Evaluation of measuring thermal conductivity of isotropic and anisotropic thermally insulating materials by transient plane source (Hot Disk) technique

Artem A. Trofimov[1] · Jerald Atchley[2] · Som S. Shrestha[2] · André O. Desjarlais[2] · Hsin Wang[1]

Abstract

The transient plane source (TPS) technique, also referred as the Hot Disk method, has been widely used due to its ability to measure the thermal properties of an extensive range of materials (solids, liquids, and powder). Recently, it has been recognized that typical Hot Disk sensors can influence TPS results of thermally insulating materials and lead to an overestimation of thermal conductivity. Although improvements have been proposed, they have not yet been implemented in the commercial TPS, leaving researchers with non-standardized modifications or options provided by a commercial Hot Disk apparatus. An empirical study of thermally insulating materials such as extruded polystyrene (XPS) and aerogel blanket is conducted in order to address the factors that affect the reliability of thermal conductivity k obtained using the commercial TPS apparatus. Sensor size, input power, duration of the measurements, applied pressure, and, in the case of anisotropic materials, heat capacity are investigated, and the results are compared with those using a Heat Flow Meter apparatus. The effect of sensor size on the k value is ascribed to heat loss through connecting leads and is more pronounced in smaller sensors and in materials with lower k values. In the case of XPS and aerogel, the effect becomes minimal for sensors with a radius $r \geq 6.4$ mm. The low input power yields a high scattering of the results and should be avoided. Applied contact pressure and the tested region of the specimen play an important role in experiments with low-density fibrous materials due to the large percentage of heat being transferred by radiation and the heterogeneous nature of the samples, respectively. Additionally, the sensitivity of anisotropic measurements to the value of the material's volumetric heat capacity (ρC_p) is shown, emphasizing the need for the precise determination.

1 Introduction

The development of modern engineering applications, such as heat flow management in the semiconductor industry, insulation for space shuttles, and home insulation using various building materials, is quickly increasing the need for novel thermally insulating materials with a ultra-low thermal conductivity due to demands for energy efficiency and energy conservation. Traditional steady-state methods used to determine thermal transmission properties by means of a Heat Flow Meter apparatus [1] and heat flux measurements and thermal transmission properties by means of a guarded-hot-plate apparatus [2] are considered to be accurate techniques for thermal characterization; however, they take a long time to perform and require large samples. Meanwhile, with the development of new materials, frequently only small specimens can be produced, making them incompatible with the aforementioned techniques because of their size. Alternatively, transient methods such as the laser flash method [3], 3ω method [4], hotwire method [5], and transient plane source (TPS) method [6] can evaluate thermal properties at a much faster rate, and they can be used for samples of even a few millimeters.

✉ Hsin Wang
wangh2@ornl.gov

1   Materials Science and Technology Division, Oak Ridge National Laboratory, One Bethel Valley Road, Oak Ridge, TN 37830, USA

2   Energy and Transportation Science Division, Oak Ridge National Laboratory, One Bethel Valley Road, Oak Ridge, TN 37830, USA

Among these techniques, commercially available TPS, commonly referred as the Hot Disk method [7–10], has been one of the most widely adopted tools in recent years. This method has been used for a broad range of materials including bulk solids and thin films [8, 11–13], powders [14], and liquids [15, 16]. Furthermore, Hot Disk can test isotropic as well as anisotropic samples [6, 17], which is especially important for low-density fibrous or cellulous thermal insulating materials, where fiber alignment introduces differences in thermal conductivity in axial and radial directions [18].

In the case of thermally insulating materials [19–22], it was recently found that commonly used Hot Disk sensors can affect TPS results and sometimes lead to an overestimation of thermal conductivity [23–25]. The finite thermal mass of the sensors was proposed as one of the reasons for the potential error [23, 24], and a number of articles that focused on the effect of Hot Disk sensors using numerical simulations were published [11, 24, 26]. Although a few improvements to the TPS technique have been proposed [12, 23, 26, 27], these improvements are either only theoretically established or have not been commercially implemented. This situation still leaves most researchers with only the capabilities that a commercial Hot Disk Thermal Constants Analyzer can provide, raising questions about the methodology for the evaluation of thermal properties of isotropic and anisotropic thermally insulating materials. Due to the nature of the Hot Disk method in which the heat source is also a temperature sensor, it generally prefers homogeneous/isotropic materials. When a material is anisotropic, i.e., the thermal conductivity in the X–Y plane is different from the Z direction, the special anisotropic measurement mode must be used. Some "super" insulation materials [18, 28, 29] have anisotropic thermal properties, and therefore, measurements and analyses using the TPS method must be treated with care.

The present work attempts to focus on factors that need to be considered and decisions that need to be made to get accurate data using the Hot Disk apparatus. This is done by means of experimental evaluation and analysis of the effects of various user-defined parameters (sensor size, input power, duration of the measurements, applied pressure, and, in the case of anisotropic material, heat capacity) on the thermal conductivity of commercial thermally insulating materials such as extruded polystyrene (XPS) of different thicknesses and aerogel blanket. The thermal conductivity values of these materials obtained using the Heat Flow Meter method were used to validate the Hot Disk results.

2 Experimental

2.1 Principle of Hot Disk technique

The typical setup of the Hot Disk experiment is to pass a constant current, with an input power P_0 defined by the user, through a Hot Disk sensor and then simultaneously record

the change in resistance. The technique uses a double spiral sensor made from nickel sealed between two thin Kapton or mica sheets. The sensor acts both as a heater and as a temperature sensor, and normally it is sandwiched between two pieces of identical samples to be tested. The behavior of a Hot Disk sensor during the transient period can be expressed using time-dependent resistance $R(t)$ [7]:

$$\begin{cases} R(t) = R_0[1 + a\Delta T(\tau)] \\ \tau = (t/\theta)^{1/2} \\ \theta = r^2/\alpha \end{cases} \quad (1)$$

where R_0 is a resistance of a Hot Disk sensor before the transient recording, a is the temperature coefficient of resistance (TCR), $\Delta T(\tau)$ is the time-dependent temperature increase of the Hot Disk sensor, t is the time that has passed since the beginning of the transient, θ is the characteristic time, r is the radius of the Hot Disk sensor, and α is the thermal diffusivity of the tested material.

By recording the change in resistance of the sensor, its temperature increase as a function of time is obtained, which is related to the thermal properties of the surrounding material [30]:

$$\Delta T(\tau) = P_0(\pi^{3/2}rk)^{-1}D(\tau), \quad (2)$$

where k is the thermal conductivity of the tested material and $D(\tau)$ is a dimensionless time function that takes into account the conducting pattern of the disk-shaped sensor, which consists of n number of concentric rings; $D(\tau)$ is defined elsewhere [7, 30]. One of the key assumptions of the TPS theory is that the sensor is placed in the infinite medium, which means that the propagation of generated heat should not reach the external boundaries of the sample during the experiment. This requirement is typically satisfied if the distance from any point of the sensor to any point on the surface of the sample is larger than the parameter of a probing depth $\Delta_p = 2(\alpha t)^{1/2}$.

Based on Eq. (2), the temperature increase of the sensor during the transient period should be linearly proportional to a function $D(\tau)$. Therefore, it must be possible to fit ΔT as a function of $D(\tau)$ with a straight line as long as the relationship between t and τ is known. In other words, characteristic time θ can be used as a fitting parameter, and its value for the best fit is used to calculate the thermal diffusivity α of the sample, as shown in Eq. (1). The slope of the obtained line is $P_0(\pi^{3/2}rk)^{-1}$, which is used to extract a thermal conductivity k.

2.2 Anisotropic materials

Equation (2) describes the situation when the tested material is isotropic, implying that the radial and the axial thermal

conductivities of the sample are the same ($k_{radial} = k_{axial} = k$ or $k_x = k_y = k_z = k$, where the x- and y-axes are in the plane of the sensor). In cases where the specimen is anisotropic and its thermal properties are the same along x- and y-axes but different from those along z-axis ($k_x = k_y \neq k_z$), the temperature increase of a Hot Disk sensor surrounded by this material as a function of time is described as

$$\Delta T(\tau_x) = P_0[\pi^{3/2}r(k_xk_z)^{1/2}]^{-1}D(\tau_x), \tag{3}$$

where k_x and k_z are the thermal conductivities along the x-axis and z-axis, respectively, $\tau_x = (t/\theta_x)^{1/2}$, and $\theta_x = r^2/\alpha_x$. Accordingly, θ_x is used as a fitting parameter to calculate thermal diffusivity α_x along the x-axis. Next, the previously determined volumetric heat capacity (ρC_p) is required to calculate $k_x = \alpha_x \rho C_p$. Finally, k_z is obtained from determined k_x and the slope of the line described in Eq. (3) [6, 17].

2.3 Materials and methods

The thermal properties of the investigated materials were measured by a Hot Disk thermal constant analyzer (Hot Disk Inc., Sweden). A TPS 3500 Hot Disk system with a bridge circuit, a digital voltmeter, and a data analysis module was used. Hot Disk Kapton sensors with radii of 2.001 mm,

3.189 mm, 6.403 mm, 9.719 mm, 9.868 mm, and 14.61 mm were used. The input power and the duration of the measurement were selected such that the requirements of a probing depth and a characteristic time were met. An experimental setup is illustrated in Fig. 1a, b. Measurements were repeated three to five times for each set of parameters. Experiments were performed at room temperature and ambient conditions under stainless steel cover to avoid temperature fluctuations from drafts of air to the sample. The times between the measurements were long enough to avoid any temperature drift, which was measured for 40 s before every test.

The reference mild steel SIS2343, extruded polystyrene (XPS) foam, and aerogel blanket were investigated as representative materials. The steel sample was a cylinder with a 50 mm diameter and a 20 mm thickness and was provided by Hot Disk AB. The XPS samples were purchased in three thicknesses of 20 mm, 10 mm, and 5 mm, and the aerogel blanket had a thickness of 10 mm. XPS and aerogel samples were cut to have 50×50 mm^2 surface area. The steel, XPS, and aerogel samples are shown in Fig. 1c.

Parameters such as sensor size, output power, duration of the experiment, applied pressure, and volumetric heat capacity were tested in either isotropic or anisotropic mode. A contact pressure of 3.6 kPa (unless specified otherwise) was always applied using free weight to guarantee
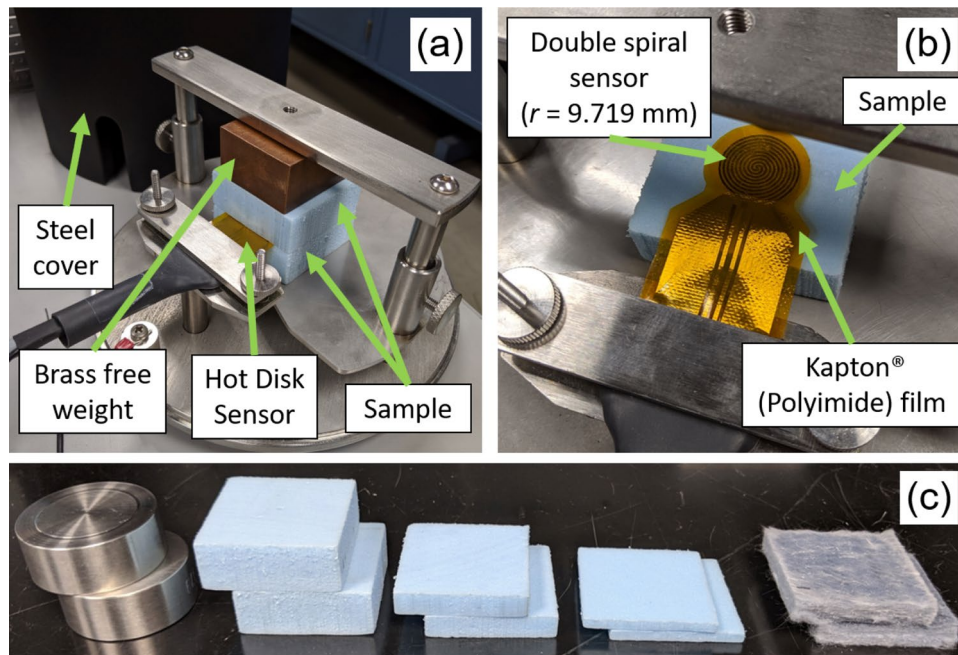
Fig. 1 a Hot disk setup depicting hot disk sensor sandwiched between two 20 mm thick extruded polystyrene (XPS) samples to form a sample-sensor-sample arrangement. Brass free weight is used to ensure a good contact between the sensor and the samples. A stainless steel cover is used to avoid temperature fluctuations caused by drafts of air during the experiment. **b** Double spiral sensor (radius $r = 9.719$ mm) sealed by Kapton on top of the XPS sample. **c** Set of samples used in the investigation (from left to right): 20 mm thick mild steel SIS2343, XPS foams (20, 10, and 5 mm thick), and 10 mm thick aerogel blanket. The steel sample had a diameter of 50 mm; the other samples had a surface area of 50×50 mm^2

reproducibility. When one or more parameters were kept constant during the experiment, its value was listed in the captions to the corresponding figure. Analyses of the experiments were based on Eq. (2) for isotropic mode, and on Eq. (3) for anisotropic mode.

The thermal conductivities of XPS and aerogel were compared with those obtained using a Heat Flow Meter apparatus (HFMA). The HFMA tests were conducted on 30 cm×30 cm samples at thicknesses of 5, 10, and 20 mm for XPS and 10 mm for aerogel. The HFMA tests were conducted according to ASTM C518-17 [1].

The specific heat capacities (C_p) of XPS and aerogel blanket were measured using a TA Instruments Q2000 Differential Scanning Calorimeter (DSC) in the temperature range of − 100 to 100 °C at 20 °C min^{-1}. Tzero hermetic aluminum pans and lids were used during the DSC runs. An empty pan with lid served as a reference, and a sapphire was used as a standard. Two samples of XPS (2.60 mg and 4.25 mg) and two samples of aerogel (7.87 mg and 10.34 mg) were run three times. The heating and cooling were performed in air, and upon completion of the heating cycle, the samples were held at a maximum temperature for 20 min before the cooling cycle was started.

3 Results and discussion

3.1 Isotropic measurements

The thermal conductivity of a reference material (mild steel SIS2343) was measured at room temperature and in isotropic mode using sensors with different radii to confirm the reliability of the Hot Disk thermal constants analyzer and to obtain a baseline for a measurement's variation as a function of the sensor size. As shown in Fig. 2, the thermal conductivity of the steel sample averaged over the investigated sensor sizes was $k_{Steel} = 13.66 \pm 0.15$ W mK^{-1} (1.2% variation), which was in a good agreement with the value of 13.61 ± 0.018 W mK^{-1} (0.13%) that was provided by Hot Disk AB using a sensor with the radius of $r = 6.4$ mm.

Figure 2 also compares the results for three different thicknesses of XPS with the XPS thermal conductivity k_{XPS} obtained for the same three thicknesses using Heat Flow Meter apparatus. The 5 mm thick XPS was only measured using the three smallest Hot Disk sensors because the use of larger sensors would lead to violation of the model assumption of infinite material (i.e., the available probing depth was smaller than thermal penetration depth). The situation

Fig. 2 Thermal conductivity k of steel, extruded polystyrene (XPS), and aerogel blanket (thicknesses are indicated) as a function of Hot Disk sensor radius measured in isotropic mode. A 20 mm thick aerogel was made by stacking two 10 mm thick samples. Standard deviations were obtained from the experiments of various durations and input powers. Applied pressure was kept at 3.6 kPa. Dotted horizontal lines indicate the range of thermal conductivity of XPS and aerogel obtained using Heat Flow Meter apparatus
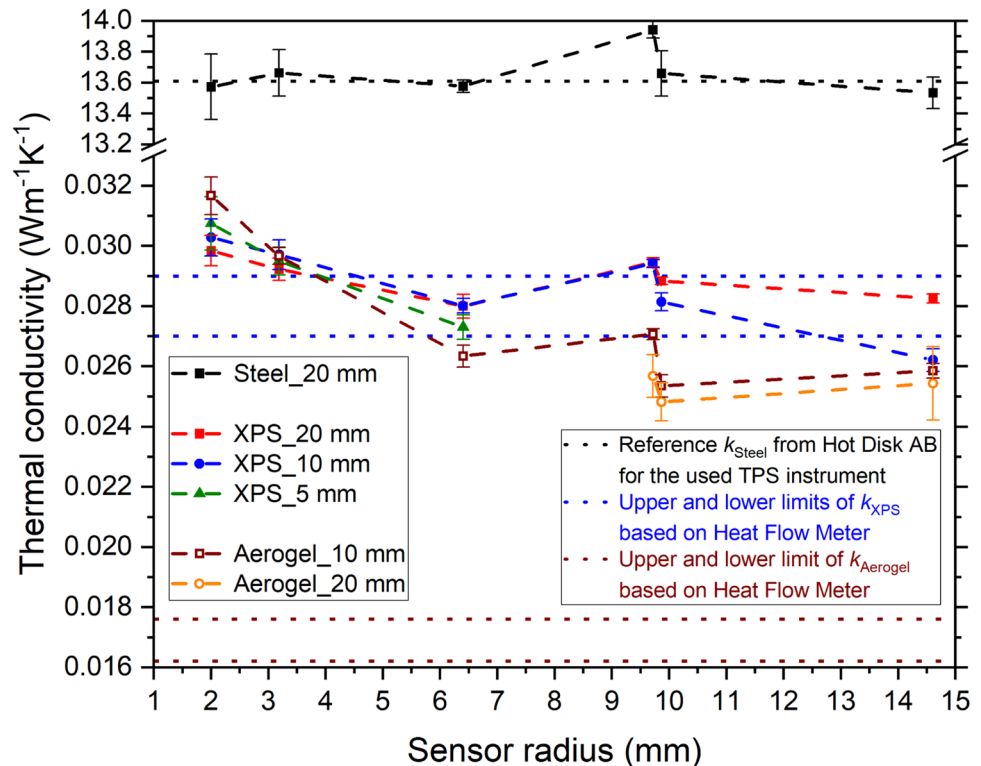
is the same in tests of the 10 mm thick XPS using the largest sensor with a radius of $r = 14.6$ mm. In this case, the propagation of heat reaches the external sample boundaries, leading to the effects of the surrounding air and its thermal conductivity on the transient recording; as a result, the k_{XPS} obtained with this sensor is noticeably lower than the average value and below the value from Heat Flow Meter. Excluding the results for the largest sensor, thermal conductivity stayed constant as a function of thickness and sensor size within 3.5%: $k_{XPS} = 0.0289 \pm 0.001$ W mK^{-1}, which was comparable to the Heat Flow Meter results of 0.028 ± 0.001 W mK^{-1} (3.6%).

Unlike XPS, aerogel appeared to be the most sensitive material to the size of the sensors. As shown in Fig. 2, after an initial decrease of *ca.* 20% for smaller sensors (from 0.0316 W mK^{-1} for $r = 2.0$ mm to 0.0263 W mK^{-1} for $r = 6.4$ mm), the measured $k_{Aerogel}$ stabilized at an average value of 0.0258 ± 0.001 W mK^{-1} (3.9%) for $r = 6.4$–9.8 mm. One possible cause of the decreasing trend in thermal conductivity as a function of sensor size is heat loss through the connecting leads to the double spiral. Since the connecting leads are the same for all sensor sizes, the heat losses through these leads have a greater effect on the results obtained using Hot Disk sensors with a smaller radius than using the sensors with a larger radius. As a result, due to a larger amount of heat loss for the smaller sensors, the experiments yielded apparent thermal conductivity values that were higher for the smaller sensors than for the larger ones. For sufficiently large sensors, the relative influence of heat loss through the connecting wires will be negligible compared with the total heating power in the experiment, and the thermal conductivity will not change with the radius, as shown in Fig. 2 for $r \geq 6.4$ mm.

In Fig. 2, XPS showed a trend similar to that of aerogel for 2.0 mm $\leq r \leq$ 6.4 mm, but the change in magnitude of thermal conductivity was much smaller. Since the "true" thermal conductivity of XPS is higher than that of aerogel (indicated by Heat Flow Meter results), the variation in the magnitude of thermal conductivity with sensor radius becomes less due to a smaller relative amount of heat loss to the connecting wires. It can also be concluded that the effects of heat loss through connecting wires for sufficiently conducting materials cannot be observed. The results for stainless steel indicate this trend.

Even though an aerogel did not show a dependence on thickness within 3%, measured values of thermal conductivity were significantly higher than those obtained with a Heat Flow Meter, which were 0.0169 ± 0.0007 W mK^{-1} (4.1%). Aerogel poses several challenges that can contribute to this observed discrepancy that are discussed further; however, other parameters that have an effect on Hot Disk experiments when testing thermally insulating materials are discussed first.

3.2 Influence of input power and experiment duration

Figure 3 shows the thermal conductivity k of XPS and aerogel as a function of (a) input power and (b) duration of the measurement. To make comparison of these materials easier, the values were normalized to the average conductivity calculated based on the results measured by the Hot Disk technique and shown in Fig. 2. According to Fig. 3a, lower power measurements led to larger fluctuations of k, which was the result of a higher noise level. Since a Hot Disk sensor is also a resistance thermometer, use of low input power implies low current through the sensor, and low current, in
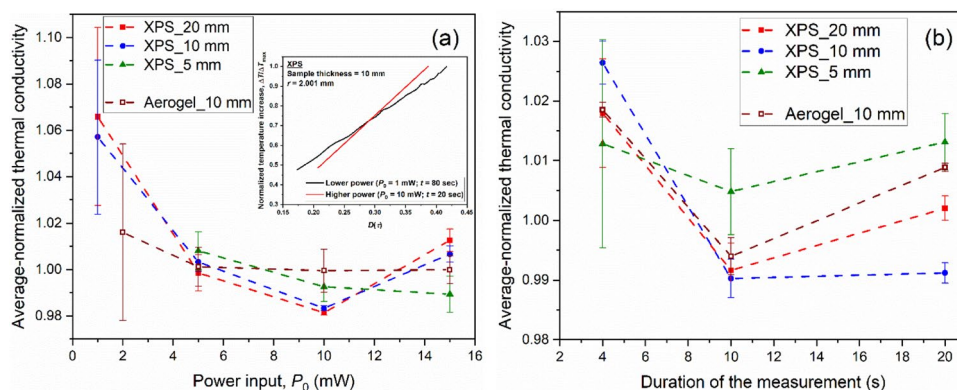
Fig. 3 Normalized thermal conductivity k of extruded polystyrene (XPS) and aerogel as a function of **a** input power and **b** duration of the measurement obtained in isotropic mode. Inset of **a** highlights the difference between the noise levels of the temperature reading when using lower and higher input power. Thermal conductivity was normalized to the average values calculated from Fig. 2. k values obtained at the lowest power (1 or 2 mW) were excluded from calculations of the average values. Standard deviation was based on five repetitions under the same conditions. A sensor with a radius of 6.4 mm was used, and the applied pressure was kept at 3.6 kPa

turn, makes the recording of the change in resistance (temperature) less precise (see inset of Fig. 3a); therefore, caution is needed when selecting the input power. Meanwhile, this parameter did not affect thermal conductivity values above the limit of the low power (1–2 mW in the case of XPS and aerogel), the variation of thermal conductivity of XPS for all three thicknesses from 5 to 15 mW of input power was only ± 2% relative to the average value, which is within the instrument uncertainty of 2–5%, and the fluctuation of the values at each input power was ca. 1% for XPS and less than 0.01% for aerogel. A similar conclusion can be applied to the parameter of experiment duration (Fig. 3b): the materials did not show a dependence on the duration at given power as long as the low power was avoided.

3.3 The effect of applied pressure and position on $k_{Aerogel}$

Although aerogel thermal conductivity was not subject to changes as a function of input power and experiment duration within less than a percent (Fig. 3), an applied pressure necessary for a good contact between samples and a sensor was shown to strongly influence the measured value due to the material's compressibility. Since the relative change in thermal conductivity is considered, determined trends were valid even though the "true" values of thermal conductivity can be different from the measured ones. Figure 4a highlights the variations in the $k_{Aerogel}$ as a function of applied pressure for the three smallest sensors. Larger sensors were not shown since their values were close to the k value obtained with $r = 6.4$ mm within 4%, as shown in Fig. 2. Thermal conductivity rapidly decreased with an increase in applied pressure until reaching a stable value within 3% for pressure of ca. 3–6.5 kPa. This behavior can be understood by noting that the Hot Disk measures apparent thermal conductivity, which implies a heat transfer consisting of solid conduction, gas conduction, and radiation. Insulating low-density fibrous materials, such as that investigated here with aerogel blanket, allow a large percentage of heat transfer by radiation, which is strongly dependent on a material's density [31, 32]. Therefore, an increase of density causes a reduction in radiant heat flow, and simultaneously, leads to an increase of solid conduction. Figure 4a shows that the thermal conductivity of aerogel blanket as a function of applied pressure first decreases and then plateaus. Therefore, it was concluded that, initially (i.e. pressure of ca. 0–3 kPa), the decrease in a radiant transfer is stronger than the increase in a solid conduction; however, at higher applied pressure (ca. 3–6.5 kPa) a plateau-like region appears due to a comparable magnitude of the decrease in radiant transfer and increase in solid conduction.

Another feature that must be considered in evaluation of thermally insulating low-density fibrous materials is their homogeneity. Due to the availability of various sensor sizes for the Hot Disk Apparatus, the instrument allows a local volume of the samples to be probed and tested if the material is heterogeneous. Figure 4b denotes the importance of this aspect, showing variation in $k_{Aerogel}$ with the position on top and bottom sides of aerogel sample using sensor of $r = 2.0$ mm. Variation of the thermal conductivity with the position on the sample does not show a specific trend and could be attributed to the nonuniform fiber distribution and inhomogeneous structure of the sample. However, alternating from one side of the sample to the other while probing the same spot tends to show a smaller apparent k value for the top side (see Fig. 4b and its inset), in which six out nine positions showed a lower thermal conductivity at the top than at the bottom side. Upon visual and physical inspection, the top side was found to be denser, apparently due to the conditions required to produce continuous aerogel blankets during manufacturing. Thus, the observed trend is attributed to a smaller heat transfer by means of radiation through the volume closer to the denser top side when compared with the bottom side.

Fig. 4 **a** Thermal conductivity k of 10 mm thick aerogel blanket measured in isotropic mode as a function of **a** applied pressure (using three different sensor sizes, as indicated by the radius r) and **b** position on the specimen (using the sensor with the $r = 2.0$ mm and a pressure of 3.6 kPa; positions and appearance of the specimen are shown in the inset). Standard deviation was based on 3 repetitions under the same conditions
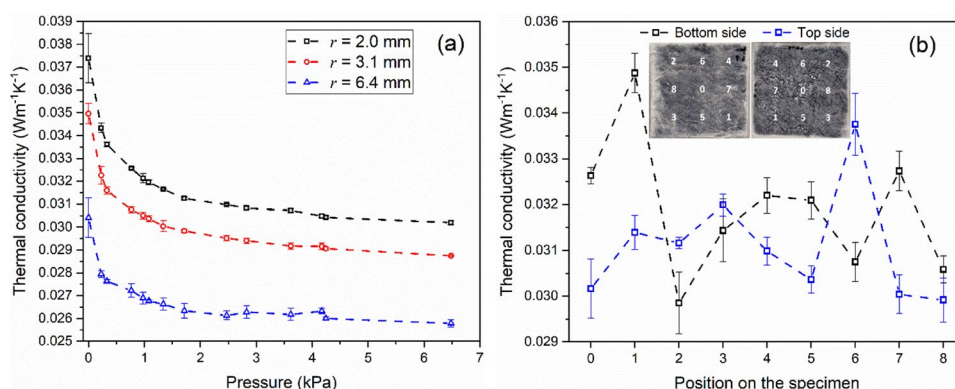
3.4 Anisotropic measurements

Regarding the discrepancy between $k_{Aerogel}$ obtained using the Hot Disk method and Heat Flow Meter noted previously in Fig. 2, the Hot Disk results were obtained at a fixed pressure and fixed samples arrangement using isotropic mode, which assumes direction-independent thermal conductivity. However, if the material is anisotropic, this mode will lead to a volume-averaged k value that is often approximately the mean value between radial and axial thermal conductivities. Hot Disk also allows to perform evaluation of the thermal properties of anisotropic materials in a like manner; however, it requires the same k in x- and y-directions that are responsible for radial thermal conductivity (x- and y-axis are in the plane of a Hot Disk sensor). The technique can be applied when $k_x = k_y \neq k_z$ (or $k_{radial} \neq k_{axial}$) but not if $k_x \neq k_y \neq k_z$. A priori knowledge of volumetric heat capacity ρC_p of the sample is also necessary. If ρC_p is known, one can also use anisotropic mode to check the properties when whether the material is isotropic or not is uncertain. Inversely, if radial and axial k are known, anisotropic measurements can be used to determine volumetric heat capacity.

The steps described in Sect. 2.2 were followed to analyze the experimental data, and the dependence of thermal conductivity on volumetric heat capacity was obtained by manually changing the ρC_p value by 0.01 MJ m^{-3} K^{-1} steps in the described calculations. The correct heat capacity was based on the DSC measurement at room temperature. In such manner, Fig. 5 highlights the sensitivity of anisotropic mode of the Hot Disk technique to the heat capacity and emphasizes the necessity of precise determination of material's heat capacity.

An example is shown in Fig. 5a, where XPS specimens of two thicknesses were measured in anisotropic mode using various values of ρC_p. The inset of Fig. 5a shows the experimentally determined specific heat capacity of XPS measured with a DSC and its corresponding best linear fit. The two XPS samples were run three times each, and the inset shows the average values of all six measurements. According to the linear fit of C_p (T), the specific heat capacity of XPS at 25 °C was $C_p \approx 1511$ J kg^{-1} K^{-1}, which agreed well with typical values of 1450–1500 J kg^{-1} K^{-1} [32–35]. Using an apparent density of investigated samples of $\rho \approx 25.2$ kg m^{-3} and obtained C_p, the volumetric heat capacity was calculated to be $\rho C_p \approx 0.38$ MJm^{-3} K^{-1}. In this situation, $k_{axial} = 0.0279 \pm 0.0007$ W mK^{-1} and $k_{radial} = 0.0281 \pm 0.0004$ W mK^{-1} averaged over the two thicknesses. These values correlate with the results of Hot Disk experiments in isotropic mode (Fig. 2) as well as with the Heat Flow Meter data shown in Fig. 5a, confirming the isotropic or quasi-isotropic nature of the material. Additionally, Fig. 5a highlights the importance of having a precise heat capacity value because, for example, even a small change in ρC_p from 0.38 to 0.35 MJ m^{-3} K^{-1} will lead to the values of $k_{axial} = 0.0303$ W mK^{-1} and $k_{radial} = 0.0259$ W mK^{-1} (average values for two thicknesses), suggesting the material is anisotropic.

In contrast to XPS, axial and radial thermal conductivities of aerogel blanket can be expected to be different. Typically, pristine aerogel must be mixed with the fibers to give it flexibility in order to make an aerogel blanket, such as the one investigated in this work. Consequently, differences in the heat propagation along the fibers relative to the direction perpendicular to the fibers are possible. Figure 5b displays k_{axial} and k_{radial} values of 10 mm thick aerogel as a function of volumetric heat capacity as well as the results of a Heat Flow Meter. The inset in Fig. 5b shows the specific heat capacity of the aerogel blanket measured with a DSC as a
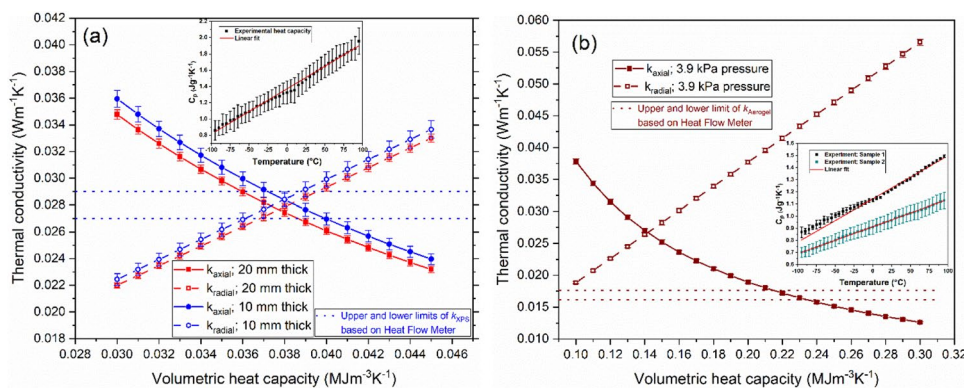
Fig. 5 Thermal conductivity k of **a** extruded polystyrene (XPS) and **b** aerogel blanket (10 mm thick; applied pressure is 3.6 kPa) measured in anisotropic mode as a function of predetermined volumetric heat capacity ρC_p. Dotted horizontal lines indicate the range of thermal conductivity obtained using a Heat Flow Meter apparatus. Standard deviation was based on five repetitions under the same conditions. A

sensor with a radius of 6.4 mm was used. Insets show experimentally determined specific heat capacity curves and corresponding best linear fits. Inset **a** is the average of six measurements (two XPS samples×three repetitions). Inset **b** is the average of three measurements for each aerogel sample

function of temperature. The inset shows two C_p curves corresponding to two aerogel samples, in which each of them was measured three times. Difference in the specific heat capacity of the two samples can be attributed to the different ratios of the components blended in the material. By nature, the aerogel blanket is a composite material made from various forms of silica, fibers, and additives. The distribution of these components within the blanket is unknown, but the heterogeneity of the specimen was earlier noted in Fig. 4b. Sample preparation for the DSC measurements could also have affected the composition since the material easily collapses and crumbles.

According to the linear fit of $C_p(T)$ for both cases, specific heat capacity of the aerogel blanket at 25 °C was determined to be $C_p^1 \approx 1235$ J kg^{-1} K^{-1} and $C_p^2 \approx 971$ J kg^{-1} K^{-1}. Using an apparent density of $\rho \approx 161$ kg m^{-3}, the volumetric heat capacity was calculated to be $\rho C_p^1 \approx 0.20$ MJ m^{-3} K^{-1} and $\rho C_p^2 \approx 0.16$ MJ m^{-3} K^{-1}. According to Fig. 5b, k^1_{axial} 0.0185 ± 0.0002 W mK^{-1} and $k^1_{radial} = 0.0377 \pm 0.0003$ W m K^{-1} in the first case, while $k^2_{axial} = 0.0231 \pm 0.0002$ W mK^{-1} and $k^2_{radial} = 0.0302 \pm 0.0003$ W mK^{-1} in the second case. The axial thermal conductivity of sample 1 is closer to the value from the Heat Flow Meter (0.0169 ± 0.0007 W mK^{-1}), and the measured specific heat capacity is noticeably more repeatable than in the case of sample 2 (see standard deviation in the inset of Fig. 5b). This behavior highlights the need to test multiple samples from different locations for anisotropic and inhomogeneous materials such as aerogel, and in the present situation, it shows that sample 1 more closely resembles the structure and composition of the aerogel blanket specimen used with the Heat Flow Meter apparatus. Therefore, further discussion is focused on the results of sample 1.

The axial thermal conductivity k^1_{axial} of the aerogel blanket is within 10% of the value obtained using a Heat Flow Meter, while the radial component is over two times higher. This situation can be explained by the nature of the measurements taken by the Heat Flow Meter apparatus. The instrument establishes steady-state one-dimensional heat flow through the sample, avoiding transverse heat flow [1]; thus, it only evaluates thermal properties in one specific direction. In the present study, the experiments were performed in the through-thickness direction, which matched the axial direction in the Hot Disk experiments, thus yielding similar results between k_{axial} from Hot Disk and k from Heat Flow Meter, and showing the effectiveness of Hot Disk method in estimation of thermal conductivity in the case of a low-density and anisotropic insulation like aerogel blanket. The fact that k_{radial} was also obtained using the Hot Disk Apparatus allowed the anisotropy ratio to be evaluated as $k_{radial}/k_{axial} \approx 2.2$.

Based on these findings, the following factors should be considered to ensure the reliability of test results.

- Knowing whether the material is anisotropic or isotropic is crucial, as was demonstrated with the aerogel sample; if this is not known, the material can be tested using the Hot Disk method provided its volumetric heat capacity was determined in the separate experiment.
- Tests of multiple samples of anisotropic and inhomogeneous materials such as the aerogel blanket are necessary due to variations in their structure and composition.
- If the material is compressible, as in the case of low-density fibrous materials, the applied pressure used to ensure a good sample-sensor contact needs to be taken into account because higher pressure leads to lower apparent thermal conductivity due to a reduction in radiant heat flow.
- Hot Disk sensors of different sizes will probe different volumes of the sample; therefore, the material's homogeneity should be considered. If the material is inhomogeneous, smaller sensor sizes can be used to test the level of heterogeneity, while the sensors closer to the sample dimensions will evaluate a thermal conductivity of the sample as a whole.
- Caution is needed when choosing the input power: low input power (below 3 mW in the case of XPS and aerogel) should be avoided since it leads to a low signal-to-noise ratio when recording the temperature change, which converts into a large scattering of calculated thermal conductivity values. Above this limit, input power and the duration of the measurements do not affect the resulting thermal conductivity values.

4 Conclusions

In the present investigation, the Hot Disk technique was applied to a set of isotropic and anisotropic thermally insulating materials to measure their thermal properties. The results were then compared with values obtained using a Heat Flow Meter to demonstrate the capability of a systematic method to test small low-thermal-conductivity samples using only the capabilities of the commercially available system. The sensor size was shown to affect the apparent thermal conductivity k of such materials, which was assigned to heat loss through the connecting leads. The effect increased for smaller sensor sizes and for materials with a lower k value; however, both XPS and aerogel blanket showed stable results for sensors with a radius $r \geq 6.4$ mm. Low input power causes the recording of a temperature change to be noisy and less precise. Applied contact pressure and the tested region of the specimen played an important role in TPS experiments with low-density fibrous materials due to a large percentage of heat transfer by radiation and the heterogeneous nature of the samples, respectively. Additionally, the sensitivity of anisotropic measurements to the value of a

material's heat capacity was shown, emphasizing the need for a precise determination of ρC_p.

6. References

1. ASTM C518-17, *Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus* (ASTM International, West Conshohocken, 2017)

2. ASTM C177-19, *Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus* (ASTM International, West Conshohocken, 2019)

3. ASTM E1461-07, *Standard Test Method for Thermal Diffusivity by the Flash Method* (ASTM International, West Conshohocken, 2007)

4. D.G. Cahill, R.O. Pohl, Thermal conductivity of amorphous solids above the plateau. Phys. Rev. B **35**, 4067–4073 (1987)

5. ASTM C113/C1113M-09, *Standard Test Method for Thermal Conductivity of Refractories by Hot Wire (Platinum Resistance Thermometer Technique)* (ASTM International, West Conshohocken, 2019)

6. ISO 22007-2:2015, *Plastics—Determination of Thermal Conductivity and Thermal Diffusivity—Part 2: Transient Plane Heat Source (Hot Disc) Method* (International Organization for Standardization, Geneva, 2015)

7. S.E. Gustafsson, Transient plane source techniques for thermal conductivity and thermal diffusivity measurements of solid materials. Rev. Sci. Instrum. **62**, 797–804 (1991)

8. M. Gustavsson, E. Karawacki, S.E. Gustafsson, Thermal conductivity, thermal diffusivity, and specific heat of thin samples from transient measurements with hot disk sensors. Rev. Sci. Instrum. **65**, 3856–3859 (1994)

9. T. Log, S.E. Gustafsson, Transient plane source (TPS) technique for measuring thermal transport properties of building materials. Fire Mater. **19**, 43–49 (1995)

10. V. Bohac, M.K. Gustavsson, L. Kubicar, S.E. Gustafsson, Parameter estimations for measurements of thermal transport properties with the hot disk thermal constants analyzer. Rev. Sci. Instrum. **71**, 2452–2455 (2000)

11. H. Zhang, M.-J. Li, W.-Z. Fang, D. Dan, Z.-Y. Li, W.-Q. Tao, A numerical study on the theoretical accuracy of film thermal conductivity using transient plane source method. Appl. Therm. Eng. **72**, 62–69 (2014)

12. M. Ahadi, M. Andisheh-Tadbir, M. Tam, M. Bahrami, An improved transient plane source method for measuring thermal conductivity of thin films: Deconvoluting thermal contact resistance. Int. J. Heat Mass Transf. **96**, 371–380 (2016)

13. S.A. Al-Ajlan, Measurements of thermal properties of insulation materials by using transient plane source technique. Appl. Therm. Eng. **26**, 2184–2191 (2006)

14. S. Flueckiger, T. Voskuilen, Y. Zheng, T.E. Pourpoint, Advanced Transient Plane Source Method for the Measurement of Thermal Properties of High Pressure Metal Hydrides, in: *ASME 2009 Heat Transfer Summer Conference collocated with the InterPACK09 and 3rd Energy Sustainability Conferences: Heat Transfer in Energy Systems; Thermophysical Properties; Heat Transfer Equipment; Heat Transfer in Electronic Equipment*, 2009, vol. 1, pp. 215–221.

15. R.J. Warzoha, A.S. Fleischer, Determining the thermal conductivity of liquids using the transient hot disk method. Part I: establishing transient thermal-fluid constraints. Int. J. Heat Mass Trans. **71**, 779–789 (2014)

16. R.J. Warzoha, A.S. Fleischer, Determining the thermal conductivity of liquids using the transient hot disk method. Part II: establishing an accurate and repeatable experimental methodology. Int. J. Heat Mass Trans. **71**, 790–807 (2014)

17. M. Gustavsson, S.E. Gustafsson, On the use of transient plane source sensors for studying materials with direction dependent properties, in: R. Dinwiddie, R. Mannello (eds.) 26th international thermal conductivity conference/14th international thermal expansion symposium, vol. 26, Cambridge, MA, 2005, pp. 367–377.

18. B. Wicklein, A. Kocjan, G. Salazar-Alvarez, F. Carosio, G. Camino, M. Antonietti, L. Bergström, Thermally insulating and fire-retardant lightweight anisotropic foams based on nanocellulose and graphene oxide. Nat. Nanotechnol. **10**, 277–283 (2015)

19. D. Wu, R. Fu, Requirements of organic gels for a successful ambient pressure drying preparation of carbon aerogels. J. Porous Mater. **15**, 29–34 (2008)

20. C. Scherdel, R. Gayer, T. Slawik, G. Reichenauer, T. Scherb, Organic and carbon xerogels derived from sodium carbonate controlled polymerisation of aqueous phenol-formaldehyde solutions. J. Porous Mater. **18**, 443–450 (2011)

21. J. Feng, J. Feng, C. Zhang, Thermal conductivity of low density carbon aerogels. J. Porous Mater. **19**, 551–556 (2012)

22. Y. Liu, Z. Chen, J. Zhang, S. Ai, H. Tang, Ultralight and thermal insulation carbon foam/SiO_2 aerogel composites. J. Porous Mater. **26**, 1305–1312 (2019)

23. R. Coquard, E. Coment, G. Flasquin, D. Baillis, Analysis of the hot-disk technique applied to low-density insulating materials. Int. J. Therm. Sci. **65**, 242–253 (2013)

24. H. Zhang, Y. Jin, W. Gu, Z.-Y. Li, W.-Q. Tao, A numerical study on the influence of insulating layer of the hot disk sensor on the thermal conductivity measuring accuracy. Prog. Comput. Fluid Dyn. **13**, 191–201 (2013)

25. P. Johansson, B. Adl-Zarrabi, C.-E. Hagentoft, Using transient plane source sensor for determination of thermal properties of vacuum insulation panels. Front. Arch. Res. **1**, 334–340 (2012)

26. A. Elkholy, H. Sadek, R. Kempers, An improved transient plane source technique and methodology for measuring the thermal properties of anisotropic materials. Int. J. Therm. Sci. **135**, 362–374 (2019)

27. H. Zhang, Y. Li, W. Tao, Effect of radiative heat transfer on determining thermal conductivity of semi-transparent materials using transient plane source method. Appl. Therm. Eng. **114**, 337–345 (2017)

28. S. Fantucci, A. Lorenzati, G. Kazas, D. Levchenko, G. Serale, Thermal energy storage with super insulating materials: a parametrical analysis. Energy Procedia **78**, 441–446 (2015)

29. T. Li, J. Song, X. Zhao, Z. Yang, G. Pastel, S. Xu, C. Jia, J. Dai, C. Chen, A. Gong, F. Jiang, Y. Yao, T. Fan, B. Yang, L. Wågberg, R. Yang, L. Hu, Anisotropic, lightweight, strong, and super thermally insulating nanowood with naturally aligned nanocellulose. Sci. Adv. **4**, 3724 (2018)

30. Y. He, Rapid thermal conductivity measurement with a hot disk sensor: Part 1. Theoretical considerations. Thermochim. Acta **436**, 122–129 (2005)

31. C.M. Pelanne, Heat flow principles in thermal insulations. J. Therm. Insul. **1**, 48–80 (1977)

32. ASHRAE, Chapter 26: heat, air, and moisture in building assemblies—material properties, in: 2017 ASHRAE Handbook—Fundamentals (ASHRAE, 2017), pp. 26.21–26.23.

33. G. Chaykovskiy, *Comparison of Thermal Insulation Materials for Building Envelopes of Multi-storey Buildings in Saint-Petersburg* (Mikkeli University of Applied Sciences, Mikkeli, 2010)

34. S. Schiavoni, F. D'Alessandro, F. Bianchi, F. Asdrubali, Insulation materials for the building sector: a review and comparative analysis. Renew. Sustain. Energy Rev. **62**, 988–1011 (2016)

35. Rigid Polystyrene Foam (EPS, XPS), Technical data sheet. https ://products.basf.com. Accessed 26 Nov 2019.

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.