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A New Panel Test Facility for Effective Thermal Conductivity Measurements up to 1,650°C

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Abstract A new steady-state panel test facility is presented which has been designed and constructed for effective thermal-conductivity measurements of insulations in the temperature range between 300 and 1,650°C following ASTM C201-93 and DIN V ENV-1094. Square-shaped samples (length of 400 mm) are used, heated from above and settled on a water-cooled calorimeter system to obtain a one-dimensional steady-state temperature field. The heat is supplied by electrical heating elements freely hanging inside a furnace which is completely constructed from ceramic components to withstand temperatures up to about 1,800°C. The calorimeter system consists of a square central measuring zone (length of 100 mm) surrounded by guard loops to avoid heat losses in all directions. The samples, e.g., a number of fiber mats, one on top of the other up to a maximum height of 110 mm, are open to ambient pressure and atmosphere (air). Measurements include the heat flow rate (taken in the central calorimeter), temperature differences across individual layers of the sample (measured by a series of thermocouples which regularly have to be calibrated), and the thickness of the respective layers (before and after the experiment). The thermal conductivities range from 0.025 to $2\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$, and both isotropic and non-isotropic materials can be investigated due to the one-dimensional characteristic of the temperature field. Measurements for alumina fiber mats are presented, and good agreement is found with respective results from other methods and test facilities.

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1 Introduction

The development and application of extremely high-temperature insulations is a big challenge for thermophysical properties measurements. Related investigations have been carried out for more than 25 years by a research group at the Institut für Wärmetechnik und Thermodynamik (IWTT) of TU Bergakademie Freiberg. Measurement facilities have been designed, constructed, and operated that represent various measuring principles. For more details regarding temperature and thermal conductivity ranges, the geometry of the samples etc. [1,2].

Extended experiments showed that non-isotropic materials such as, e.g., fiber mats are best investigated with panel test procedures following the DIN V ENV-1094 and ASTM C201/C202/C182 standards (dating from 2004) despite the high efforts of the test procedures [1,2]. Up to now three generations of panel test facilities have been developed by the IWTT group following this principle with step-by-step improved construction details and a maximum application temperature which increased from 1,150°C in the first setup to 1,450°C in the next facility, PMA2, which is still in operation (for more details, see [3,4]). In this contribution a new facility, PMA4, will be presented which has been designed and constructed for effective thermal conductivity measurements of insulations in the range from 0.025 to $2\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$ and at temperatures between 300°C and 1,650°C. Due to the chosen measuring principle and arrangement, PMA4 measurements are restricted to porous media containing ambient atmosphere (air) at ambient pressure.

2 Design and Operation of PMA4

2.1 Measuring Principle

The panel test facility PMA4 is designed for steady-state measurements closely following the DIN V ENV-1094 standard. A square-shaped plane sample is used (for details see Table 1) uniformly heated and cooled at its upper and lower front surfaces, respectively, by using electrical heating elements and cooling water passing through a calorimeter system for the measurement of the heat flow rate $\dot{Q}$. After establishment of a steady state, the effective thermal conductivity can easily be evaluated from the measured temperature difference ΔT across the sample following

$$\lambda_{\mathrm{eff}} = \frac{\dot{Q}}{A}\frac{d}{\Delta T} \tag{1}$$

where A and d are the cross-sectional area and thickness of the sample, respectively.

This sounds straightforward; however, numerous problems have to be overcome with respect to the establishment of one-dimensional heat flow and to minimization of other measuring errors.

Table 1 Some details of the panel test facilities PMA2 and PMA4

	PMA2	PMA4
Temperature range (°C)	300–1,450	300–1,650
Sample dimensions (mm)	300 × 30 × 120 (max)	400 × 400 × 110 (max)
Active cross section (mm)	100 × 100	100 × 100
Heating elements		
Number	18	15
Material	SiC	Moly-D (U shaped)
Arrangement	Horizontal	Hanging vertically
Construction materials		
Heating section	Lightweight firebricks	Ceramic fiberboards
Measuring section	Lightweight firebricks	Lightweight firebricks

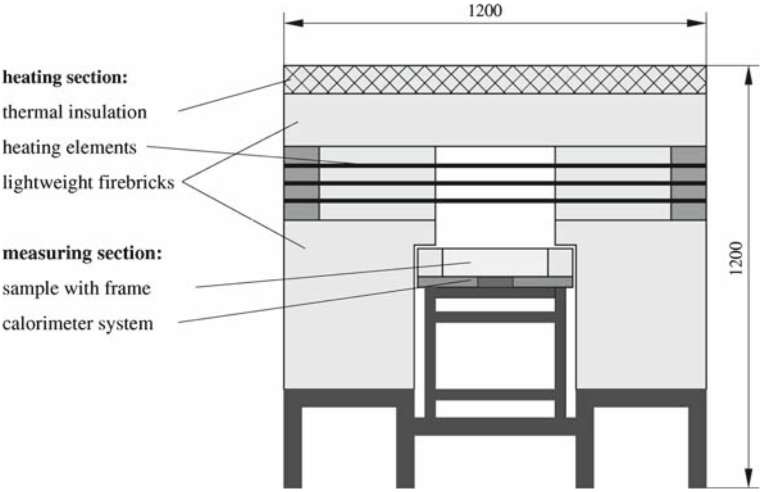

Fig. 1 Schematic drawing of panel test facility PMA2 (dimensions in mm)

2.2 Design

Both facilities, PMA2 and PMA4, are composed of two sections, namely, the *heating section* as the fixed upper part and the removable *measuring section* as the lower part. Both of the facilities differ from each other with respect to the construction principle of the heating section, applied materials, and heating elements; see Figs. 1 and 2 and also Table 1 for some details of the design.

2.2.1 Heating Section

The PMA4 heating section is composed of ceramic fiberboards with four different specifications depending on the temperature requirements. The lower part of the heating

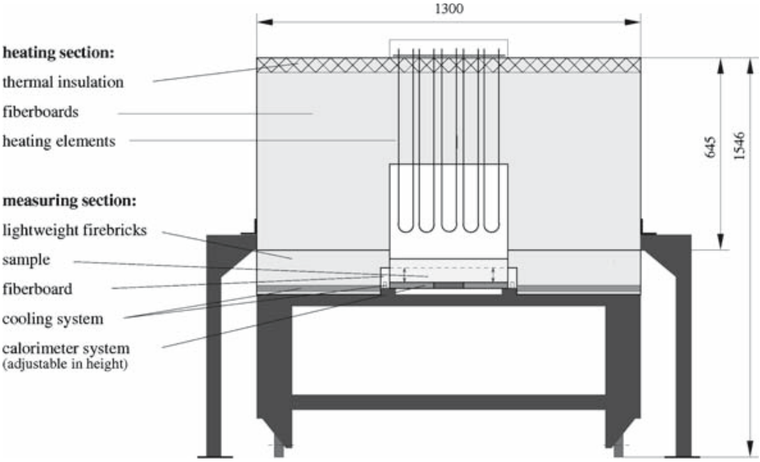

Fig. 2 Schematic drawing of panel test facility PMA4 (dimensions in mm)

section is formed by vertical plates that are cut off in the central part creating an enclosure with a square cross section (length 400 mm, height 280 mm). About 15 U-shaped Moly-D heating elements freely hanging inside this furnace are arranged for realizing a uniform temperature distribution at the upper sample's front surface. With respect to the designed maximum temperature of 1,650°C, heavy duty fiberboards have been chosen (maximum service temperature of 1,800°C). The furnace temperature is measured and controlled by means of a type B thermocouple positioned in the center of the enclosure. The heating section is completely surrounded by further insulation materials and finally covered by an outer skin of aluminum as a mechanical protection sheet. The ceramic fiberboards forming the heart of the heating section are fixed in their position by a complex frame system composed of ceramic and metallic materials which have to withstand the weight in an extreme thermal environment.

2.2.2 Measuring Section

The measuring section is designed for convenient handling of the samples with the embedded thermocouples. This is done by a carriage including the sample which is pressed upwards to the heating section during operation. When removed, the sample is lowered in a first step, and after that, it is moved by the carriage outside the heating section. The heart of the measuring section is a system of calorimeters directly positioned below the sample and embedded in and surrounded by lightweight firebricks. The calorimeter system can be moved vertically allowing for a maximum height of the samples up to 110 mm. The calorimeter system covering a cross section of $400 \times 400\,\mathrm{mm}^2$ consists of one central calorimeter for the heat flow measurements ($100 \times 100\,\mathrm{mm}^2$) surrounded by three additional calorimeters for thermal protection. Water is supplied to this system in a closed cycle with a controlled flow rate and temperature by respective cooling systems.

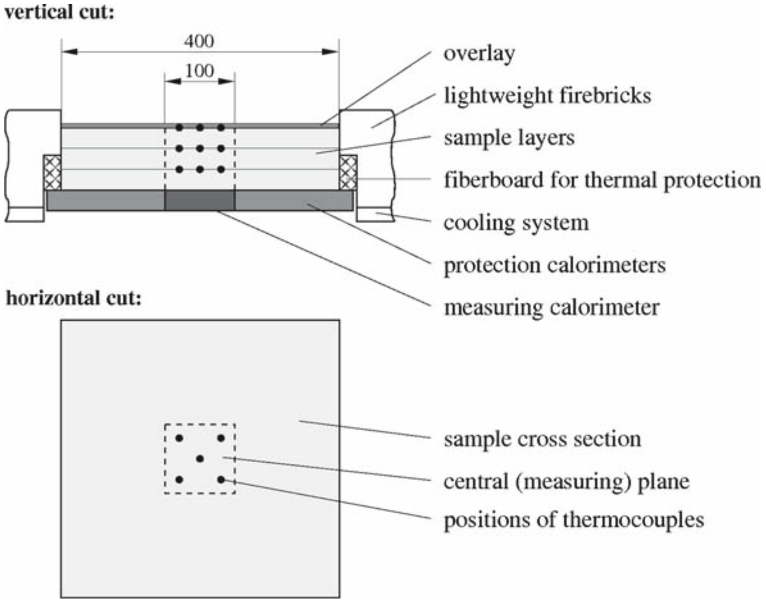

Fig. 3 Arrangement of thermocouples for a sample consisting of three layers (dimensions in mm)

Much is done to avoid any lateral, i.e., horizontal, heat flow from/to the central (measuring) calorimeter: the introduction of a 0.5-mm slit between the measuring and the first protection calorimeter, exact control of the water temperature increase in the various parts of the calorimeter system (see Sect. 2.4), application of additional cooling coils in the surrounding refractories, etc.

2.3 Sample Temperature Measurements

The PMA4 measuring device is designed for plate- and mat-shaped insulations with a cross section of $400 \times 400\,\mathrm{mm}^2$. Within the total height of 110 mm, several samples can be used, one on top of the other with temperature measurements above, below, and in between. Using this arrangement, two or more measuring points can be obtained from one steady-state experiment by evaluation of the readings for the various layers at different mean temperatures. For these measurements five thermocouples are located in each of the cross sections where type K and B elements are used below and above 1,000°C, respectively, being regularly calibrated before and after application. The uppermost thermocouples are secured to the surface of the samples. In the lower layers they are fixed in their position by the surrounding frame. As an example, a three-sample arrangement is shown in Fig. 3 enabling data logging and evaluation for the upper two ones.

2.4 Heat Flow Measurements

The heat flow rate $\dot{Q}$ vertically passing through the sample is measured by means of the calorimeter system as expressed by

$$\dot{Q} = \frac{V_{\text{water}}}{\Delta\tau} \left(\rho c_p\right)_{\text{water}} (\Delta T)_{\text{water}} \qquad (2)$$

where V_{water} is the volume of a measuring cylinder and $\Delta\tau$ is the time to fill the cylinder between two levels determined by two electric resistance sensors. Before starting a water flow-rate measurement, the difference of the outlet temperatures of the measuring section and the first protection calorimeter is controlled to be zero by respective variations of the flow rate. By doing this, optimum adiabatic conditions are realized for the central calorimeter as both of the calorimeters contain meandering channels in parallel, with the inlet positions and also the outlet positions close to each other. This important temperature difference is measured by a series of three type J thermocouples, and the temperature increase $(\Delta T)_{\text{water}}$ in the central part by a series of five type J elements as well.

2.5 Control and Data Aquisition

The various systems for control and organization of the measuring procedure and for the measurements are operated in a completely automated way. Two pcs, one for control and operation, the other for measurements, communicate with each other and additionally with the temperature control unit of the heating device. By this, not only the sample temperature measurements and the step-by-step temperature increase and decrease but also the measuring and adjusting processes for the calorimeter system are organized. All results are available online enabling the plot of spatial and temporal temperature profiles and also complete data evaluation and documentation.

One typical measuring series consists of a stepwise increase of the furnace temperature up to a given maximum value including intermediate steady states for the measurements. The duration of such a series typically amounts to more than 7 days in the automated version. Respective protection systems for, e.g., emergency cooling allow the facility to be run 24 h and 7 days a week with online information by the internet about status and measured results.

3 First Experiments and Numerical Simulation

3.1 Experiments

For the first tests a ceramic fiberboard has been used as a three-layer sample with a total height of 92 mm. The regular set of thermocouples (see Sect. 2.3) has been supplemented by additional temperature sensors outside the central (measuring) cross section to obtain more information about the temperature profile. In these experiments the heating rate was chosen to be $0.5\,\text{K} \cdot \text{min}^{-1}$ with eight steady-state interrupts. After finishing the first run, a visual inspection of the furnace (heating section) showed a couple of cracks as expected which proved to stabilize in the next few experiments without negative effects on the operation.

Detailed evaluation of the measured temperature histories shows the establishment of a steady state after roughly 8 or 10 h. The readings of the respective five thermocouples in one measuring cross section are regularly found to keep within the limits of accuracy (for example ±4 K for type B elements at 1,600°C). Outside this central region the temperatures begin to decline symmetrically in the radial direction. This finding is supported by slight concentric modifications of the refractory surface color at the upperside of the measuring section.

The accuracy and sensitivity of the calorimetric heat flow measurements have been checked by varying water flow rates and respective temperature increases. The water temperature difference $(\Delta T)_{\text{water}}$ decreases with the furnace temperature due to the simultaneously reduced heat flow rate $\dot{Q}$, and 500°C should be regarded as the lower limit of the furnace temperature for effective thermal-conductivity measurements with the PMA4. Below that, the uncertainty of the water temperature difference measurement starts to increase rapidly. Nevertheless, the limiting furnace temperature (500°C) allows thermal-conductivity measurements at a mean temperature of about 300°C in the lower layers of the sample.

3.2 Numerical Investigations

These first experiments have been supplemented by a three-dimensional finite element (FEM) simulation of the measuring facility using a commerical code by MSC-MARC. With the temperature-dependent properties taken from producers' catalogs and from our measurements with different facilities, the steady-state temperature and heat flux distributions have been obtained. These results will not be discussed here in detail; however, important findings and conclusions will be summarized as follows:

- *Validation:* Numerous additional thermocouples have been installed inside the PMA4 during the construction to obtain more information about establishment of the steady state, but also for validation of the FEM simulations. All the measurements inside and close to the sample and also inside the surrounding heating section show excellent agreement with the FEM calculations, i.e., the temperature differences are well within the accuracy of measurements. Some deviations are found at far away locations in the outer parts of the lightweight firebricks in the measuring section, see Fig. 2, with the calculated temperatures slightly above the measured ones. This is probably due to questionable data for the refractories as taken from producers' catalogs.
- *Temperature distribution inside the sample:* The isotherms have been confirmed to be exactly plane surfaces being parallel to each other and also to the upper side of the calorimeter. This holds for the central part, and deformation of the isotherms (decreasing temperatures) clearly begins outside the measuring cross section.
- *Heat flow distribution inside the sample:* Temperature gradients orthogonal to the vertical (measuring) direction would yield lateral heat losses resulting in erroreous measuring results. All the FEM simulations with wide ranging parameter variations confirmed the unidirectional character of the heat flow in the central (measuring) part of the sample with lateral heat losses only far outside this region.

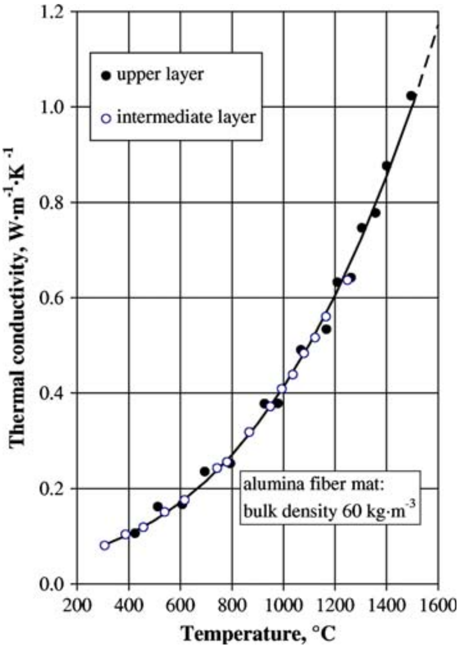

Fig. 4 PMA4 measurements: thermal conductivity of an alumina fiber mat (bulk density of 60 kg · m^{-3})

4 Effective Thermal Conductivity Results with Fiber Mats

After finishing the first experiments and numerical studies, PMA4 was part of an extended research project where commercial insulations have been investigated with various thermal-conductivity measuring devices. A comparison of alumino-silicate fiber mat measurements (maximum service temperature of 1,250°C) showed very good agreement for all panel test results, including those from PMA4 [1,2].

Further measurements have focused on materials with a higher application temperature (i.e., alumina fiber mats, 72% Al_2O_3, maximum service temperature of 1,650°C). Aging proved to be a very serious problem for the uppermost thermocouples where a strong drift of the reading was found after repeated application above 1,300°C which is thought to be due to diffusion processes. Some of the first measured series had to be repeated, and after that, only new thermocouples have been used for such sensible locations. Regular calibration of all the applied thermocouples is a must.

Figures 4 and 5 show some of the results measured with a three-layer sample with individual thickness of 23–24 mm and the furnace temperature up to 1,650°C. Measurements have been taken for increasing and decreasing temperatures.

Depending on the three-layer arrangement, two series of results have been obtained with excellent agreement, one for the upper and one for the intermediate layer with maximum mean temperatures of 1,500 and 1,250°C respectively (Fig. 4). These results are supplemented (Fig. 5) by respective PMA2 results (maximum furnace temperature of 1,300°C), and additional data for larger bulk densities are included. For the latter

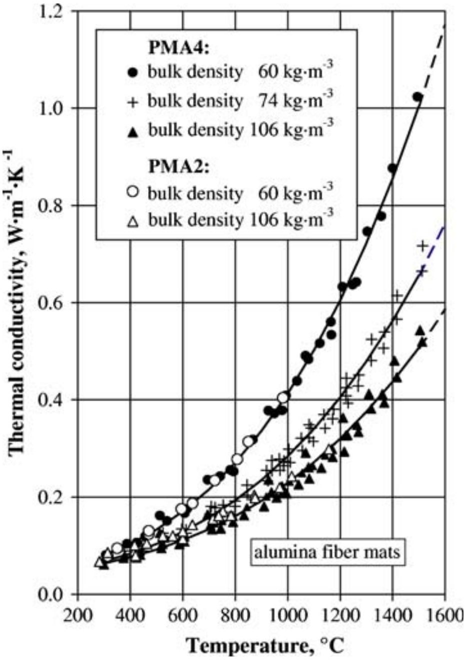

Fig. 5 PMA4 measurements: thermal conductivity of alumina fiber mats in comparison with PMA2 results

ones, the scatter of data proved to be much stronger, a behavior which can be attributed [1,2] to the general experience of strong local density variations in these fiber mats. The relative expanded uncertainty of the measurements has been evaluated to be within 1.6 and 6.2%, following the *Guide to the Expression of Uncertainty in Measurements* [5], including the limited precision of the involved measuring instruments and also statistical deviations of the measured values.

5 Advanced Evaluation—'True' Versus 'Mean' Thermal Conductivity

Thermal conductivities of high-temperature porous media measured with various methods occasionally exhibit different results. This can partially be due to the relatively high-temperature difference within the sample leading to the mean effective thermal conductivity,

$$\lambda_{\text{eff}}\,(T_{\text{mean}}) = \lambda_{\text{eff}}|_{T_2}^{T_1} = \frac{1}{T_1 - T_2}\int_{T_2}^{T_1} \lambda_{\text{eff}}\,(T)\mathrm{d}T \tag{3}$$

In contrary to steady-state procedures, transient hot-wire measurements are performed at nearly constant temperature yielding results which occasionally are called the 'true' thermal conductivity [6,7]. The difference between 'mean' and 'true' values may grow very large in cases of a strong nonlinear conductivity versus temperature relationship and, additionally, for large temperature differences within the sample.

Both the comparison of results from two different sources and also the practical application of measured data are needed for transformation of 'mean' into 'true' values by respective advanced evaluation of measured data. Bolte [8] was the first to suggest a procedure for the point-by-point transformation based on the temperature relationship $T_2 = f\,(T_1)$ for the sample with T_1 and T_2 as the hot- and cold-side temperatures, respectively, and additionally on a knowledge of the 'true' thermal conductivity $\lambda_{\mathrm{eff}}\,(T_2)$ for the lower temperature limit which is, however, usually not available.

An advanced evaluation procedure can be applied if the type of function $\lambda_{\mathrm{eff}}\,(T)$ in Eq. 3 is available, e.g., from the physical background. There will be some unknown parameters in it, which can easily be adjusted by application of the mean square error minimization method on Eq. 3 to minimize the difference of calculated (left-hand side) and measured thermal conductivities (right-hand side):

$$f\,(a, b, c, \ldots, T) = \sum_{i=1}^{k} \left[\left(\frac{1}{T_1 - T_2} \int_{T_2}^{T_1} \lambda_{\mathrm{eff}}\,(a, b, c, \ldots, T)\ \mathrm{d}T \right)_i - \left(\lambda_{\mathrm{eff}} |_{T_2}^{T_1} \right)_i \right]^2 \rightarrow \text{minimum} \tag{4}$$

A common function type for the effective thermal conductivity of fiber mats is suggested, e.g., by the DIN V ENV-1094 standard,

$$\lambda_{\mathrm{eff}}\,(T) = a\sqrt{T} + bT^3 \tag{5}$$

representing the superposition of gas-phase conduction and radiation. It can be shown mathematically that with typical parameters a and b for fiber mats for this type of function, the 'mean' thermal conductivity is always larger than the 'true' one.

This prediction is also valid for insulations like calcium silicate, but

$$\lambda_{\mathrm{eff}}\,(T) = a\sqrt{T} + b\frac{1}{T} + cT^3 \tag{6}$$

here is the more appropriate function type which includes additional contributions of the solid (crystal) phase. With this, the difference between 'mean' and 'true' values often is smaller as the function is similar to a linear relationship for most of calcium silicate materials. Anyway, the decisive role is played by the temperature difference across the sample. Due to the arrangement (see Sect. 2.3) of the various layers, temperature differences are relatively small and subsequently the differences between measured mean values and the thermal conductivities from the advanced evaluation are small, usually less than 1% for a layer thickness around 25 mm which is typical for the PMA4 measurements; however, increasing up to 4 and 5% for a thickness of 50 mm. For most of the measurements, there is no need for the efforts of advanced evaluation.

6 Conclusions

The design and operation of a new panel test facility for effective thermal conductivity measurements of insulations is reported. First tests showed the successful operation up to a maximum temperature of 1,650°C. Design and construction has been accompanied by numerical simulations that confirm the application of boundary conditions and the further assumptions of the method. The first experimental thermal-conductivity results show good agreement with measurements from other facilities.

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