Int J Thermophys (2007) 28:1679–1692
DOI 10.1007/s10765-007-0278-8

Intercomparison of Insulation Thermal Conductivities Measured by Various Methods

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Published online: 16 October 2007

Abstract In the present contribution, systematic effective thermal-conductivity measurements with different methods are reported for various materials. One of the materials studied, calcium silicate, is isotropic; the other two, alumino-silicate and alumina fiber mats, are non-isotropic. The measurements were carried out with two different steady-state panel test apparatus (according to ASTM C201, designed and constructed by authors), two guarded hot-plate apparatus (ISO 8302, with either one or two samples), one steady-state radial heat flow apparatus (designed by authors), and one transient hot-wire instrument (DIN EN 993-14). These apparatus are operated at ambient pressure and atmosphere (air) between 20 and 1,650°C, and are briefly described in the article. The results show the well-known increase of effective conductivity with temperature, mainly due to radiation heat transfer. For the case of the isotropic calcium silicate material (bulk density of $220\,\mathrm{kg\,m^{-3}}$), no significant differences between the various methods have been found and the results can easily be correlated within ±10%. The fiber-mat results, however, show additional effects of the density (between 103 and $170\,\mathrm{kg\,m^{-3}}$) and the fiber orientation. Large differences exceeding 30% are found between plate and hot-wire results.

Keywords Calcium silicate · Experimental technique comparisons · Fiber mats · Steady-state methods · Thermal conductivity · Transient hot-wire technique

Paper presented at the Seventeenth European Conference on Thermophysical Properties, September 5–8, 2005, Bratislava, Slovak Republic.

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1 Introduction

The effective thermal conductivity of high-temperature insulations is one of the decisive selection criteria for furnace construction. Insulation materials consist of a porous structure, providing an adequate mechanical stability with open or closed small scaled pores filled with gas. Temperature resistivity is imperative for the solid phase when shaped as a foam, fiber mat, or powder. Accordingly, the solid path may be a continuous one or not with respective contact resistances in the latter case. Open pores normally contain the ambient atmosphere (mostly air), whereas the gas inside closed pores can be optimized for achievement of both a low gas thermal conductivity, and subsequently a low effective thermal conductivity of the insulation. For the measurement of the latter, various methods are available with different expenses in time and cost. It is well-known from the literature that different measuring methods occasionally give different results for the same material. Comparison between various insulations is made more difficult, and uncertainties arise for practical application of measured data as given in producers' catalogs, which typically do not include information about the underlying measuring principle. In the present contribution, systematic experimental investigations of commercial insulations are reported for temperatures between 20 and 1,650°C at ambient pressure covering isotropic and non-isotropic materials, various measuring instruments, along with the resulting intercomparisons.

2 Mechanisms of Heat Transfer in Porous Media

Conduction heat transfer in porous media is governed by porosity, the thermal conductivities of gas and solid, the kind of solid structure (i.e., continuous or not, fiber, or grain-shaped), pore size and its distribution, and finally the gas pressure. The conductive transport is superposed by radiation processes, which may be dominant, or they may vanish, which depends on the temperature and overall porosity. Convection effects are usually negligible if the pores are small ($d < 5\,\text{mm}$, following Häussler and Schlegel [1] corresponding to large enough bulk densities ρ in the case of fiber insulations ($\rho > 20\,\text{kg}\cdot\text{m}^{-3}$, see [2])). 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, and others. Prediction of the effective thermal conductivity without any supporting measurements 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 given boundary conditions. Such methods may be classified with respect to the underlying principle (steady state versus transient, absolute versus relative), sample geometry (plane, cylindrical, spherical), temperature, and thermal conductivity ranges, etc. A review of recommended measurement techniques and practices is given, e.g., in [3].

One of the more popular test methods is the transient hot-wire method according to DIN EN 993-14/15, ASTM C1113, and ISO/DIS 8894 standards characterized by a simple arrangement and short duration of the measurements. However, there are serious problems for cases of non-isotropic and low bulk density materials. Application of further transient methods such as hot-strip and hot-disk sensors is used less often.

Steady-state methods are most often 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, which includes 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 based on DIN 52613 and DIN EN ISO 8497 standards. Steady-state methods usually require more effort with respect to the experimental setup and are time consuming. On the other hand, these methods are characterized by high accuracy allowing measurements on non-isotropic materials due to their well defined one-dimensional heat flow on contrary to the transient hot-wire principle. The equation for the evaluation of steady-state measurements is very simple, giving however average thermal conductivities within a respective temperature range. This fact calls for additional evaluation procedures following, e.g., [4,5] for obtaining the so-called 'true' thermal conductivity at a specified temperature. The necessity of such corrections depends on the curvature of the thermal conductivity versus 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 comparisons among the various methods.

Koltermann [6] 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. [7] reported interlaboratory experiments with lightweight firebricks at temperatures up to 1,000°C where hot-wire results exceeded panel-test data by up to 15%. Davis and Downs [8] and Hagemann and Peters [9] reported similar findings, which have been attributed to a certain non-isotropy of the samples which leads to some kind of a mean value for the hot-wire experiments. Dietrichs [10] confirmed this behavior for vacuum formed fiberboards where the difference amounts to about 20%.

Aksel'rod and Vischnewskii [11] report good agreement between hot-wire and steady-state cylinder results for ceramic fiber mats and powder insulations, whereas hot-wire data exceed those from a panel-test apparatus for the case of refractories. A similar experience comes from Schlegel (Personal communication 1988) who found this behavior for calcium silicate where the differences range between 6% and 33%, which have been attributed to the much larger temperature differences for the case of the steady-state panel method. On the other hand, Neumann and Hemminger [12] found good agreement between these methods applied to a porous insulation material (trade name Microtherm), and they pointed out the importance and difficulty of problems due to non-isotropy.

More recently Andersen and Mikkelsen [13] reported hot-disk measurements of various insulations comparing their results with panel test data. Differences between the temperature fields have been discussed, which are one dimensional (cartesian) for the panel and one dimensional (cylindrical) for the hot wire. For the case of the hot disk, it is characterized by some kind of superposition resulting in 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 believed 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. Therefore, the application of the panel-test method has to be preferred since one-dimensional heat flow in this method corresponds to insulation practice.

Litovsky et al. [14] found the thermal conductivity of alumina fiber mats directly measured by a panel-test apparatus to significantly exceed those, which have been evaluated from thermal-diffusivity measurements with a method emploting symmetrical monotonic heating at a constant rate. This is believed 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 fiber mats by application of complex radiation transport models for heterogeneous media with strong multiple scattering. For both methods, the respective radiation contributions have been subtracted from the measured results to obtain the purely conductive transfer, and the agreement has found to be good.

Some initial conclusions from the literature review are as follows:

- the occurrence of differences between effective thermal conductivities measured with various methods is widely known,
- this is especially true for non-isotropic materials,
- the interpretations of the respective phenomena are inconsistent and no systematic studies have been reported up to now.

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

4 Measurement Apparatus

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

The panel test apparatus PMA1 has been constructed from commercially available components, and it is applicable for thermal-conductivity measurements of plate-shaped poorly conducting materials at temperatures close to ambient. The effective thermal conductivity is evaluated from the measured electrical heating power and the temperature difference across one single plate.

PMA3 is a commercially guarded-hot-plate apparatus (Anter Corp., Pittsburgh, PA) 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 of known geometry.

Table 1 Apparatus for effective thermal-conductivity measurements of insulating materials available at IWTT of TU Bergakademie Freiberg

	Method	Sample dimensions (mm)	Temperature (°C)	Atmosphere
PMA1	Guarded hot plate (steady state)	250 × 250 × 70 (max)	20–80	Air
PMA2	Panel test (steady state)	300 × 300 × 120 (max)	300–1,450	Air
PMA3	Guarded hot plate (steady state)	2 × (300 × 300 × 70 (max))	20–400	Air
PMA4	Panel test (steady state)	400 × 400 × 110 (max)	300–1,650	Air
RA1	Radial heat flow (steady state)	60/12 (d_a/d_i) × 180	400–1,450	Inert gas
HW1	Hot wire (transient)	2 × (120 × 125 × 65)	20–1,200	Air

There are two additional apparatus with plate-shaped samples, PMA2 and PMA4 [5,15], which have been designed and constructed at IWTT. Both of them are operated using 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 (see Table 1).

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

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 specimens of calcium silicate characterized by different material properties (Table 2) have been investigated. Preliminary results for the first specimen have been published earlier, see, e.g., [16].

Figure 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, see Fig. 3). As expected for this kind of material, isotropic behavior 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.

Table 2 Characterization of the calcium silicate materials under investigation

	Material 1	Material 2
Composition: CaO/SiO_2	41%/42%	45%/47.5%
Max. service temperature	900°C	1,100°C
Bulk density	$220\,kg \cdot m^{-3}$	$280\,kg \cdot m^{-3}$
Pore radius: Mean value/range	0.3 μm/0.15–0.35 μm	0.2 μm/0.1–0.3 μm
Phase analysis	Xonothlite	Xonothlite

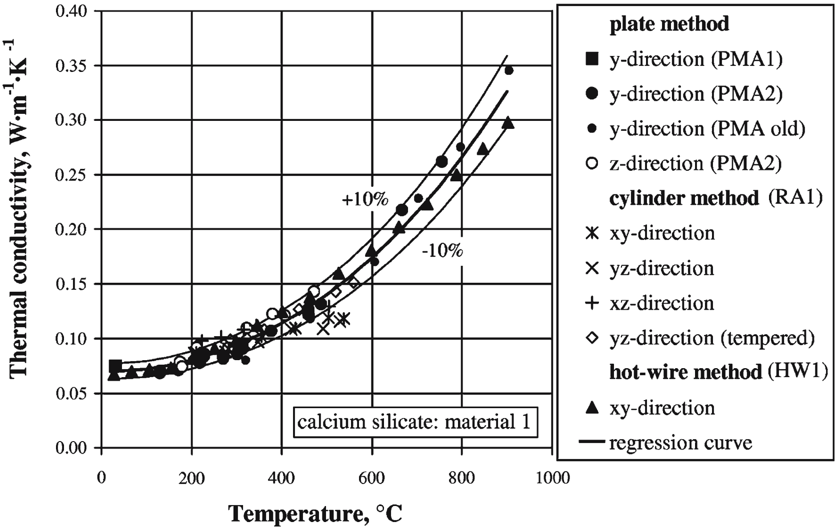

Fig. 1 Thermal conductivity of calcium silicate (material 1)

This behavior is attributed to physical transformations inside the sample materials at elevated temperatures. During the first heating of calcium silicate, water vapor escapes from the sample, see [17], and cellulose fibers embedded along the production process are oxidized leaving gas-filled pores, which modify the effective thermal conductivity. Such effects happen at about 400°C for this material. Therefore, all measurements were initiated at the intended maximum temperatures. However, it has been found after the first test series that the temperature gradient in the sample of RA1 was so large that the outer regions of the sample never achieved 400°C. Therefore, the samples have been tempered at 500°C prior to the measurements and after doing this, the RA1 results are also found within the ±10% range (Fig. 1).

Material 2 has also been tempered at 500°C, and the thermal-conductivity results confirm again the isotropic behavior (Fig. 2). The reproducibility has been tested by two PMA2 measurement series with different samples from the same material, and the results are found within ±10% limits where the variations are attributed to the observed large 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

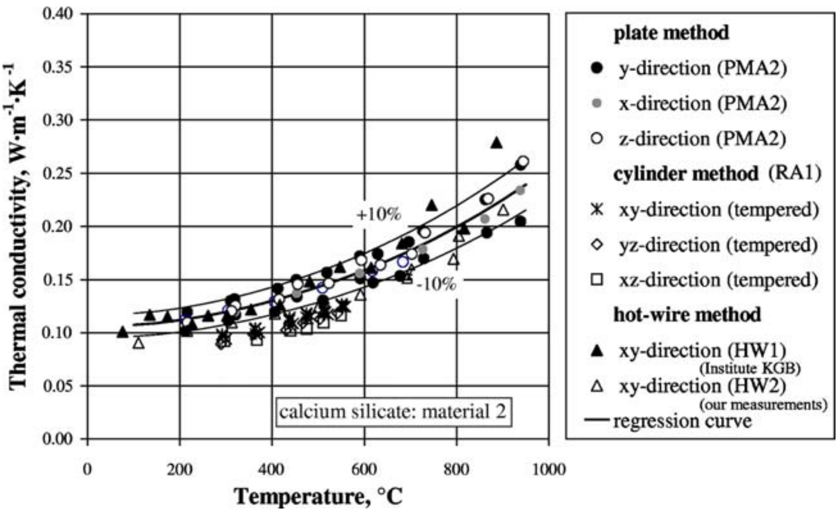

Fig. 2 Thermal conductivity of calcium silicate (material 2)

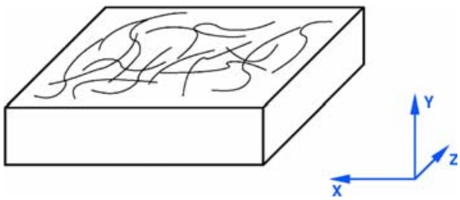

Fig. 3 Definition of the x-, y-, and z-directions (fiber mats)

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 have not been completed in the course of preconditioning material 2 at 500°C.

6 Results for Non-isotropic Fiber Mats of Alumina and Alumino-silicate

Fiber mats are characterized by strong non-isotropy with the preferred fiber orientation (Fig. 3) in the primary plane of a fiber mat (called the xz-plane, see Fig. 4). This is due to the production process with the orthogonal (y) direction showing low thermal conductivity when the insulation is applied.

The panel-test apparatus allows one-dimensional measurements in the normal direction (y), but also orthogonal to this (x and z) after cutting the fiber mats into strips, which subsequently have been re-arranged. In the RA1 arrangement, the samples can be rolled in a spiralic manner (heat flow in y-direction), or they can be cut leading to a combined xz-arrangement with radial heat flow. The latter situation is also found for the hot-wire measurements where the xy-, yz-, and xz-directions have been investigated.

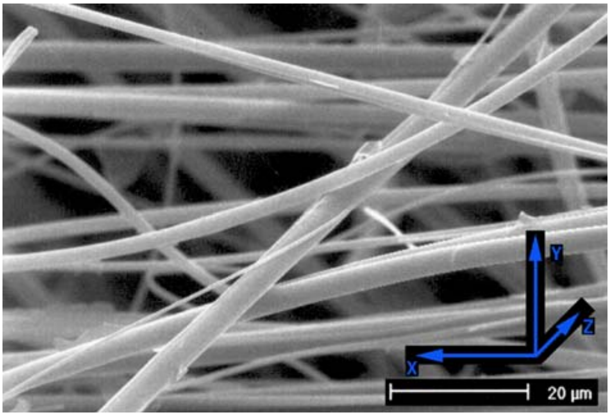

Fig. 4 Picture of an alumino-silicate fiber mat (scanning electron microscope)

Table 3 Characterization of the ceramic fiber mats under investigation

	Material 3	Material 4
Composition: Al_2O_3/SiO_2	Alumino-silicate 52%/48%	Alumina 72%/28%
Max. service temperature	1,400°C	1,650°C
Bulk density	$95–170\,kg \cdot m^{-3}$	$55–120\,kg \cdot m^{-3}$
Mean fiber diameter	$2.5\,\mu m$	$4\,\mu m$

Two different ceramic fiber mat materials have been selected (Table 3), both cover wide ranges of their bulk density, and the first results have been reported by Gross et al. [18] and Wulf et al. [19].

6.1 General Remarks

The fiber mat measurements revealed some additional problems which will be discussed as follows:

– Measurements with the various test apparatus at a certain fixed bulk density proved to be extremely difficult. Sample densities varied within the same charge as delivered from the producer, and they diverged from respective catalog values. Due to its great influence on the effective thermal conductivity, the bulk density has been varied over wide ranges leading to numerous measurements with the various test apparatus at respective constant temperatures. The resulting thermal conductivity versus density dependences allows comparisons at selected densities.
– For the alumino-silicate fiber mats the bulk density has been found to increase during the experiment by typically 15%, and up to 30% in extreme cases. This is attributed to recrystallization processes which depend on temperature and duration of exposure; see [20]. Crystallization effects have been found by X-ray analysis, whereas scanning electron microscopy shows no indication of any modification of the fiber 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 fiber mats, material 4 (alumina) did not show any density variations due to high-temperature exposure.

- All hot-wire measurements show strong scatter for temperatures above 800°C, and evaluation of data proved to be impossible. Phase transitions are considered as one possible reason for this. Besides this, hot-wire measurements are strongly disturbed by radiation effects in highly porous materials, see [21], an effect which becomes more serious at higher temperatures.
- RA1 sample preparation proved to be very difficult due to the small dimensions and the compressibility of the fiber mats showing inhomogeneities and corresponding thermal conductivity variations, which are apparent, especially for small bulk densities.

6.2 Bulk Density Effects

Figures 5 and 6 show effective thermal conductivity (y-direction) versus bulk density plots for materials 3 and 4, respectively, at selected temperatures between 400 and 1,200°C.

The thermal conductivity of the alumino-silicate fiber mats (Fig. 5) is found to decrease monotonically with an increase in the bulk density. This effect obviously is enhanced at higher temperatures, which is due to the 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. The decrease of thermal conductivity with increasing density becomes smaller at lower temperatures and negligible at room temperature (not shown in Fig. 5). The same holds for the alumina fiber mats (Fig. 6) where a strong decrease is observed in the lower density range (55–90 $\text{kg} \cdot \text{m}^{-3}$) followed by a minimum around 95 $\text{kg} \cdot \text{m}^{-3}$ and a slight increase after that. The results show a scatter of about $\pm 8\%$. This behavior of fiber materials has to be taken into consideration when measurements are compared.

6.3 Comparison of Results from Different Methods

Figures 7 and 8 show alumino-silicate fiber mat results at two selected densities (103 and 170 $\text{kg} \cdot \text{m}^{-3}$ representing the lower and upper ends of the investigated range) measured by various methods and evaluated by interpolation from the effective thermal conductivity versus bulk density curves (Fig. 5).

Comparisons with the hot-wire measurements are restricted to temperatures below 800°C as mentioned above.

For the smallest investigated bulk density ($\rho = 103\,\text{kg} \cdot \text{m}^{-3}$, Fig. 7), the various results can be summarized as follows:

- All results evaluated from the various plate apparatus measurements in the y-direction from room temperature up to 1,200°C can be expressed by a unique function. The results of RA1 in this direction are found to be in very good agreement.

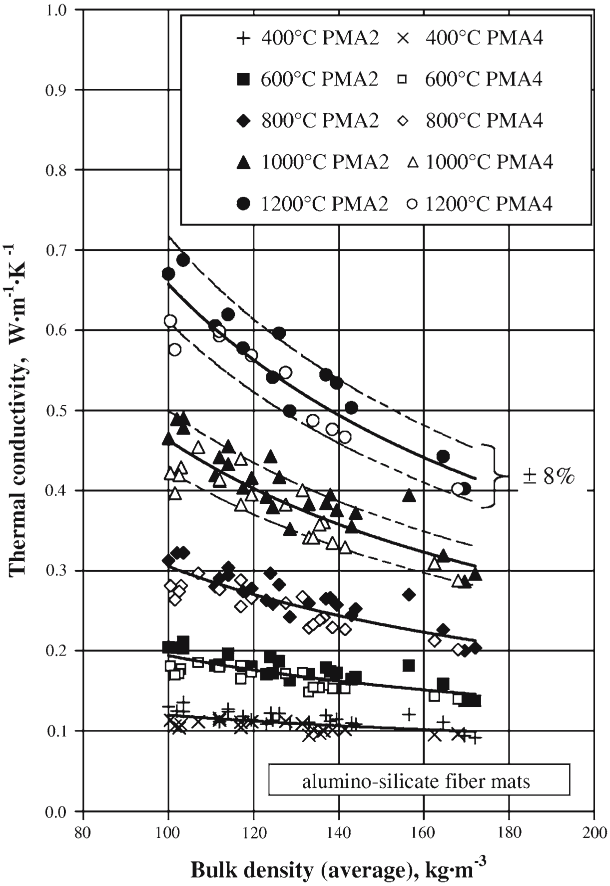

Fig. 5 Panel test results in y-direction: thermal conductivity versus bulk density (alumino-silicate fiber mats)

– The curve for plate measurements in the x- and z-directions, i.e., along both of the remaining directions of the fiber mat, is found to be 30–40% higher, depending on the temperature. The agreement with results from the hot-wire measurements obtained in the xz-direction, e.g., with the wire in the y-direction is very good. The difference between the curves for the y-direction on one side and the xz-direction on the other side is believed to be due to radiation transport, which is favored in the xz-direction due to the larger extension of the fiber mat pores in this direction when compared to those orthogonal to the main fiber orientation.

– One curve lies between these limits as obtained for the pure y- or xz-directions. These results come from the hot-wire measurements with the wire embedded between two fiber mats leading to some average thermal conductivity in the xy- and yz-directions. These results are found to be significantly higher than the effective thermal conductivity in the normal direction of application as measured by the plate apparatus.

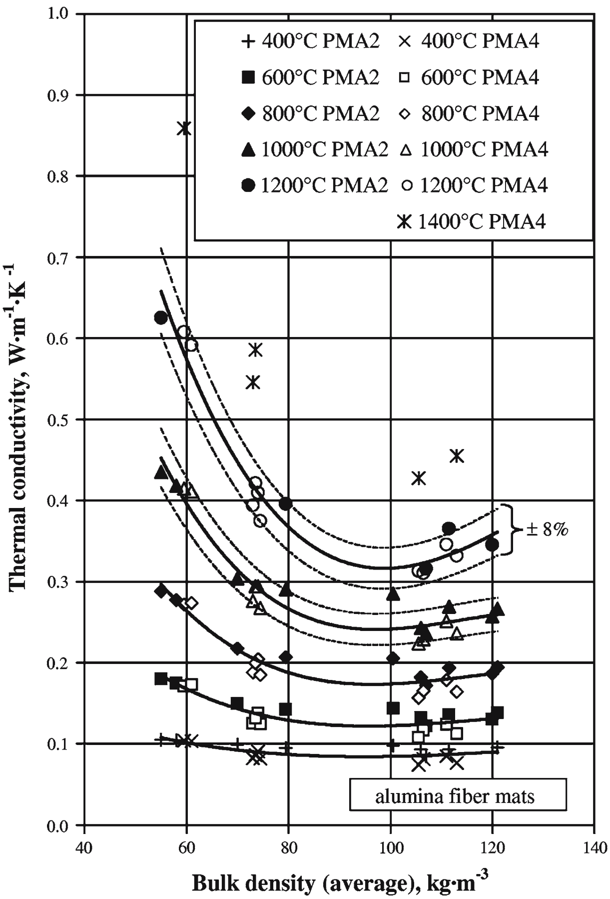

Fig. 6 Panel test results in *y*-direction: thermal conductivity versus bulk density (alumina fiber mats)

Almost the same characteristic has been found for the larger bulk density ($\rho = 170\,\mathrm{kg \cdot m^{-3}}$, Fig. 8), where the difference between both of the curves representing *y*- and *xz*-directions is somewhat smaller (25–35%). All these results show a very strong non-isotropy of the alumino-silicate fiber mat effective thermal conductivities with large differences exhibited between the *y*- and *xz*-directions. The thermal conductivities from hot-wire measurements are clearly higher than those for the normal direction of application of fiber mat insulations, and exactly the same characteristics have also been obtained for the alumina fiber mats.

7 Conclusions

The effective thermal conductivity of both isotropic and highly non-isotropic materials has been measured over wide temperature ranges by various methods including steady-state plate and cylindrical apparatus and also a transient hot-wire instrument.

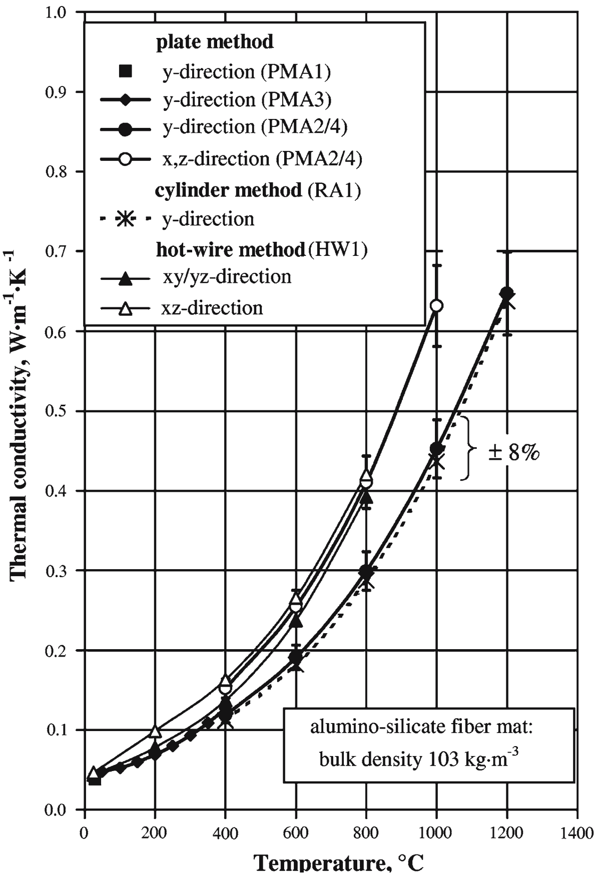

Fig. 7 Comparison of measurement results: alumino-silicate fiber mats at a bulk density of $103\,\mathrm{kg \cdot m^{-3}}$

The isotropic materials showed excellent agreement among all the results, and the transient hot-wire method proved to be most advantageous due to its high reliability in connection with small efforts of time and cost. The fiber-mat results demonstrate a strong non-isotropy with large differences between the normal direction of a fiber-mat insulation and directions orthogonal to it. Good agreement has been obtained for results from various measuring apparatus where the heat flow is in line with one of the main directions, i.e., plate and cylinder apparatus with the rolled samples in the latter case. The hot-wire results are analyzed 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 for the case of non-isotropic materials, despite the long duration of these measurements and the related costs, due to the high reliability over the entire temperature range investigated, and also the agreement between the directions of measurement and practical applications of insulations. The hot-wire method should not be used for effective thermal-conductivity measurements of non-isotropic materials like fiber mats where

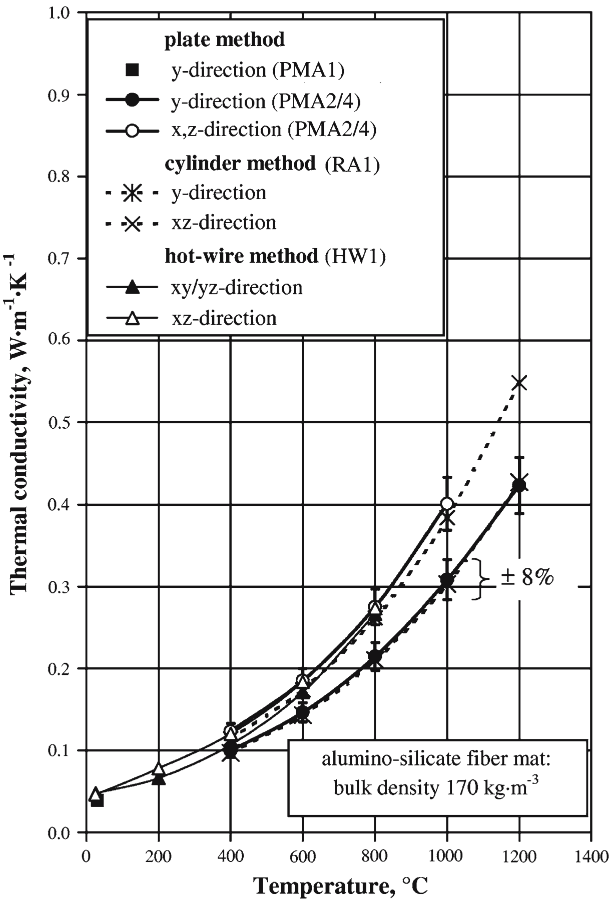

Fig. 8 Comparison of measurement results: alumino-silicate fiber mats at a bulk density of $170\,\mathrm{kg \cdot m^{-3}}$

this method completely fails in ranges of low extinction coefficients, i.e., low bulk densities.

Acknowledgment The support of this investigation by the Deutsche Forschungsgemeinschaft (DFG) is greatly appreciated.

References

1. K. Häussler, E. Schlegel, Freiberger Forschungshefte **A834**, 10 (1995)
2. C. Stark, Dissertation, Universität Würzburg Germany, 1991
3. K.D. Maglic, A. Cezairliyan, V.E. Peletsky, *Compendium of Thermophysical Property Measurement Methods – Part 1: Survey of Measurement Technique*. (Plenum Press, New York, 1984)
4. W. Bolte, Brennstoff-Wärme-Kraft **9**, 373 (1957)
5. G. Barth, U. Gross, R. Wulf, in *Proceedings of the 17th European Conference on Thermo-physical Properties*, Bratislava, Slovak Republic, 2005 (to be published in *Int. J. Thermophys.*) doi: 10.1007/s10765-007-0272-1

6. M. Koltermann, Tonindustriezeitung **85**, 399 (1961)
7. A. Eschner, B. Grosskopf, P. Jeschke, Tonindustriezeitung **98**, 212 (1974)
8. W.R. Davis, A. Downs, Trans. J. Br. Ceram. Soc. **79**, 44 (1980)
9. L. Hagemann, E. Peters, Interceram **31**, 131 (1982)
10. P. Dietrichs, Silikattechnik **38**, 349 (1987)
11. E.I. Aksel'rod, I.I. Vishnewskii, Ogneupory **4**, 49 (1984)
12. W. Neumann, W. Hemminger, cfi/Ber. DKG **65**, 480 (1988)
13. F.B. Andersen, J. Mikkelsen, in *Light Metals 2000*, ed. by R.D. Peterson (TMS, 2000), pp. 429–435
14. E. Litovsky, J.I. Kleiman, N. Menn, High Temp. High Press. **35/36**, 101 (2003)
15. G. Barth, U. Gross, W. Staudte, H. Gründler, Proc. Eurotherm Seminar **44**, 183, ISBN 972-9486-03-04 (1995)
16. U. Gross, G. Barth, R. Wulf, L.T.S. Tran, Freiberger Forschungshefte **A851**, 96 (1999)
17. E. Schlegel, Freiberger Forschungshefte **A789**, 8 (1989)
18. U. Gross, G. Barth, R. Wulf, L.T.S. Tran, High Temp. High Press. **33**, 141 (2001)
19. R. Wulf, U. Gross, G. Barth, Keramische Zeitschrift **56**, 554 (2004)
20. P. Dietrichs, W. Krönert, Gas-Wärme-Int. **30**, 338 (1981)
21. U. Gross, L.T.S. Tran, Int. J. Heat Mass Transfer **47**, 3279 (2004)