Intercomparison of Insulation Thermal Conductivities Measured by Various Methods

R. Wulf, G. Barth, U. Gross

Institut fuer Waermetechnik und Thermodynamik, Technische Universitaet Bergakademie Freiberg, 09596 Freiberg, Germany, email: gross@iwtt.tu-freiberg.de

Abstract

In the present contribution systematic effective thermal conductivity measurements with different methods are reported for various materials. Some of these are isotropic (calcium silicate) some of them not (alumino-silicate and alumina fibre mats). The measurements were done with two different steady-state panel test facilities (according to ASTM C201, self designed and constructed), two guarded hot plate facilities (ISO 8302, with one and with two samples respectively), one steady-state radial heat flow facility (self designed) and one transient hot-wire instrument (DIN EN 993-14). These facilities are operated at ambient pressure and atmosphere (air) between 20 °C and 1650 °C, and they are briefly described in the paper. The results show the well known increase of conductivity with the temperature mainly due to radiation heat transfer. In case of the isotropic calcium silicate material (bulk density 220 kg m^{-3}) no significant differences between the various methods have been found and the results can easily be correlated within ±10 %. The fibre-mat results, however, show additional effects of the density (between 103 and 170 kg m^{-3}) and the fibre orientation. Big differences exceeding 30 % are found between plate and hot-wire results.

1. Introduction

The effective thermal conductivity of high temperature insulations is one of the decisive selection criteria for furnace construction. Respective materials consist of a porous structure providing an adequate mechanical stability with open or closed small scaled pores filled by gas. The solid phase may be continuous or not forming foams, fibre mats and powders respectively with a temperature resistivity which is imperative. Open pores regulary contain the ambient atmosphere (mostly air) whereas the gas inside closed pores can be optimized for achievement of both a low molecular and subsequently a low effective conductivity of the

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insulation. For the measurement of the latter one various methods are available with different expenses in time and cost. It is well known from the literature that different measuring methods occasionally bring different results for one and the same material. Comparison between various insulations is made more difficult by this and uncertainties arise for practical application of measured data as given in producers´ catalogues which typically do not include informations about the underlying measuring principle. In the present contribution systematic experimental investigations of commercial insulations are reported for temperatures between 20 °C and 1650 °C at ambient pressure covering isotropic and non isotropic materials, various measuring devices and respective intercomparisons.

2. Mechanisms of heat transfer in porous media

Conduction heat transfer in porous media is govered by porosity, the molecular conductivities of gas and solid, the kind of solid structure (i.e. continuous or not, fibre or grain shaped), pore size and its distribution and finally the gas pressure. The conductive transport is superposed by radiation processes which may the dominating ones, or they may vanish which depends on temperature and overall porosity. Convection effects are usually negligible if the pores are small enough ($d < 5$ mm, following Häussler and Schlegel (1995)) corresponding to large enough bulk densities in case of fibre insulations ($\rho > 20$ kg m^{-3}, see Stark (1991)). The governing processes of heat conduction and radiation are strongly linked to each other depending on the kind of structure. Physical models which have been developed for the various insulations are more often very complicated requiring fundamental properties like the extinction coefficient, refractive index etc. Prediction of the effective thermal conductivity without any measurement of this overall characteristic is still impossible up to now.

3. Previous comparative studies

All the methods for effective thermal conductivity measurements are based on solutions of Fourier´s differential equation for the temperature field adapted to respective boundary conditions. Such methods may be classified with respect to the underlying principle (steady state vs. transient, absolute vs. relative), to the sample geometry (plane, cylindrical, spherical), to the temperature and conductivity ranges and others. A review of standard procedures is given, e.g., by Maglic et al. (1984) .

One of the commonest is the transient hot-wire method according to DIN EN 993-14/15, ASTM C1113 and ISO/DIS 8894 standards characterized by simple arrangement and short duration of the measurements with, however, serious problems in cases of non isotropic and low bulk density materials. Application of further transient methods with hot-strip and hot-disk sensors respectively are less often found.

Steady-state methods are mostly operated with plane plate-shaped samples as applied in the guarded hot-plate and the heat flow meter methods (DIN 52617, DIN EN 12664/12667 and ISO 8302 respectively) and the panel-test method including a calorimeter for the heat flow rate measurements (ASTM C201/C202/C182 and DIN V ENV 1094-3). Hollow cylindrical samples are occasionally used in steady-state procedures standardized as DIN 52613 and DIN EN ISO 8497 respectively. Steady-state methods usually require more efforts with respect to the experimental set-up and they are time consuming. On the other hand, these methods are characterized by high accuracy enabling measurements of non isotropic materials due to their well defined one dimensional heat flow in contrary to the transient hot-wire principle. The equation for the evaluation of steady-state measurements is very simple delivering however averaged thermal conductivities within a respective temperature range. This calls for additional evaluation procedures following, e.g., Bolte (1957) or Barth et al. (2005) for getting the socalled ´true´ thermal conductivity at a specified temperature. The necessity of such corrections depends on the curvature of the conductivity vs. temperature plot and also on the extension of the measured temperature difference across the sample.

The effective thermal conductivities of high temperature construction and insulation materials measured with the various methods quite often strongly deviate from each other. An additional problem is the lack of high temperature standard reference materials for appropriate comparison of the various methods.

There are some previous comparative reports in the open literature. Koltermann (1961) was the first to find differences between the effective thermal conductivities measured by various methods which have briefly been discussed and attributed to experimental insufficiency.

Some years later, Eschner et al. (1974) report interlaboratory experiments with lightweight firebricks at temperatures up to 1000 °C where hot-wire results exceed respective panel-test data by up to 15 %. Some time later, Davis and Downs (1980) and Hagemann and Peters (1982) report similar findings which have been attributed to a certain non isotropy of the samples which leads to some kind of a mean value in case of the hot-wire experiments.

Dietrichs (1987) confirmed this behaviour for vacuum formed fibreboards where the difference amounts to about 20 %.

In contradiction to this, Aksel´rod and Vischnewskii (1984) report good agreement between hot-wire and steady-state cylinder results for ceramic fibre mats and powder insulations, whereas hot-wire data exceed those from a panel-test facility in case of refractories. Similar experiences come from Schlegel (1988) who found this behaviour for calcium silicate where the differences range between 6 % and 33 % which has been attributed to the much higher temperature differences in case of the steady-state panel method. On the other hand, Neumann and Hemminger (1988) found good agreement between these methods applied to a porous insulation material (trade name Microtherm) and they pointed out the importance of problems due to non isotropy.

More recently Andersen and Mikkelsen (2000) reported hot-disk measurements of various insulations comparing their result with panel test data. They discuss the differences between the temperature fields which are one dimensional (cartesian) for the panel, one dimensional (cylindrical) for the hot wire, and in case of the hot disk it is characterized by some kind of superposition bringing an almost elliptical temperature field. Therefore, differences have to be expected for non isotropic materials. Another reason for differences between results from the various methods is thought to be due to the resulting ´mean´ and ´true´ thermal conductivities and also the fluctuation of properties within the sample. In all cases, hot-disk measurements exceed the panel test results despite the fact that the samples have been isotropic. As a consequence the application of the panel-test method has to be preferred as the one dimensional heat flow corresponds to insulation practice.

Litovsky et al. (2003) found the thermal conductivity of alumina fibre mats directly measured by a panel-test facility to exceed by far those which have been evaluated from diffusivity measurements with a method of symmetrical monotonic heating at constant rate. This is thought to be due to a remarkable reduction of radiation in the latter case. Additionally the effective radiation contribution has been evaluated from measured optical properties of the fibre mats by application of complex radiation transport models for heterogeneous media with strong multiple scattering. For both of the methods the respective radiation contributions have been subtracted from the measured results for getting the pure conductive transfer, and the agreement has found to be good.

As a conclusion the literature review brought the following:

- the occurance of differences between effective thermal conductivities measured with various methods is widely known since a long time,
- this is especially true for non isotropic materials,
- the interpretation of the respective phenomena diverges and no systematic research has been reported up to now.

This has been motivation for the present investigations which are intended to provide a better understanding.

4. Measurement facilities

The experiments have been carried out with the various measuring facilities available at the *Institut für Wärmetechnik und Thermodynamik* (IWTT) of *TU Bergakademie Freiberg* (table 1).

	Method	Sample dimensions mm	Temperature °C	Atmosphere
PMA1	Guarded hot plate (steady state)	250 x 250 x 70 (max)	20 to 80	Air
PMA2	Panel test (steady state)	300 x 300 x 120 (max)	300 to 1450	Air
PMA3	Guarded hot plate (steady state)	2 x (300 x 300 x 70 (max))	20 to 400	Air
PMA4	Panel test (steady state)	400 x 400 x 110 (max)	300 to 1650	Air
RA1	Radial heat flow (steady state)	60/12 (d_a/d_i) x 180	400 to 1450	Inert gas
HW1	Hot wire (transient)	2 x (120 x 125 x 65)	20 to 1200	Air

Table 1: Facilities for the effective thermal conductivity measurements of insulating materials available at IWTT of TU Bergakademie Freiberg

The panel test facility **PMA1** has been constructed from commercially available components and it is applicable for thermal conductivity measurements of plate shaped bad conducting materials close to room temperature. The effective thermal conductivity is evaluated from the measured electrical heating power and the temperature difference across one single plate.

PMA3 is a commercial guarded hot plate facility (Anter Corp., USA) with two identical plate shaped samples and the heat source in between. The thermal conductivity is evaluated from the supplied electric power and the measured temperature differences across both of the two samples.

There are two additional facilities with plate shaped samples, **PMA2 and PMA4**, see Barth et al. (1995), Barth et al. (2005) which have been designed and constructed at IWTT. Both of them are operated following the steady-state panel test method. The heat flow rate is measured by means of the central part of a guarded calorimeter system and the temperature differences across two or three plate shaped samples arranged one on top of the other. PMA2 and PMA4 differ with respect to maximum operation temperatures and some details of design, construction materials, electrical heating elements and the sample dimensions.

RA1 is a cylindrical steady-state facility also self designed and constructed. The heat flow is created electrically by a central heating rod from graphite and after passing radially through the sample it is absorbed in a water cooled jacket enclosing the entire arrangement. This cooling envelope is made from three sections one after the other in axial direction with the middle part acting as calorimeter for the heat flow rate measurements. The effective thermal conductivity is evaluated from the temperature differences measured at two different radial positions each with three thermocouples. The entire RA1 facility can be evacuated and filled with various gases which, however, must not be oxidizing. Due to its compact arrangement the time for one single measurement (around 3 hours) is much shorter when compared with the panel test facility (up to 10 hours).

The transient hot-wire method, **HW1**, has been applied in the cross wire configuration with the measurements mainly done at the *Institut für Keramik, Glas- und Baustofftechnik* of TU Bergakademie Freiberg.

5. Results for isotropic calcium silicate materials

Two different types of calcium silicate have been investigated (table 2) and preliminary results for the first one have been published earlier, see Gross et al. (1999).

	Material 1	Material 2
Composition: CaO / SiO_2	41 % / 42 %	45 % / 47.5 %
Maximum service temperature	900 °C	1100 °C
Bulk density	220 kg m^{-3}	280 kg m^{-3}
Pore radius: Mean value / range	0.3 µm / 0.15 to 0.35 µm	0.2 µm / 0.1 to 0.3 µm
Phase analysis	Xonothlite	Xonothlite

Table 2: Characterization of the calcium silicate materials under investigation

Fig. 1 shows results for material 1 measured with the various methods in all of the three cartesian directions (x-y-z, referred to an arbitrary system). As expected for this kind of material an isotropic behaviour is found with good agreement of all the results. The RA1 data exhibit a slightly smaller increase with temperature and some of the points do not meet the ±10 % range covering all the other data. This behaviour is attributed to physical transformations inside the sample materials at elevated temperatures. In course of the first heating up of calcium silicate, water vapour escapes from the sample, see Schlegel (1989), and cellulose fibres embedded along the production process are oxidized leaving gas filled pores which modifies the effective thermal conductivity. Such effects happen at about 400°C for this material. Due to this experience all measurements have started at the intended maximum temperatures. However, it has been found after the first test series that temperature gradient in the sample of RA1 is so lange that the outer regions of the sample never achieve 400°C. That´s why we had to temper the samples at 500°C earlier to the measurements. With this the RA1 results are also found within the ±10 % range (fig. 1).

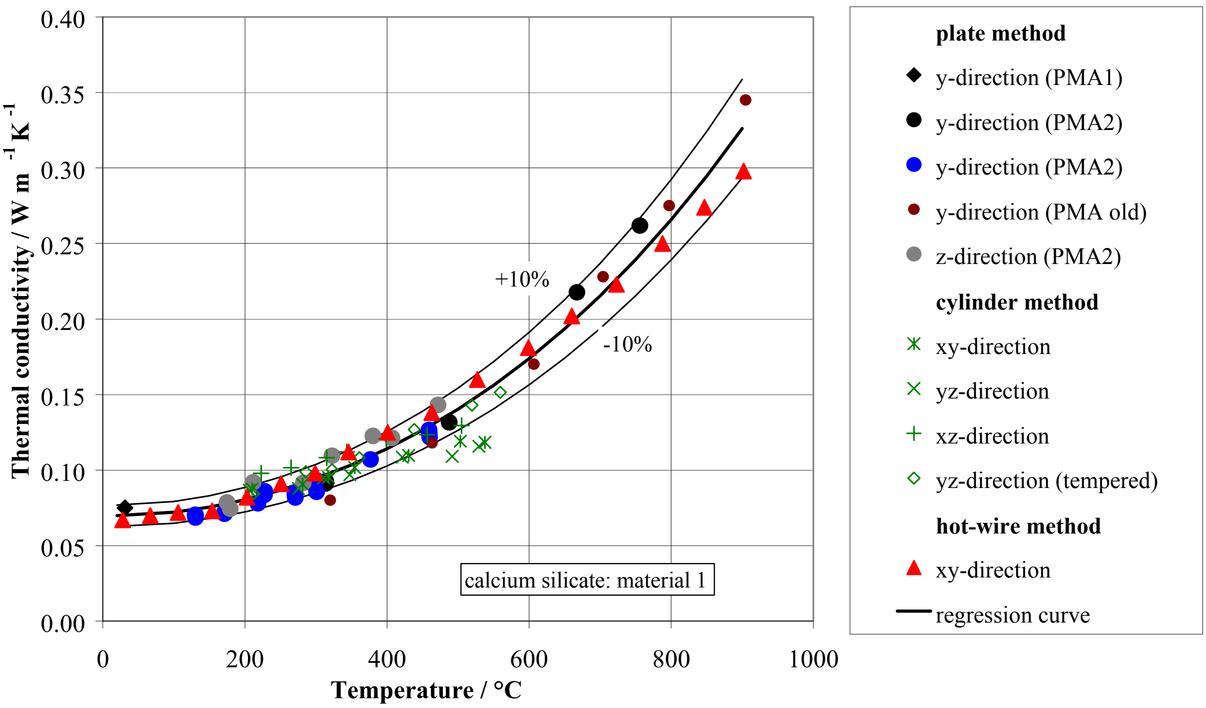

Fig. 1: Thermal conductivity of calcium silicate (material 1)

Material 2 has also been tempered at 500 °C and the thermal conductivity results confirm again the isotropic behaviour (fig. 2). The reproducibility has been tested by two PMA2

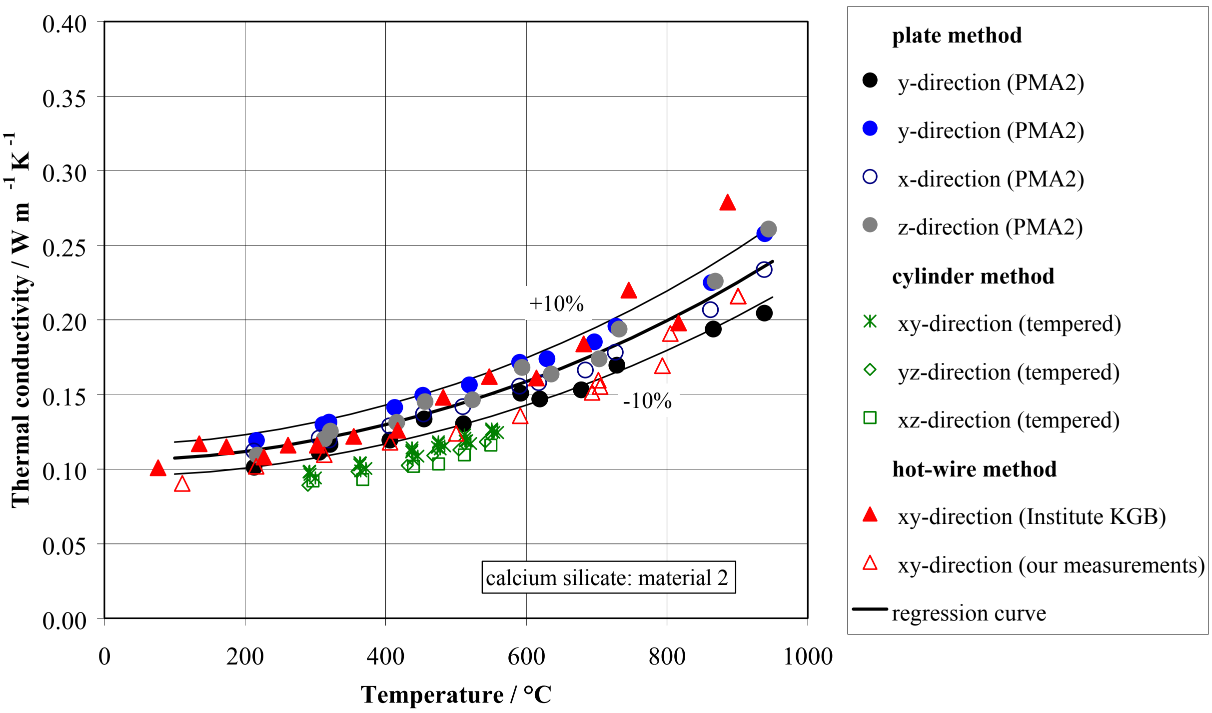

Fig. 2: Thermal conductivity of calcium silicate (material 2)

measuring series with different samples from the same material and the results are found within ±10 % limits where the variations are attributed to the observed big non homogeneity within this kind of material. All the data measured with the various methods are again found within this ±10 % range besides some from RA1 which are systematically smaller. Surprisingly these results agree well with those from hot-wire experiments (not shown in fig. 2) which have been taken without tempering the sample. This leads to the assumption that the physical transformations of the RA1 material has not been completed in course of preconditioning material 2 at 500 °C, and even higher temperatures would have been needed for it.

6. Results for non isotropic fibre mats of alumina and alumino-silicate

Fibre mats are characterized by a strong non isotropy with the preferred fibre orientation (fig. 3) in the main plain of a fibre mat (called xz-plane, see fig. 4) due to the production

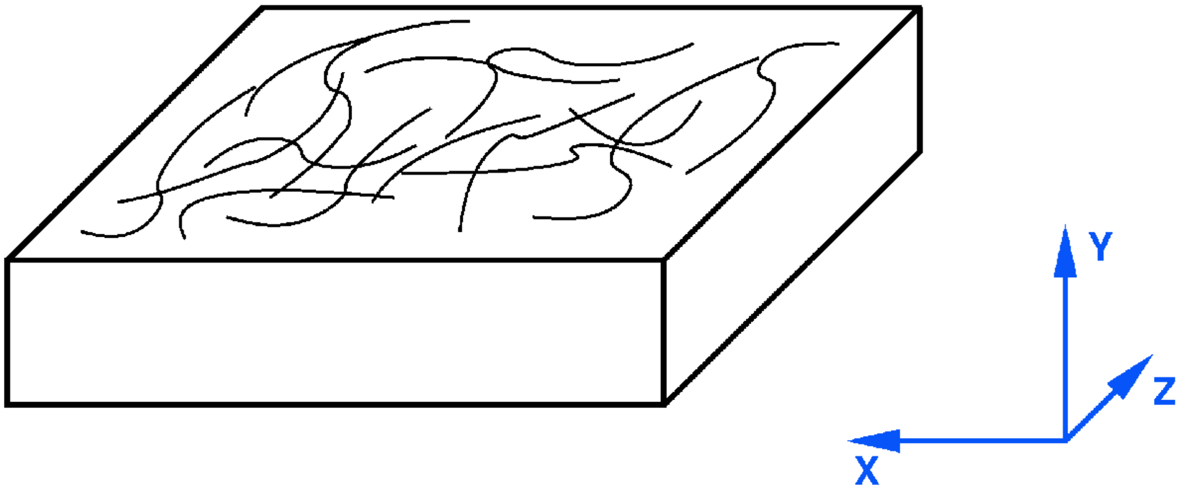

Fig. 3: Definition of the x-, y-, and z-directions (fibre mats)

process with the orthogonal (y) as the low conductivity direction when the insulation is applied. The panel-test facilities allow one dimensional measurements in the normal direction (y) but also orthogonal to this (x and z) after respective preparation of the fibre mat samples. In the RA1 arrangement the samples can be rolled in a spiralic manner (heat flow in y-direction) or they can be punched leading to a combined xz-arrangement with radial heat flow. The latter one is also found for the hot-wire measurements where the xy-, yz- and xz-directions have been investigated.

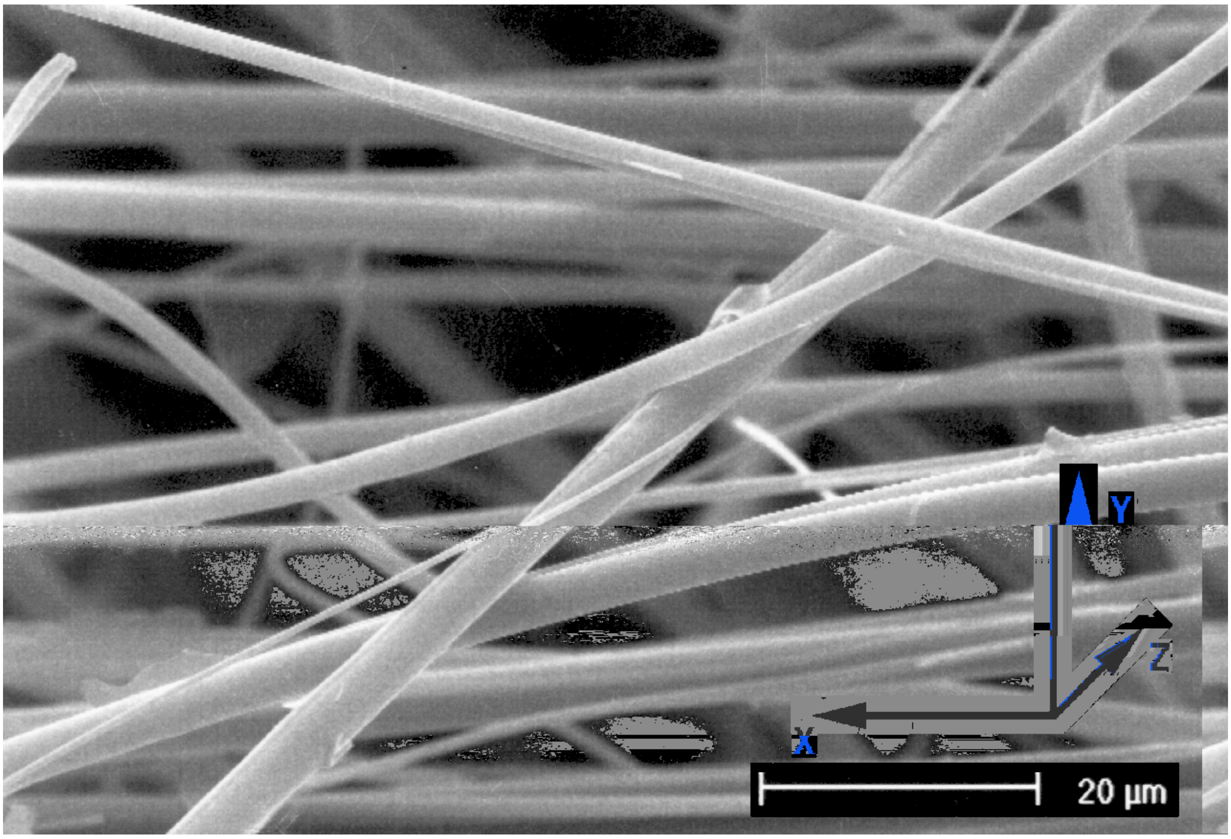

Fig. 4: Picture of an alumino-silicate fibre mat (scanning electron microscope)

Two different ceramic fibre mat materials have been selected (table 3), both of them in wide ranges of their bulk density, and first results have been reported by Gross et al. (1999) and Wulf et al. (2004).

	Material 3	Material 4
Composition: Al_2O_3 / SiO_2	Alumino-silicate 52 % / 48 %	alumina 72 % / 28 %
Maximum service temperature	1400 °C	1650 °C
Bulk density	95 to 170 kg m^{-3}	55 to 120 kg m^{-3}
Mean fibre diameter	2.5 µm	4 µm

Table 3: Characterization of the ceramic fibre mats under investigation

6.1 General remarks

The fibre mat measurements brought some additional problems which will be discussed in a first step:

- Measurements with the various test facilities at a certain fixed bulk density proved to be extremely difficult as the density of the samples varied within one and the same charge as delivered from the producer, and it diverged from the respective catalogue values. Due its great influence on the effective thermal conductivity the bulk density has been varied in wide ranges leading to lots of measurements with the various test facilities at respective constant temperatures. The resulting conductivity vs. density dependences allow comparisons at selected densities.
- For the alumino-silicate fibre mats the bulk density has been found to increase along with the experiment by typically 15 %, and up 30% in extreme cases. This is attributed to recristallization processes which depend on temperature and duration of exposition see, e.g., Dietrichs and Krönert (1981). Cristallization effects have been found by X-ray analysis whereas scanning electron microscopy gives no indications for any modification of the fibre structure. The shrinkage could only partially be anticipated by tempering. The measured effective thermal conductivities have finally been referred to the arithmetic mean of the bulk density before and after the measurements. In contrast to the alumino-silicate fibre mats, material 4 (alumina) did not show any density variations due to high temperature exposure.
- All hot-wire measuring series show a strong scattering for temperatures above 800 °C and evaluation of respective data proved to be impossible. Phase transitions are considered as one possible reason for this. Beyond this, hot-wire measurements are strongly disturbed by radiation effects in highly porous materials, see Gross and Tran (2004), an effect which becomes more serious with rising the temperature.
- RA1 sample preparation proved to be very difficult due to the small dimensions and the compressibility of the fibre mats yielding to non homogeneities and respective thermal conductivity variations which holds especially for the small bulk densities.

6.2 Bulk density effects

Figs. 5 and 6 show effective thermal conductivity (y-direction) vs. bulk density plots for the materials 3 and 4 respectively at selected temperatures between 400 °C and 1200 °C. The

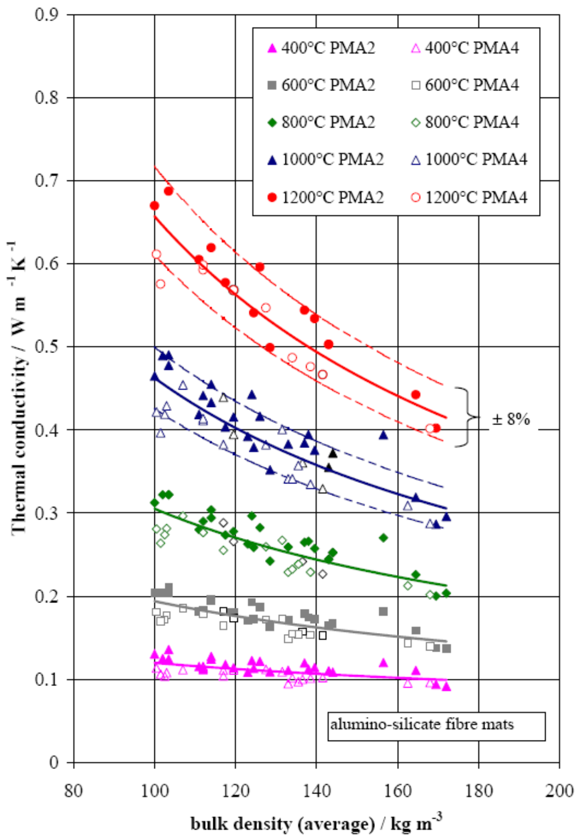

Fig. 5: Panel test results in y-direction: thermal conductivity vs. bulk density (alumino-silicate fibre mats)

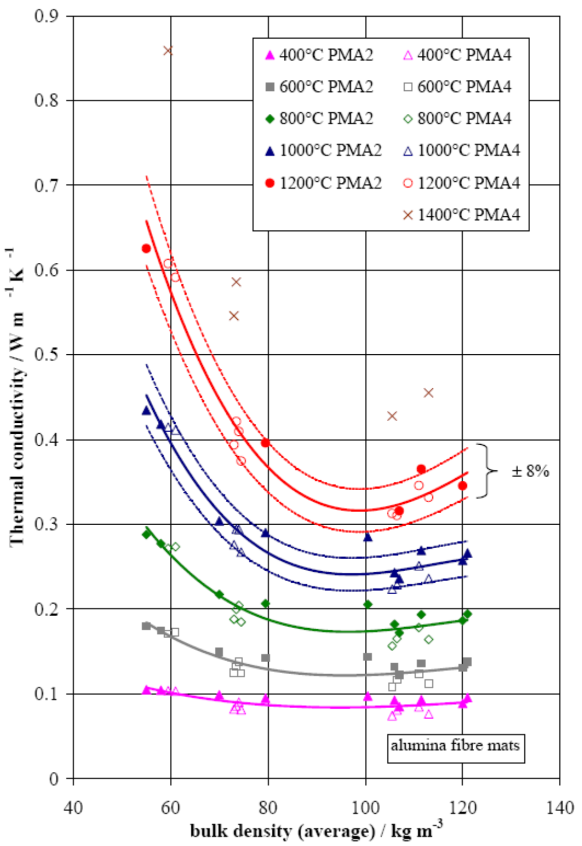

Fig. 6: Panel test results in y-direction: thermal conductivity vs. bulk density (alumina fibre mats)

thermal conductivity of the alumino-silicate fibre mats (fig. 5) is found to decrease monotonously when the bulk density in rised. This effect obviously is enhanced at the higher temperatures which is due to a respective variation of the radiation effect dominating the heat transfer under the given conditions. This tendency has been confirmed by measurements with the various methods and also for the x- and z-directions. At the other end, the decrease of conductivity with rising density gets smaller for the lower temperatures and it becomes negligible when room temperatures is approached (not shown in fig. 5). The same holds for the alumina fibre mats (fig. 6) where a strong decrease is observed in the lower density range (55 to 90 kg m^{-3}) followed by a minimum around 95 kg m^{-3} and a slight increase after that. The results show a scattering of about ±8%. This behavior of fibre materials has to be taken into consideration when measurements are compared.

6.3 Comparison of the results from different methods

Figs. 7 and 8 show alumino-silicate fibre mat results at two selected densities (103 kg m^{-3} and 170 kg m^{-3} representing the lower and upper end of the range investigated) measured by various methods and evaluated by interpolation from the effective thermal conductivity vs. bulk density curves (fig. 5). Comparison with the hot-wire measurements are restricted to temperatures below 800 °C as mentioned above.

For the smallest investigated bulk density (ρ = 103 kg m^{-3}, fig. 7) the various results essentially can be summarised as follows:

- All results evaluated from the various plate facility measurements in y-direction from room temperature up to 1200°C can be expressed by a unique function. The results of RA1 in this direction are found to be in a very good agreement.
- The curve for plate measurements in x- and z-direction, i.e. along both of the remaining directions of the fibre mat, is found 30 to 40 % above, depending on temperature. The agreement with results from the hot-wire measurements obtained in xz-direction, e.g., with the wire in y-direction is very good. The difference between the curves for y-direction on one side and xz-direction on the other side is thought to be due to the radiation transport which is favoured in xz-direction due to the larger extension of the fibre mat pores in this direction when compared to the those orthogonal to the main fibre orientation.

- One curve is lying between these limitations as obtained for the pure y- or xz-direction respectively. These results come from the hot-wire measurements with the wire

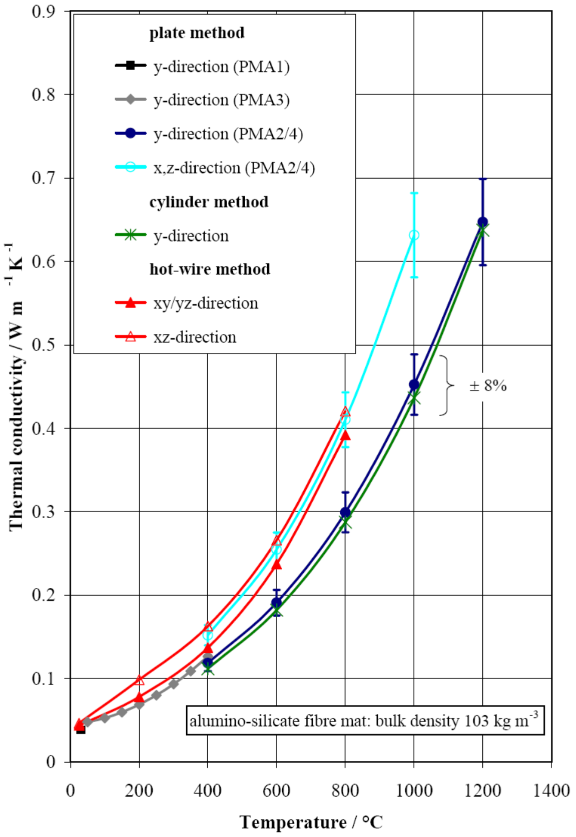

Fig. 7: Comparison of measurement results: alumino-silicate fibre mats at bulk density 103 kg m^{-3}

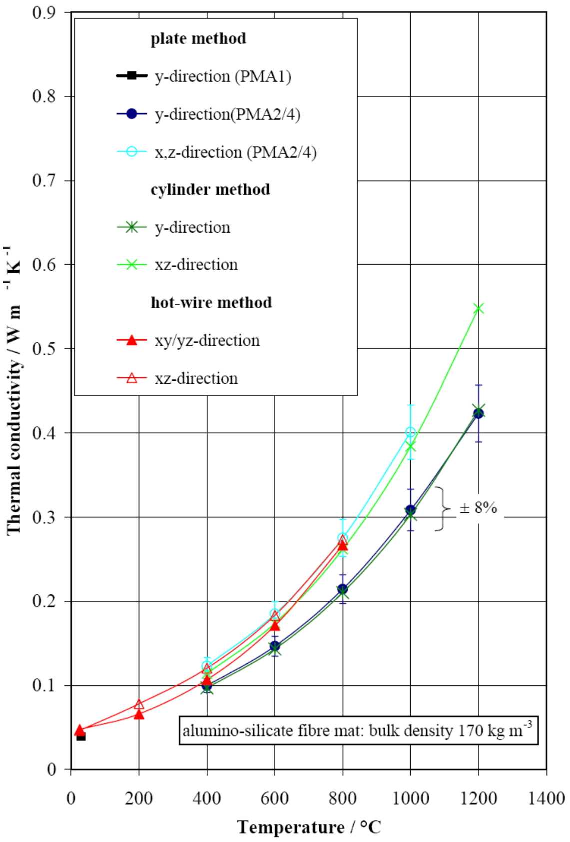

Fig. 8: Comparison of measurement results: alumino-silicate fibre mats at bulk density 170 kg m^{-3}

embedded between two fibre mats leading to some average thermal conductivity in the xy- and yz-directions respectively. These results are found to be far above the effective thermal conductivity in the normal direction of application as measured by the plate facilities.

Almost the same characteristic has been found for the larger bulk density (ρ = 170 kg m^{-3}, fig. 8) where the difference between both of the curves representing y- and xz- directions is somewhat smaller (25 to 35 %). All these results show a very strong non isotropy of the alumino-silicate fibre mat effective thermal conductivities with big differences between y- and xz-directions. The thermal conductivities from hot-wire measurements are clearly above those for the normal direction of application of fibre mat insulations, and exactly the same characteristics have been obtained for the alumina fibre mats, too.

7 Conclusions

The effective thermal conductivity of both isotropic and highly non isotropic materials has been measured in wide temperature ranges by various methods including steady-state plate and cylindrical facilities and also a transient hot-wire instrument. The isotropic materials brought excellent agreement of all the results and the transient hot-wire method proved to be most advantageous due its high reliability in connection with low efforts of time and cost. The fibre-mat results demonstrate a strong non isotropy with big differences between the normal direction of a fibre-mat insulation and directions orthogonal to it. Good agreement has been obtained for results from various measuring facilities where the heat flow is inline with one of the main directions, i.e. plate and cylinder facilities with the rolled samples in the latter case. The hot-wire results are analysed to be a kind of superposition of the thermal conductivities in the respective directions orthogonal to the wire. As a consequence the steady-state panel test procedure is superior to the hot-wire method in case of non isotropic materials despite of the long duration of those measurements and the related costs due to high reliability in the entire temperature range investigated and also the agreement between the directions of measurement and practical application of insulations. The hot-wire method should be kept out of consideration for effective thermal conductivity measurements of non isotropic materials like fibre mats where this method completely fails in ranges of low extinction coefficient, i.e. low bulk densities.

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