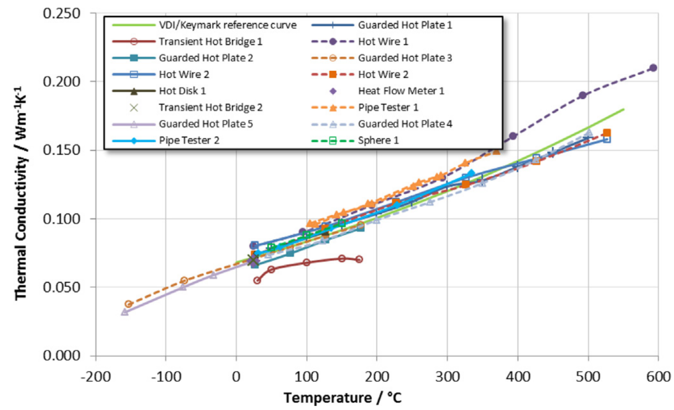

Fig. 5. Experimentally determined thermal conductivity values of all participants and the VDI/Keymark reference curve as a function of temperature.

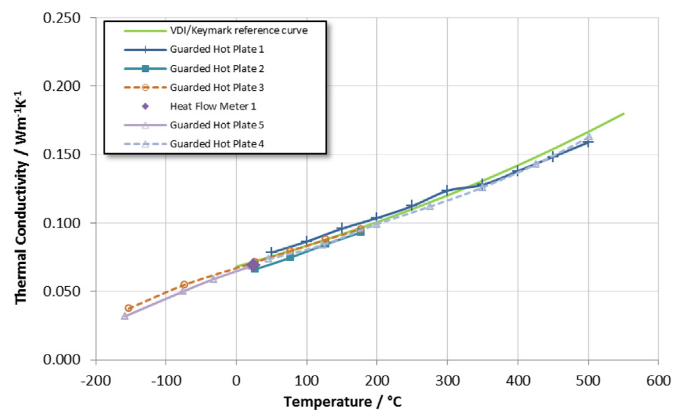

Fig. 6. All measured values of the steady state method for flat specimens, and the VDI/Keymark reference curve as a function of temperature.

Table 3
Applied apparatuses of the participants of the round robin test of the German expert group "AK-Thermophysik".

Apparatus	Measurement principle, specimen geometry	Temperature range of the apparatus	Relative uncertainty of thermal conductivity stated by the participant
guarded hot plate 1 (commercial)	steady state, flat	−160 °C to 600 °C	2 % − 4 %
guarded hot plate 2 (own-build)	steady state, flat	30 °C to 230 °C	4%
guarded hot plate 3 (own-build)	steady state, flat	−170 °C to 230 °C	4%
guarded hot plate 4 (own-build)	steady state, flat	−180 °C to 30 °C	5%
guarded hot plate 5 (own-build)	steady state, flat	30 °C to 800 °C	5%
heat flow meter 1 (commercial)	steady state, flat	approx. 30 °C	not specified
pipe tester 1(own-build)	steady state, others	80 °C to 380 °C[a]	4 % − 5 %
pipe tester 2(own-build)	steady state, others	30 °C to 350 °C[a]	3%
sphere 1(own-build)	steady state, others	30 °C to 150 °C[a]	3%
hot wire 1(commercial)	transient	130 °C to 530 °C	5%
hot wire 2(own-build)	transient	30 °C to 530 °C	5 %–10 %
hot disk 1(commercial)	transient	30 °C to 130 °C	2 %–4 %
transient hot bridge 1 (commercial)	transient	approx. 25 °C	4 %–6 %
transient hot bridge 2 (commercial)	transient	approx. 30 °C	not specified

[a] Mean temperature.

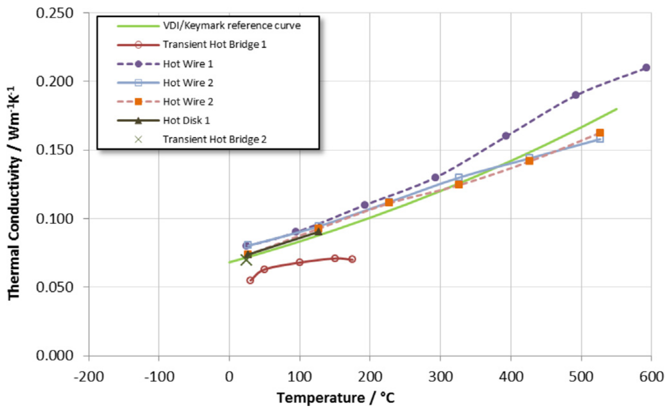

Fig. 7. All measured thermal conductivity values of the transient method and the VDI/Keymark reference curve as a function of temperature.

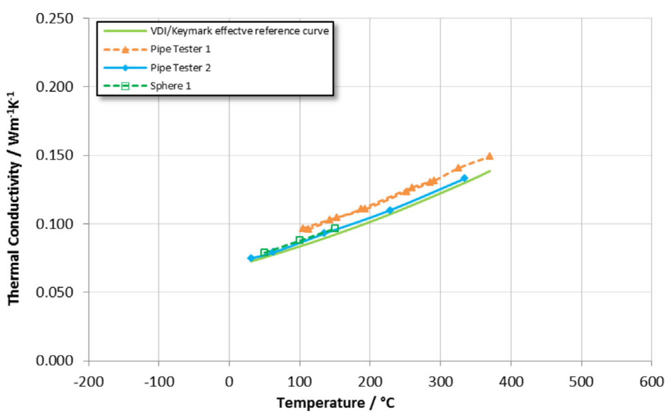

Fig. 8. Measured values of the steady state method for “other” apparatus and the VDI/Keymark effective reference curve as a function of temperature at higher temperature differences.

density within the tolerances of the manufacturer (220 ± 22) kg m^{-3}.

The steady state method for flat specimens tends to have the smallest relative mean deviations in relation to the VDI/Keymark reference curve. This is based on the minimized contact resistance

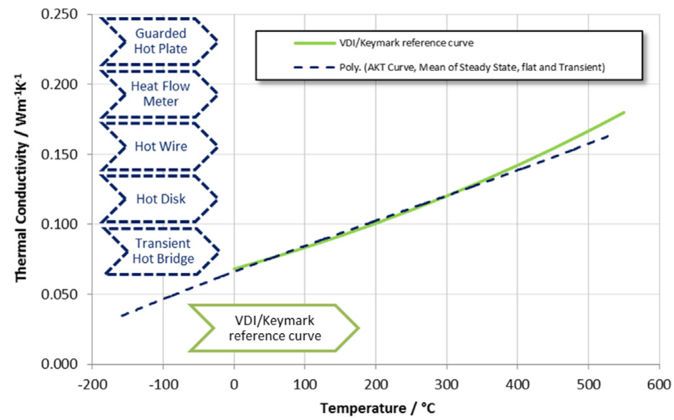

Fig. 9. Comparison of VDI/Keymark reference curve (dashed curve) with the AKT Curve, mean polynomial of the measured values of the steady state, flat, and transient methods (solid line) as a function of temperature.

between the plates and the granulate as well as the large metering area compared to the transient methods. The measurement values received by the Pipe Tester 1, Hot Wire 1 and Transient Hot Bridge 1 apparatus show significant deviations from the VDI/Keymark reference curve, which do not correspond to the given uncertainty for the reference curve and for the specific test method (cf. Table 3). Therefore the calculated relative mean deviation was also calculated without the obtained values by these apparatuses (cf. Table 4). In this case an average relative mean deviation of 4% could be stated. It is also the tendency that for the transient methods higher variations of the determined thermal conductivity values can be observed due to the fact that a smaller sensor area and therefore a smaller specimen volume is scanned during a measurement and preparation effects and a careful design of the experiment becomes more important.

In Fig. 9 the derived thermal conductivity curves representing the VDI/Keymark reference curve and the averaged result of the round robin test (AKT curve) are depicted as function of the temperature.

The average mean values of the determined thermal conductivity values of the round robin test (AKT curve) can be expressed as a third degree polynomial:

Table 4
Relative mean deviation to the VDI/Keymark reference curve for all measurements during the round robin tests.

Apparatus/Group	Steady state, flat	Transient	Steady state, others	Total
Guarded Hot Plate 1	3%			3%
Guarded Hot Plate 2	5%			5%
Guarded Hot Plate 3	1%			1%
Guarded Hot Plate 4	4%			4%
Guarded Hot Plate 5	3%			3%
Heat Flow Meter 1	4%			4%
Pipe Tester 1			(12%)	(12%)
Pipe Tester 2			3%	3%
Sphere 1			5%	5%
Hot Wire 1		(11%)		(11%)
Hot Wire 2		7%		7%
Hot Wire 2		4%		4%
Hot Disk 1		3%		3%
Transient Hot Bridge 1		(22%)		(22%)
Transient Hot Bridge 2		3%		3%
Total amount of measurements	Σ 6	Σ 6/Σ 4[a]	Σ 3/Σ 2[a]	Σ 15/Σ 12[a]
Relative mean deviation	4%	8%/4 %[a]	7%/4 %[a]	6%/4 %[a]

[a] Results without values in brackets.

$$\lambda(\vartheta) = \left(6.6088\cdot10^{-2} + 1.8917\cdot10^{-4}\frac{\vartheta}{^{\circ}C} - 4.9413\cdot10^{-8}\frac{\vartheta^2}{^{\circ}C^2} + 7.3283\cdot10^{-11}\frac{\vartheta^3}{^{\circ}C^3}\right)Wm^{-1}K^{-1}. \quad (3)$$

For this calculation the values from the measurement setups 'Pipe Tester 1', 'Hot Wire 1' and 'Transient Hot Bridge 1' were omitted. The two totally different approaches of the determination of the mean polynomial function of the temperature-dependent thermal conductivity of the expanded glass granulate lead to nearly the same curve below 350 °C. For temperatures up to 550 °C both curves are consistent within the stated uncertainties. Thus it can be concluded that an extension of the VDI/Keymark reference curve to the low temperatures is possible and that we have reached the level of the "true thermal conductivity" of the expanded glass granulate for temperatures below 550 °C.

A small systematic deviation of both curves at temperatures above 350 °C can be observed. This deviation is within the given uncertainties of the measurements. With the exception of the values derived by the pipe tester and sphere methods all considered values of the AKT curve are smaller in comparison to the values of the VDI/Keymark reference curve, whether steady state or transient methods are considered. Thus no significant influence of thermal radiation depending on the applied measuring method can be observed.

8. Conclusion

Because there is no European- or USA-certified reference material for thermal insulation products at higher temperatures, the search for an appropriate material started in 2004. The extensive work with the expanded glass granulate by the QAC/Lab-group, with the help of registered laboratories, has led to the determination of a reliable European level of thermal conductivity in the temperature range of 0 °C–550 °C with a moderate uncertainty. Using expanded glass granulate for this project, additional experience with transient methods and thermal conductivity values in an extended temperature range was gained. The comparison with the reference curve of the VDI/Keymark shows very good compliance for most of the cases, i.e. a relative mean deviation of 4%. All these comparative tests with the expanded glass granulate as a round robin material became even more important since August 2012, where the European CE-marking became mandatory for industrial insulation products and the temperature dependent thermal conductivity, declared by the manufacturer, was introduced as a limit value. This challenge to determine the thermal conductivity within a temperature range from −160 °C up to 600 °C with different test methods and apparatuses is also an important step to evaluate the level of the "true thermal conductivity" of the expanded glass granulate.

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