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### Determination of the anisotropic thermal conductivity of an aerogel-based plaster using transient plane source method

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Abstract. Aerogel-based plasters are composite materials with declared thermal conductivities in the range of traditional insulating materials, i.e. 30-50 mW/(m·K). Based on the results from reported field measurements, aerogel-based plasters can significantly reduce the thermal transmittance of uninsulated walls. However, the in-situ measured thermal conductivities have sometimes been higher than the declared values measured in laboratory and in the main direction of the heat flow. Meanwhile, the anisotropic thermal performance of aerogel-based plasters, i.e., deviating thermal performance in the different directions of heat flow, has not been explored yet. The objective of this study is thus to evaluate the anisotropic thermal conductivity of an aerogelbased plaster. This is done in a set of laboratory measurements using the transient plane source method. Six identical and cubic samples with the dimensions of 10x10x10 cm<sup>3</sup> were paired two and two, creating three identical sample sets. In total, 360 measurements of thermal conductivity and thermal diffusivity, and 130 measurements for specific heat capacity were conducted. The results indicate a weak anisotropy of less than  $\pm 6.5$  % between the three directions (x, y, z). Considering the accuracy of the selected measurement technique, better than  $\pm 5$  %, supplementary measurements using another technique are recommended.

#### 1. Introduction

Aerogel-based plasters (APs) are high thermal insulation plasters, with an overall thermal conductivity of lower than 0.1 W/(m·K) [1]. Aerogels are porous and low-density super insulation materials with thermal conductivities around 0.010-0.020 W/(m·K). APs, typically consisting of a lime- and white cement-based binder, a high proportion (>50%) of hydrophobized aerogel granules as aggregates and other additives are considered as inhomogeneous composites. Their level of inhomogeneity can highly be affected during the mixing phase and application process of the fresh mortar.

The development of APs was initiated around one decade ago in Europe [2]. Today, there exist several commercial AP products in the European market. The declared thermal conductivity of these commercially available APs, measured in laboratory, is around 0.030-0.050 W/(m·K) [1]. Previous research has shown that APs have the potential to be a promising solution in many complicated renovation cases of historical buildings, where the number of technical solutions is limited [3,4]. In previous full-scale studies, the application of 1.5-6 cm of AP has reduced the thermal transmittance, Uvalue  $(W/(m^2 \cdot K))$ , of the uninsulated wall elements by approximately 30-70%. At the same time, it has been reported that the measured thermal performance of APs on-site and under real conditions differed sometimes from the declared values measured in laboratory [4–6]. Unlike the in-situ measurements, where the conditions can vary freely depending on the outdoor climate and initial conditions in the studied elements, the conditions in the laboratory, such as the temperature and moisture content of both Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution Ð of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.

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the surrounding environment and the studied element, can be controlled during the measurements. The higher thermal conductivity of APs on-site, compared to the declared values, has often been explained by the higher moisture content during the early phases after the application [3–6].

The declared thermal conductivities of APs are measured in laboratory and in one direction only, i.e. in the main direction of the heat flow. However, for inhomogeneous composites such as APs, there is a theoretical possibility for anisotropic thermal performance. In the case of anisotropic thermal conductivity, the thermal performance of the AP may be deviating in the different directions of heat flow. Thus, it can be expected that the temperature distribution in the AP can be affected as well. This can in turn have an impact on the drying process of moisture and result in an uneven drying rate in the AP applied on a façade. To the authors' best knowledge, the anisotropic thermal performance of APs has not yet been studied. In this study, as a contribution to better understand the thermal performance of APs, the anisotropic thermal conductivity of an AP is investigated.

The aim of this paper is to experimentally evaluate the triaxial anisotropic thermal conductivity of an AP. The experimental study was performed through laboratory-based measurements on thermal conductivity ( $\lambda$ ), thermal diffusivity ( $\alpha$ ) and volumetric heat capacity ( $c_p$ ), using Transient Plane Source (TPS) method (ISO 22007-2 [7]). Three identical sets of samples were prepared in laboratory. The thermal conductivity and thermal diffusivity of the samples were measured in three defined directions by using the so-called standard module of the TPS method. The heat capacity of the samples was measured separately by the so-called specific heat module of the TPS method. In total 360 standard TPS measurements and 130 measurements of heat capacity were performed. Based on the results of the measurements and further analyses, the thermal conductivities of the samples in all three directions (x, y, z) were determined.

#### 2. Experimental measurements

In this chapter, the details concerning the preparation of the samples of AP, the experimental measurements, and the corresponding analyses are presented.

#### 2.1. Sample preparation

A commercially available AP product was selected for the study. The declared thermal conductivity is 0.035 W/(m·K), with a bulk density of 180 kg/m<sup>3</sup>. The mortar was mixed, casted, and cured according to the instructions from the producer. In total, six cubic samples with the dimensions 10x10x10 cm<sup>3</sup> were prepared. The samples were paired two and two and three sets of identical samples (1-3) were created. The samples were cured in 28 days under controlled temperature (T) and relative humidity (RH) conditions: 7 days at 20 °C/95% RH and 21 days at 20 °C/65% RH. The samples were later stored at 20 °C/50% RH in a climate-controlled room for four weeks prior the start of the measurements.

#### 2.2. Transient plane source method (TPS)

The measurements were performed according to the TPS-method [7]. The TPS-method is a transient thermal measurement method that performs measurements during the process of heating a sample. In the TPS method, a double spiral shaped sensor, made of a 10 µm thick Nickel-metal, is used both as a heat source and as a thermometer to measure the resistance [8]. During a measurement, the time dependent temperature increase is recorded by the TPS instrument. In each measurement, 200 resistance recordings are taken during a pre-set time. Based on the recorded values, the relation between the temperature increase and the time is established. As the heat source, the applied power and the corresponding time, and the temperature increase in respect to the power and time are known, the differential equation of heat conduction is solved, and the thermal properties of the samples are determined accordingly. Unlike the other typical steady-state measurement methods, guarded hot plate and guarded heat flow meter [9] that requires a longer measuring time of normally several hours/days, the TPS measurements are done faster and within a few seconds/minutes. Also, the shape and dimension of the samples can be selected more freely in a TPS measurements compared to the steady-state measurement methods. On the other hand, a larger number of measurements are normally required for the TPS-method to achieve reliable results.

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The measurements in this study were performed using the core instrument "Hot Disk TPS 2500 S" [10]. The declared accuracy of the used TPS instrument is better than  $\pm 5$  % [10] and it is capable of measuring thermal conductivities within the range of 0.005 to 1800 W/(m·K). Prior to the presented study, the TPS instrument was controlled by the authors, following the test protocol of the instrument. Ten repeated measurements (power 0.8 W, measurement time 10 seconds) on a test sample made of mild steel, SIS2343, were performed. The highest deviation from the reference values was 1.15 %.

In a standard TPS measurement, described here, the thermal conductivity ( $\lambda$ ) and thermal diffusivity ( $\alpha$ ) of an isotropic material can be measurement. Based on the measured values, the heat capacity ( $c_p$ ) of the material is calculated and provided by the TPS instrument. In a standard TPS measurement, the values provided are based on the assumption of isotropy, i.e. the same properties in all directions are assumed by default. The volumetric heat capacity of the samples was experimentally determined by using the specific heat module of the TPS method. This method is described in detail in the Hot Disk user manual [11]. In this module, a TPS-sensor is attached to a sample holder made of gold. The volumetric heat capacity of a sample can be measured in two steps. The first measurement considers the temperature increase of the empty sample holder (reference measurement). In the second measurement, the sample holder is filled with the considered material and the temperature increase is recorded (sample measurement). By combining the measured results from the reference and sample measurement, the volumetric heat capacity of the sample is obtained. In figure 1 (left) and figure 3, the measurement setups used in this study, both the standard module and the heat capacity module, are shown respectively.



**Figure 1.** Left: Photo showing the setup for the standard TPS measurements for sample set 1. The TPSsensor is clamped between two samples on the x-y plane. Right: Schematic showing the details of the prepared samples, the defined directions (x, y, z) and planes (x-y, x-z, y-z). The selected positions of the TPS-sensor on the x-y plane  $(P_{xy1}-P_{xy.4})$  are illustrated.

#### 2.3. Experimental setup and results

A standard measurement with the TPS method needs a pair of samples. As described in section 2.1, three sets of samples (1-3) were created by pairing the specimens. The TPS-measurements were performed in three directions (x, y, z). The definition of different directions and planes for a cubic specimen is illustrated in figure 1 (right). In each plane, measurements were conducted at four different positions and the mean values of the measured properties in each direction were calculated. The four positions on the x-y plane ( $P_{xy1}$ -  $P_{xy4}$ ) are illustrated in figure 1 (right). On the other two planes (x-z and y-z), sensor positions were selected likewise to the positions illustrated for the x-y plane. The reason for measuring at different positions was to mitigate the potential effects of heterogeneity in the samples and different surface conditions, such as roughness and air voids. At each position, 10 transient measurements were performed. Thus, for each set of samples, 40 transient measurements were performed in each direction. The minimum time between two measurements was set to two hours.

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The results of the measurements are presented in figure 2 and table 1. As described in section 2.2, in the standard TPS-measurements, isotropic conditions are assumed by default. The test conditions (time and power) were optimized through an iterative pre-study of several measurements as suggested in the Hot Disk user manual [11]. Generally, for low thermal conductivity materials as the AP, a selection of a low power for a longer time is recommended. However, it is important that the penetration depth for the generated heat does not exceed the total available thickness of the samples. In this study, a power of 15 mW and a time of 80 seconds (s) were selected for all measurements. The penetration depth was less than 2 cm for all measurements while the thickness of the samples was 10 cm. The selected TPS-sensor had a radius of 6.4 mm.



Figure 2. The results from the conducted standard TPS-measuremnts (average of 40 measurements) for sample sets 1-3 and for measuremnts of  $\lambda$  ( $W/(m \cdot K)$ ) and  $\alpha$  ( $mm^2/s$ ). The presented values for  $c_p$  ( $MJ/(m^3 \cdot K)$ ) were calculated by the TPS-instrument. The lables describe the position of the TPS sensor for the considered measurement. "xy.TPS": Measured thermal properties by TPS, in the plane of the sensor, at which the sensor was positioned on plane x-y, sew figure 1 (right).

Table 1. The coefficient of variation, CV	V (%), for the conducted standard TPS-measurem	ients.
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Thermal Property	Sample 1	Sample 2	Sample 3
$\lambda_{xy.TPS}$	3.0	4.3	4.2
$\alpha_{xy.TPS}$	8.9	5.1	4.4
$C_{p_{xyTPS}}$	6.5	1.9	2.9
$\lambda_{yz.TPS}$	1.6	0.9	2.8
$\alpha_{yz.TPS}$	3.3	3.9	8.2
$C_{p_{NZTPS}}$	2.5	3.3	6.3
$\lambda_{xz.TPS}$	2.4	1.7	0.6
$\alpha_{xz.TPS}$	7.6	2.1	0.7
$C_n$ means	5.2	1.1	0.6

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The results presented in figure 2, indicates that there was about 0-21% and 1-26% deviation in the measured thermal conductivities and volumetric heat capacities between the different directions in each sample, respectively. These deviations could be a sign for anisotropy in the specimens. To further investigate this hypothesis, the volumetric heat capacity of the samples was measured by specific heat module of the TPS method, described in section 2.2. The details and the results of the TPS measurement for volumetric heat capacity ( $c_p$ ) are presented in table 2. In total, three different samples of the AP were prepared. The size of the samples was governed by the shape and dimension of the sample holder. The cylindrical holder had a diameter of 20 mm with a height of 5 mm. Consequently, three samples (1-3) with the same dimensions as the cylindrical holder were produced from the same samples used in the standard TPS-measurements. For these measurements, different combinations of time and power were tested. The minimum time between the measurements was two hours.

As shown in table 2, the measured values varied between  $0.1624 \text{ MJ/(m}^3 \cdot \text{K})$  and  $0.2432 \text{ MJ/(m}^3 \cdot \text{K})$ . The average volumetric heat capacity was decided to  $0.2117 \text{ MJ/(m}^3 \cdot \text{K})$ , with a coefficient of variation (CV) of 12.5 %.



**Figure 3.** Photos showing the used TPS-setup for the determination of the volumetric heat capacity of the samples. a) The sample holder is filled with AP. b) The TPS-sensor and holder are merged into one unit. c) The unit is clamped between two layers of insulation (expanded polystyrene) to mittigate the heat losses to the surrounding.

Table 2.	The details	and the results	of the separate	measurements	for determi	nation of	volumetric	heat
capacity	$(MJ/(m^3 \cdot I))$	<i>K</i> )).						

Number of tests	Power (mW) Reference measurement	Power (mW) Sample measurement	Time (s)	Measured value (average of 10
		6 1 1		measurements)
		Sample 1		
10	30	45	80	0.2288
10	30	45	160	0.2207
10	30	45	40	0.2184
		Sample 2		
10	30	45	80	0.2123
10	30	45	160	0.1713
10	30	45	40	0.2342
10	40	50	160	0.1624
10	40	50	80	0.2368
		Sample 3		
10	30	45	80	0.2144
10	30	45	160	0.1943
10	30	45	40	0.2342
10	40	50	160	0.1799
10	40	50	80	0.2432
Ave	erage of all measurements / (c	oefficient of variation, CV (%	6))	0.2117/ (12.5)

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#### 2.4. Evaluation of the measurement results: triaxial thermal conductivity

In a standard measurement by TPS method, the measured values for the thermal diffusivity of the plane are not affected by the default assumption of the isotropic behavior of the samples. However, the presented measured thermal conductivities are the geometric mean values of the in-plane conductivities and thermal conductivity in direction perpendicular to the plane, see equation (1) as an example for the plane x-y [12]. In case of an anisotropic behavior in the samples, the measured thermal conductivities presented in figure 2, need to be recalculated. The determination of the thermal conductivity in each direction, can be performed by considering the measured thermal diffusivity, presented in figure 2, the measured volumetric heat capacity, presented in table 2, and the relation shown in equation (1), (2) and (3) [12]:

$$\lambda_{xy.TPS} = \sqrt{\lambda_{xy} \cdot \lambda_z} \tag{1}$$

$$\lambda_{xy} = \alpha_{xy.TPS} \cdot c_p \tag{2}$$

$$\lambda_z = \frac{\lambda_{xy.TPS}^2}{\alpha_{xy.TPS} \cdot c_p} \tag{3}$$

where  $\lambda_{xy.TPS}$  (W/(m·K)), is the measured thermal conductivity of the plane x-y,  $\lambda_z$  (W/(m·K)) is the thermal conductivity in the z-direction,  $\alpha_{xy.TPS}$  (mm<sup>2</sup>/s) is the measured thermal diffusivity of the plane x-y, and  $c_p$  (MJ/(m<sup>3</sup> · K)) is the measured volumetric heat capacity.

Similar to the equations shown above, the thermal conductivities in the other two directions can be determined, see equation (4) and (5) [12]:

$$\lambda_x = \frac{\lambda_{yz.TPS}^2}{\alpha_{yz.TPS} \cdot c_p} \tag{4}$$

$$\lambda_y = \frac{\lambda_{xz,TPS}^2}{\alpha_{xz,TPS} \cdot c_p} \tag{5}$$

where  $\lambda_{yz.TPS}$  (W/(m·K)) and  $\lambda_{xz.TPS}$  (W/(m·K)) are the measured thermal conductivity of the plane y-z and x-z respectively,  $\lambda_x$  (W/(m·K)) and  $\lambda_y$  (W/(m·K)) are the thermal conductivity in the x- and y-direction respectively, and  $\alpha_{yz.TPS}$  (mm<sup>2</sup>/s) and  $\alpha_{xz.TPS}$  (mm<sup>2</sup>/s) are the measured thermal diffusivity of the plane y-z and x-z respectively.

The calculated thermal conductivities in all three directions (x, y, z) and the corresponding differences are presented in table 3. Among the calculated thermal conductivities in three directions and for three sample sets, the maximum different was identified for sample set 1 and between the z- and y-direction (6.51 %). At the same time, the minimum difference was for sample set 2 and between the z- and y-direction (0.26 %).

**Table 3.** The calculated thermal conductivities in different directions (x, y, z) and the corresponding differences between the directions.

$\lambda$ -value ( $W/(m \cdot K)$	())	Deviation (%)					
	$\lambda_x$	$\lambda_{y}$ $\lambda_{z}$ $\lambda_{x} \& \lambda_{y}$ $\lambda_{x} \& \lambda_{z}$ $\lambda_{y} \& \lambda_{z}$					
Sample 1	0.0481	0.0499	0.0466	3.45	3.16	6.51	
Sample 2	0.0460	0.0477	0.0476	3.53	3.28	0.26	
Sample 3	0.0487	0.0480	0.0458	1.68	6.17	4.56	

#### **3.** Conclusions

The triaxial anisotropic thermal properties of an aerogel-based plaster were determined using the Transient Plane Source (TPS) method (ISO 22007-2). Three set of samples were investigated to determine the thermal conductivity and thermal diffusivity in three directions (x, y, z), and the volumetric heat capacity of the material.

The variations in the thermal conductivity in different directions (x, y, z) were around  $\pm 6.5$  % at the highest. This result can indicate a weak anisotropic thermal performance in the material. Meanwhile, the accuracy of the TPS instrument used in this study was declared to be better than  $\pm 5$  %.

Based on the measured thermal properties of the aerogel-based plaster, the TPS-method can be a suitable testing method for the thermal evaluation of aerogel-based plasters. However, the method requires a large number of measurements compared to other steady-state methods. The main advantage of the TPS method is the shorter measurement time when steady-state conditions are not needed. On the other hand, to accurately evaluate the anisotropic thermal performance of aerogel-based plaster, the presented study by TPS method needs to be further supplemented by other testing methods.

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