# A New Panel Test Facility for Effective Thermal Conductivity Measurements up to 1650 °C

G. Barth, U. Gross, R. Wulf

*Institut für Wärmetechnik und Thermodynamik, Technische Universität Bergakademie Freiberg, 09596 Freiberg, Germany, email: gross@iwtt.tu-freiberg.de*

## 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 °C and 1650 °C following ASTM C201-93 and DIN V ENV-1094 standards. Square shaped samples (length 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 1800 °C. The calorimeter system consists of a square central measuring zone (length 100 mm) surrounded by guarding loops to avoid heat losses in all directions. The samples, e.g. a number of fibre mats one on top of the other up to maximum height 110 mm, are open to ambient pressure and atmosphere (air). Measurements include heat flow rate (taken in the central calorimeter), temperature differences across individual layers of the sample (measured by series of thermocouples which regularly have to be calibrated), thickness of the respective layers (before and after the experiment). The thermal conductivities range from 0.025 to 2 $Wm^{-1}K^{-1}$, and both isotropic and non isotropic materials can be investigated due to the one dimensional characteristic of the temperature field. Measurements for alumina fibre mats are presented and good agreement is found with respective results from other methods and test facilities.

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## 1. Introduction

Development and application of extremely high temperature insulations is a big challenge for thermophysical properties measurements. Related investigations have been carried out since more than 25 years by a research group at the Institut für Wärmetechnik und Thermodynamik (IWTT) of TU Bergakademie Freiberg. Measuring facilities have been designed, constructed and operated representing various measuring principles. For more details regarding temperature and thermal conductivity ranges, geometry of the samples etc., see Wulf et al. (2004, 2005).

Extended experiments showed that non isotropic materials like, e.g., fibre mats are best investigated with panel test procedures following the standards DIN V ENV-1094 and ASTM C201/C202/C182 respectively despite the high efforts of the test procedures (Wulf et al., 2004, 2005). 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 the maximum application temperature which increased from 1150 °C in the first set up to 1450 °C in the next facility, PMA2, which is still in operation (for more details see Barth et al. (1995) and Gross et al. (2001)). In this contribution a new facility, PMA4, will be presented which has been designed and constructed for effective thermal conductivity measurements of insulations within 0.025 and 2 $\mathrm{Wm^{-1}K^{-1}}$ in the temperature range between 300 and 1650 °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 heat flow rate $\dot{Q}$. After establishment of a steady state the effective thermal conductivity can easily be evaluated from the measured temperature difference $\Delta T$ across the sample following

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

with $A$ and $d$ as the cross section area and thickness of the sample respectively.

This sounds easy, however, lots of problems have to be overcome with respect to the establishment of an actually one dimensional heat flow and to the best possible avoidance of further measuring errors.

### 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 with some details of the design.

| | PMA2 | PMA4 |
|---|---|---|
| Temperature range / °C | 300 to 1450 | 300 to 1650 |
| Sample dimensions / mm | 300 x 300 x 120(max) | 400 x 400 x 110(max) |
| Active cross section / mm | 100 x 100 | 100 x 100 |
| Heating elements | | |
| Number | 18 | 15 |
| Material | SiC | Moly-D (U shaped) |
| Arrangement | Horizontal | hanging vertically |
| Construction materials | | |
| Heating section | lightweight firebricks | ceramic fibreboards |
| Measuring section | lightweight firebricks | lightweight firebricks |

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

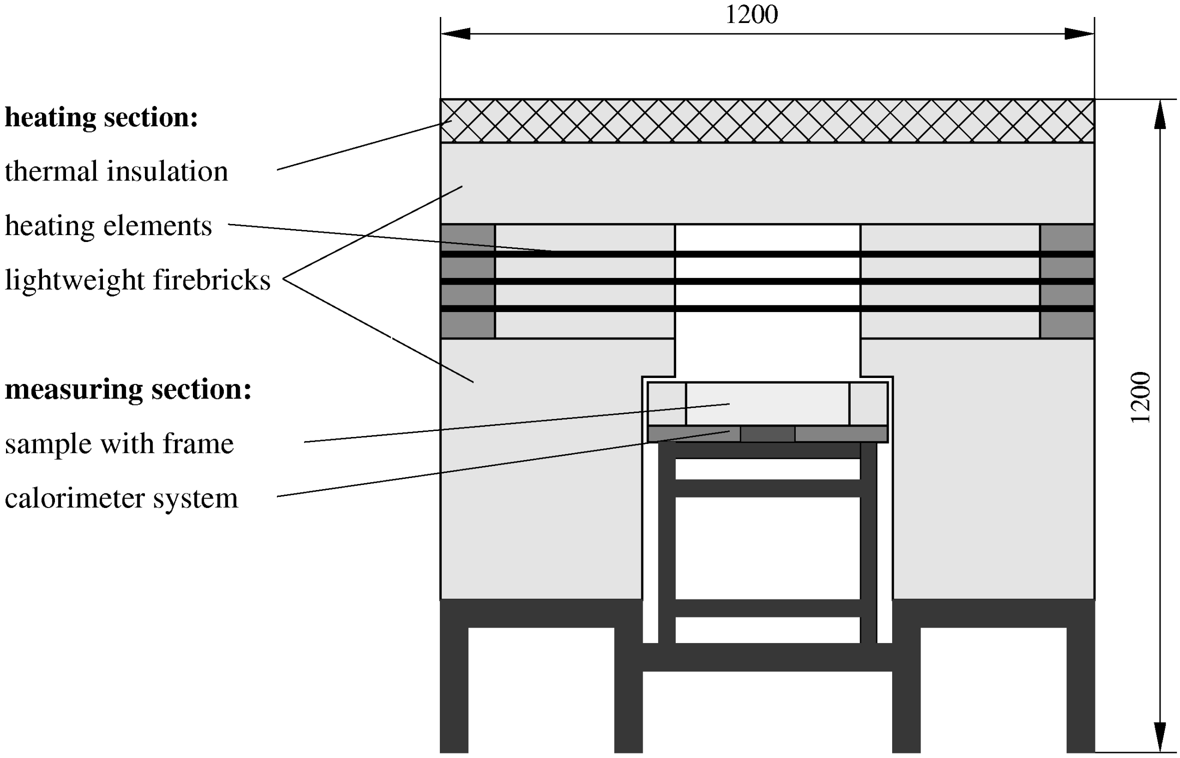

Fig. 1: Schematic drawing of panel test facility PMA2

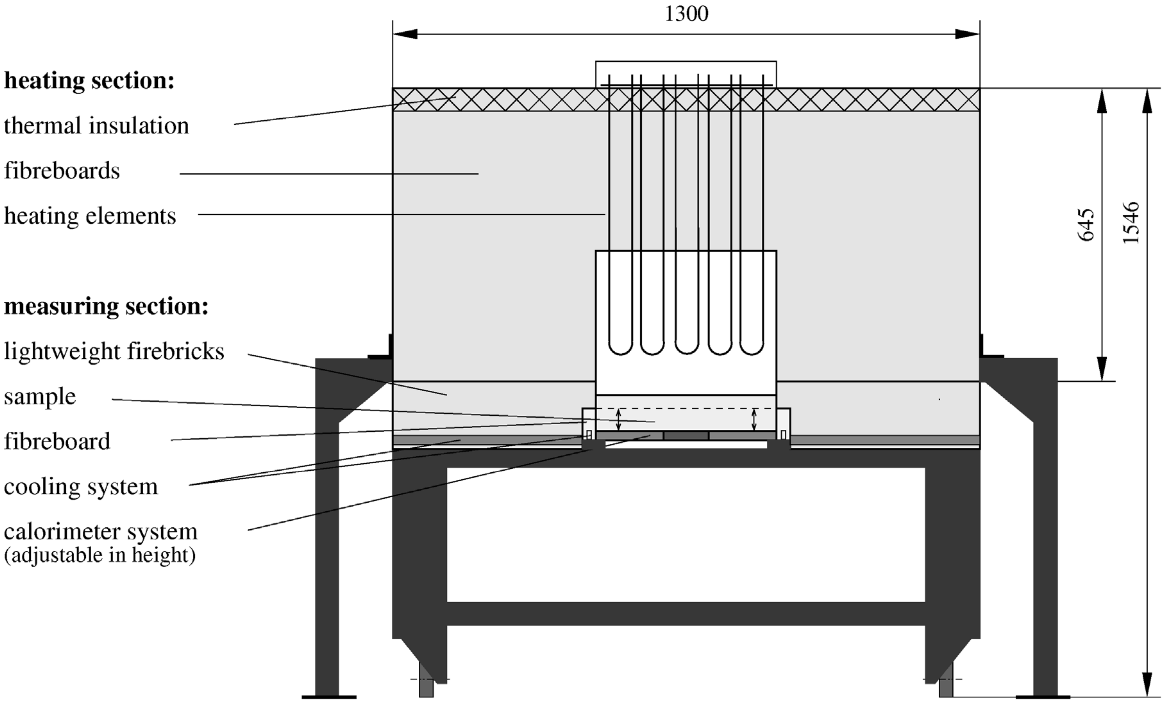

Fig. 2: Schematic drawing of panel test facility PMA4

### *Heating section*

The PMA4 heating section is erected by ceramic fibreboards with 4 different specifications depending on the temperature demands. The lower part of the heating section is formed by vertical plates which are cut off in the central part creating an enclosure with square cross section (length 400 mm, height 280 mm). 15 U-shaped Moly-D heating elements freely hanging inside this furnace are arranged for getting a uniform temperature distribution at the upper sample´s front surface. With respect to the designed maximum temperature 1650 °C, heavy duty fibreboards have been chosen (maximum service temperature 1800 °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 aluminium as mechanical protection sheat. The ceramic fibreboards 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 a that extreme thermal environment.

### *Measuring section*

The measuring section is designed for most 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 the cross section of 400 x 400 mm$^2$ consists of one central calorimeter for the heat flow measurements (100 x 100 mm$^2$) surrounded by three additional calorimeters for thermal protection. Water is supplied to this system in a closed cycle with controlled flow rate and temperature by respective cooling systems.

Much is done to avoid any lateral, i.e. horizontal, heat flow from/to the central (measuring) calorimeter: 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 section 2.4), application of additional cooling coils in the surrounding refractories, and others.

## 2.3 Sample temperature measurements

The PMA4 measuring device is designed for plate and mat shaped insulations with a cross section of 400 x 400 mm$^2$. Within the total height of 110 mm several samples can be used one on top of the other with respective temperature measurements above, below and in between. Doing this, 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 5 thermocouples are fixed in each of the cross sections where type K and B elements are used below and above 1000 °C respectively being regularly calibrated before and after application. As an example a three sample arrangement is shown in fig. 3 enabling data logging and evaluation for the upper two ones.

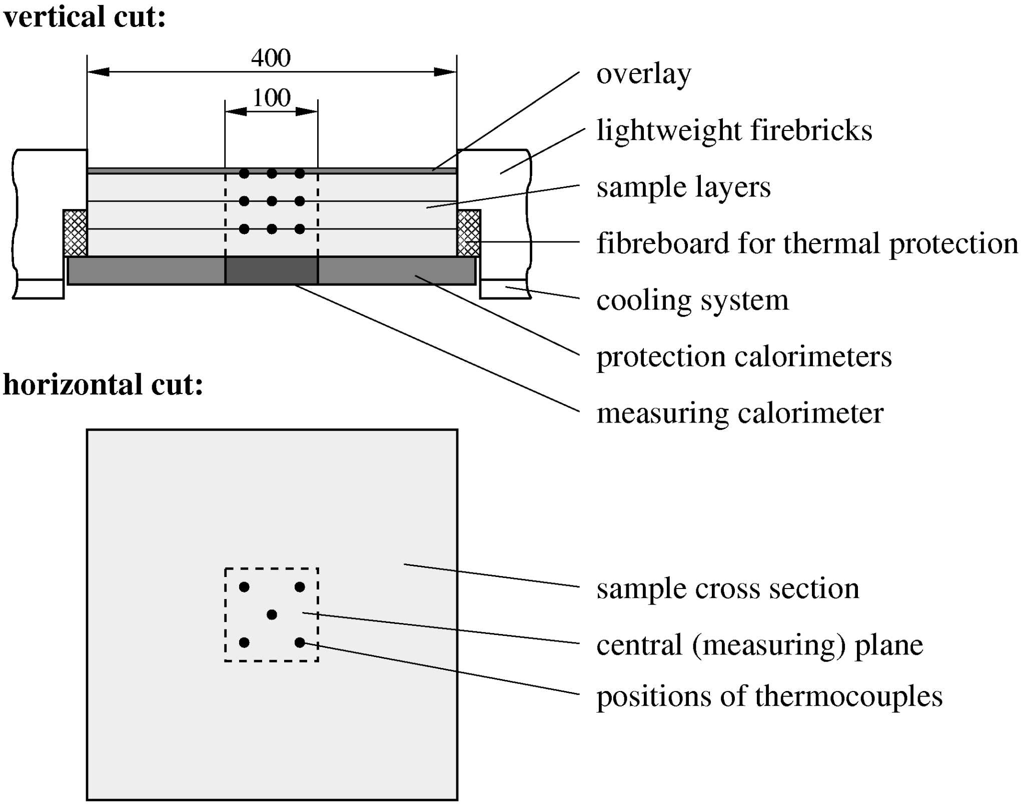

Fig. 3: Arrangement of thermocouples for a sample consisting of three layers

## 2.4 Heat flow measurements

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

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

with $V_{water}$ as the volume of a measuring cylinder and $\Delta\tau$ as the time to fill it between two levels given by two electric resistance sensors. Before starting a water flow rate measurement the difference of the outlet temperatures of the measuring and the first protection calorimeter is controlled to be zero by respective variations of the flow rate. By this, optimum adiabatic conditions are realized for the central calorimeter as both of the calorimeters contain meandering channels being in parallel, with the inlet positions but 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 $\left(\Delta T\right)_{water}$ in the central part by a respective 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 automatic way. Two PCs, one for control and operation, the other one for the 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 respectively are organized but also the measuring and adjusting processes for the calorimeter system. All results are available online enabling the plot of spacial and temporal temperature profiles and also the 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 respective intermediate steady states for the measurements. The duration of such a series typically amounts to more than 7 days in the automized version. Respective protection systems for, e.g., emergency cooling allow the facility to be run 24 hours and 7 days a week with online information by internet about status and measured results.

## 3. First experiments and numerical simulation

*Experiments*

For the first tests a ceramic fibreboard has been used as a three layer sample with the total height 92 mm. The regular set of thermocouples (see section 2.3) has been supplemented by additional temperature sensors outside the central (measuring) cross section to obtain more informations about the temperature profile. In these experiments the heating rate war chosen to be 0.5 $\mathrm{Kmin}^{-1}$ with 8 steady state interrupts. After finishing the first run, a visual inspection of the furnace (heating section) brought 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 hours. The readings of the respective five thermocouples in one measuring cross section are regularly found to keep within the limits of accuracy (for example ± 4K for type B elements at 1600°C). Outside this central region the temperatures begin to decline symmetrically in radial direction. This finding is supported by slight concentric modifications of the refractory surface colour at the upperside of the measuring section.

Accuracy and sensitivity of the calorimetric heat flow measurements have been checked for by varying water flow rates and respective temperature increases. The water temperature difference $(\Delta T)_{water}$ 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 sensibly. Nevertheless, the limiting furnace temperature (500 °C) allows thermal conductivity measurements at the mean temperature of about 300 °C in the lower layers of the sample.

*Numerical investigations*

These first experiments have been supplemented by a 3D finite element (FEM) simulation of the measuring facility using a commerical code by MSC-MARC. With the temperature dependent properties taken from producers´ catalogues and from own measurements with different facilities, 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 now:

- *Validation:* Lots of additional thermocouples have been installed inside the PMA4 in course of the construction for to get 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. Some deviations are found at far away locations in the outer parts of the measuring section which probably are due to questionable data for the refractories as taken from producers´ catalogues.
- *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 the 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 bringing 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.

## 4. Effective thermal conductivity results with fibre 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 (see Wulf et al., 2004, 2005). A comparison of aluminosilicate fibre mat measurements (maximum service temperature 1250 °C) showed very good agreement of all panel test results including those from PMA4 (see Wulf et al., 2004).

Further measurements have been focused on materials with a higher application temperature (i.e. alumina fibre mats, 72 % $Al_2O_3$, maximum service temperature 1650 °C). Ageing proved to be a very serious problem for the uppermost thermocouples where a strong drift of the reading was found after repeated application above 1300 °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.

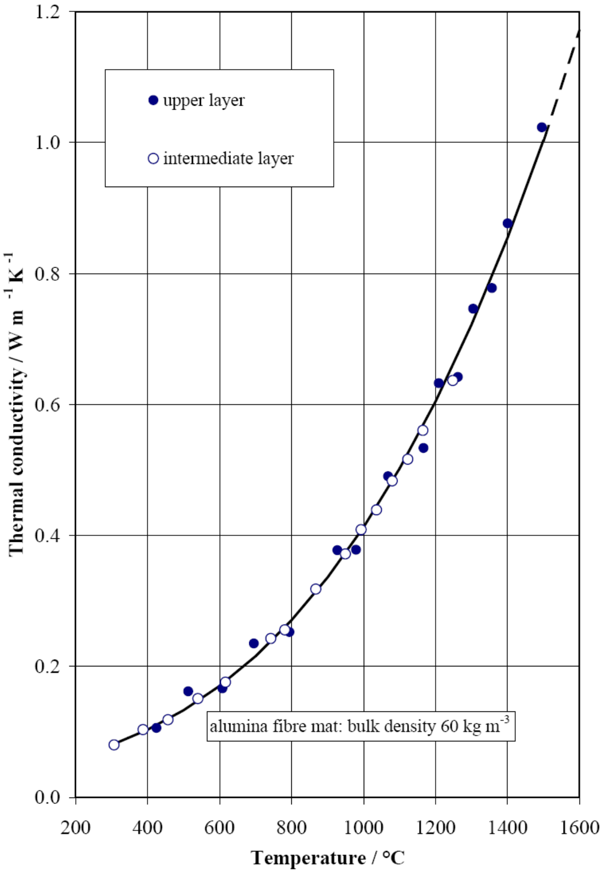

Fig. 4: PMA4 measurements: thermal conductivity of an alumina fibre mat (bulk density 60 kg $m^{-3}$)

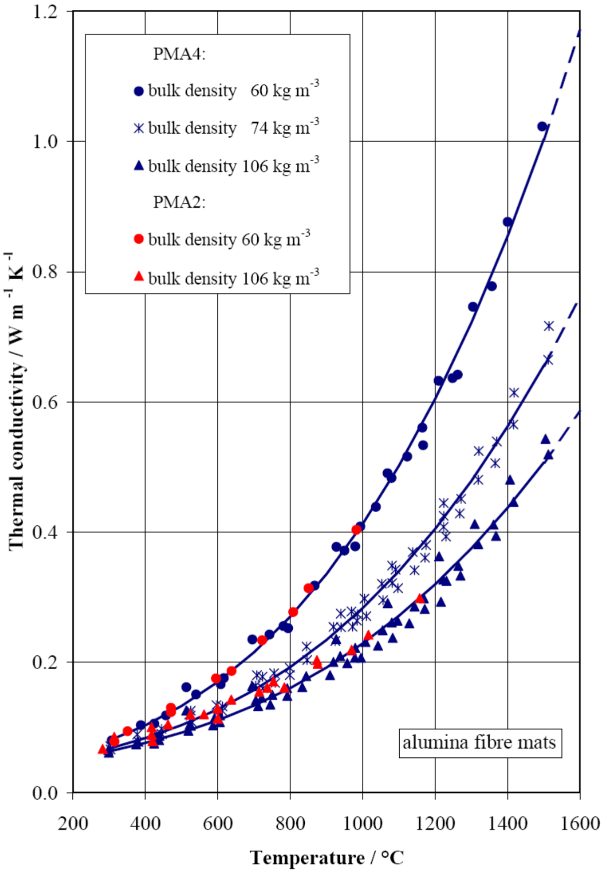

Fig. 5: PMA4 meaurements: thermal conductivity of alumina fibre mats in comparison with PMA2 results

Figs. 4 and 5 show some of the results measured with a three layer sample with individual thicknesses of 23 to 24 mm and the furnace temperature up to 1650 °C. The respective measurements have been taken for increasing and decreasing temperatures as well. 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 the maximum mean temperatures 1500 °C and 1250 °C respectively (fig. 4). These results are supplemented (fig. 5) by respective PMA2 results (maximum furnace temperature 1300 °C) and additional data for larger bulk densities are included. For the latter ones the scattering of data proved to be much stronger, a behaviour which can be attributed (see Wulf et al., 2004, 2005) to the general experience of strong local density variations in these fibre mats. The uncertainty of the measurements has been evaluated to be within 1.6 and 6.2 %, following GUM (1995).

## 5. Advanced evaluation – ′true′ vs. ′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

$$\lambda_{eff}\left(T_{mean}\right) = \lambda_{eff}\Big|_{T_2}^{T_1} = \frac{1}{T_1 - T_2}\int_{T_2}^{T_1} \lambda_{eff}\left(T\right)dT \tag{3}$$

In contrary to steady state procedures, transient hot-wire measurements are performed at nearly constant temperature yielding results which occasionally are called ′true′ thermal conductivity (Krönert, 1987 and Anderson/Mikkelson, 2000). The difference between ′mean′ and ′true′ values may grow very large in cases of a strong non linear conductivity vs. temperature relationship and, additionally, for big temperature differences within the sample. Both, the comparison of results from two different sources and also the practical application of measured data is asking for transformation of ′mean′ into ′true′ values by respective advanced evaluation of measured data. Bolte (1957) was the first to suggest a procedure for the point-by-point transformation based on the temperature relationship $T_2 = f\left(T_1\right)$ for the sample with $T_1$ and $T_2$ as the hot and cold side temperatures respectively and additionally based on the knowledge of the ′true′ thermal conductivity $\lambda_{eff}\left(T_2\right)$ for the lower temperature limit which is, however, usually not available.

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

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

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

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

representing the superposition of gas phase conduction and radiation. Easily it can be shown mathematically, that for this type of function the ´mean´ thermal conductivity is always langer than the ´true´one.

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

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

is here the more appropriate function type including additional contributions of the solid (crystal) phase. With this, the difference between ´mean´ and ´true´ values is smaller as the function type is more similar to a linear relationship. Anyway, the decisive role is played by the temperature difference across the sample. Due to the arrangement (see section 2.3) of the various layers temperature differences keep relatively small and subsequently the differences between measured mean values and the thermal conductivities from the advanced evaluation are small keeping regularly below 1 % for layer thickness around 25 mm which is typical for the PMA4 measurements, however, rising up to 4 and 5 % in case of 50 mm. For most of the measurements there will be no need for the efforts of advanced evaluation.

## 6. Conclusions

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 the maximum temperature of 1650 °C. Design and construction has been accompanied by numerical simulations which confirm the correctness of boundary conditions and further assumptions of the method. First experimental thermal conductivity results show good agreement with respective measurements from other facilities.

## Acknowledgement

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