THERMOPHYSICAL PARAMETERS OF CALCIUM SILICATE INSULATION MEASURED BY GUARDED HOT PLATE AND PULSE TRANSIENT METHODS

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Abstract

Thermal conductivity of a highly porous thermal insulation material was measured by two different methods – guarded hot plate method and pulse transient method. The application of different measurement methodologies and interpretation of results enabled to obtain more complex information on the parameters of tested material.

Key words: thermal conductivity, guarded hot plate method, pulse transient method, highly porous materials

1 Introduction

Effective thermal conductivity of highly porous calcium silicate (CS) thermal insulation material has been determined within the range of hygroscopic moisture contents by two different methods: guarded hot plate method (GHPM) and pulse transient method (PTM). The obtained results were compared with the aim to evaluate reliability and to determine possibilities of the transient method in measurements of thermophysical parameters of highly porous materials.

The tested CS insulation is an inorganic material composed of the hydrous calcium silicate - Xonotlite and the cellulose reinforcing fibres. It is produced by autoclaving the slurry of lime and silica powder, adding fibrous filler in it and then forming the resulting mixture into the desired shape. Basic material properties of CS are presented in Table. 1. The high porosity of the CS is given by the apparent fibrous microstructure of Xonotlite (Fig. 1).

Tab. 1. Material parameters of measured Calcium silicate plates

Total porosity	Bulk density	Specific surface area (MIP)	Maximum hygroscopic moisture content	Matrix volume fraction	Fibres volume fraction
$[m^3/m^3]$	$[kg/m^3]$	$[10^6 \, m^2/m^3]$	[% vol.]	[-]	[-]
0.9	280	13.14	2.0	0.0992	0.0083

2 Experimental

The GHPM is steady-state method for determination of thermal conductivity [3]. Dimensions of the measured CS samples were 0.50 x 0.50 x 0.04 m, the mean sample temperature was ca 19°C and the temperature difference was ca 6 K during the measurements.

The measurements were carried out for the moisture content values from 0 to 8% vol. The samples were moistened by spraying and then conditioned for 48 h to achieve the homogenous moisture content distribution. The goal of the measurements was to determine the effective thermal conductivity λ comprising the following mechanisms of heat transfer: the heat conduction in solid, liquid and gaseous phases and the radiation transport in the pore space. The value of thermal conductivity measured by GHPM is the apparent thermal conductivity, which includes in principle the enthalpy flow and the spatial conductivity variability due to non-linear moisture content distribution effects during the measurement. Therefore the effective thermal conductivity had to be determined by excluding these effects. For the evaluation of the effects of the moisture flow and phase change on the apparent thermal conductivity the numerical simulations of temperature-moisture field in the measured samples were used [4]. The determined dependency of the effective thermal conductivity on the moisture content for the CS is shown in Fig. 2.

The PTM is a method based on the generation of a dynamic temperature field inside the sample [6]. A small disturbance in the form of a heat pulse is applied to the sample and from the measured temperature response the thermophysical parameters as thermal diffusivity, specific heat and thermal conductivity can be calculated. Optimal geometry of the sample setup means: the optimal distance between the heat source and the thermometer and the diameter of the sample which enables to suppress the effects of heat loss from free sample surfaces and contact thermal resistance effects.

Dimensions of the measured sample were 0.15 x 0.15 x 0.038 m, the pulse width was 5 – 120 s, heat pulse energy was 5000 – 40000 $J.m^{-2}$. Stable data – not influenced by contact and heat loss effects – were found for the thermocouple distance from the heat source in the range between 15 and 22.5 mm [2].

Fig. 1 Xonotlite as seen under the scanning electron microscope

3 Analysis of results and discussion

The results of the moisture content dependent CS thermal conductivity measurements by GHPM were analysed in detail in [5]. From this analysis it follows that the thermal conductivity in the range of hygroscopic moisture contents can be expressed by a parallel configuration of particular material phases:

$$\lambda(u_v) = \lambda_s(u_v) \cdot (1 - \Phi) + \lambda_a \cdot (\Phi - u_v) + \lambda_w \cdot u_v \tag{1}$$

where: Φ is the porosity of material [-], u_v is the moisture content [m^3/m^3], $\lambda_s(u_v)$ is the thermal conductivity of skeleton, λ_a and λ_w are the thermal conductivities of air and water [W/m.K].

The thermal conductivity of skeleton changes depending on the moisture content due to water vapour sorption-desorption induced swelling-shrinkage process equals:

$$\lambda_s(u_v) = \cfrac{1 - \Phi}{\cfrac{1 - \Phi}{\lambda_{so}} + 3 \cdot \varepsilon \cdot 10^{-3} \cdot \left(\cfrac{\dfrac{u_v}{u_{h\max}}}{\lambda_w} + \cfrac{\dfrac{u_{h\max} - u_v}{u_{h\max}}}{\lambda_a} \right) + \cfrac{3 \cdot \varepsilon_{crack} \cdot 10^{-3} \cdot \dfrac{u_{h\max} - u_v}{u_{h\max}}}{\lambda_a}} \tag{2}$$

where: λ_{so} is the thermal conductivity of the solid matrix [W/m.K], ε [mm/m] is the maximum material swelling strain, representing the maximum volume of the water disjoining the solid and taking part in the creation of thermal bridges, ε_{crack} [mm/m] is the material extension due to multiple matrix cracking. In the case of measured CS material: $\lambda_{so} = 1.0$ W/m.K, $\varepsilon = 0.28$ mm/m and $\varepsilon_{crack} = 0.9$ mm/m [5].

Applying the relations (1) and (2) the following values of the thermal conductivity obtained by the GHPM have resulted for:
- the skeleton of a dry material $\lambda_s(0) = 0.042$ W/m.K,
- the dry material $\lambda(0) = 0.066$ W/m.K,
- the material at the moisture content of 0.41% vol. $\lambda(0.0041) = 0.075$ W/m.K.

The results of the PTM (Fig. 3, [2]) have given the following values of thermal conductivity for:
- the original material conditioned at 50% relative humidity: 0.081 W/m.K,
- the evacuated material (p = 100 Pa): 0.042 W/m.K,
- the aerated sample before reassembling: 0.065 W/m.K,
- the reconditioned material (in a room with ca 50% relative humidity) and the reassembled sample: 0.076 W/m.K.

From the comparison of the results achieved by GHPM and PTM it follows that the value of thermal conductivity of the original material conditioned at 50% relative humidity and measured by PTM is by 8% higher than thermal conductivity measured by GHPM at corresponding moisture content. Taking into account that the moisture content of sample measured by PTM was determined approximately, the coincidence of the results of both methods is sufficient.

The contribution of the different modes of heat transfer to the total effective thermal conductivity of high porosity materials is shown in Fig. 4. The radiation is significant only for low density materials and at the porosities lower than 0.95 is practically
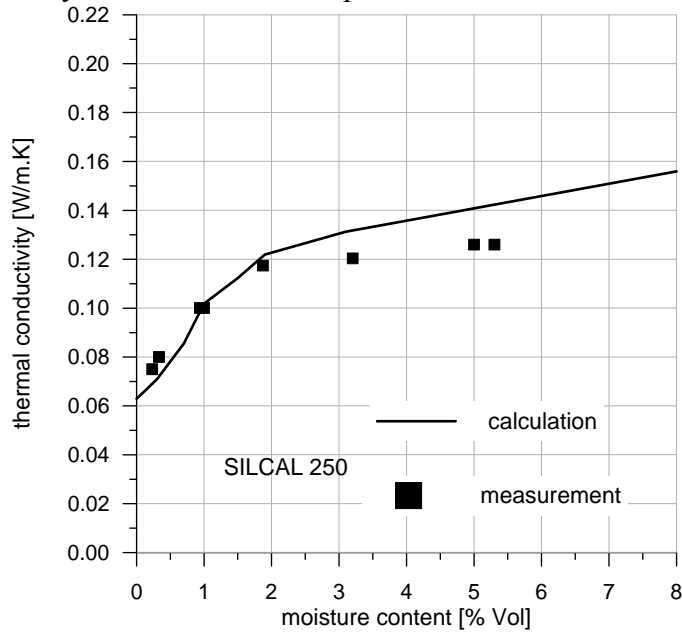

Fig. 2 The comparison of the calculated (Equations 1 and 2) and GHPM measured effective thermal conductivity for the calcium silicate with bulk density of 280 kg/m^3.
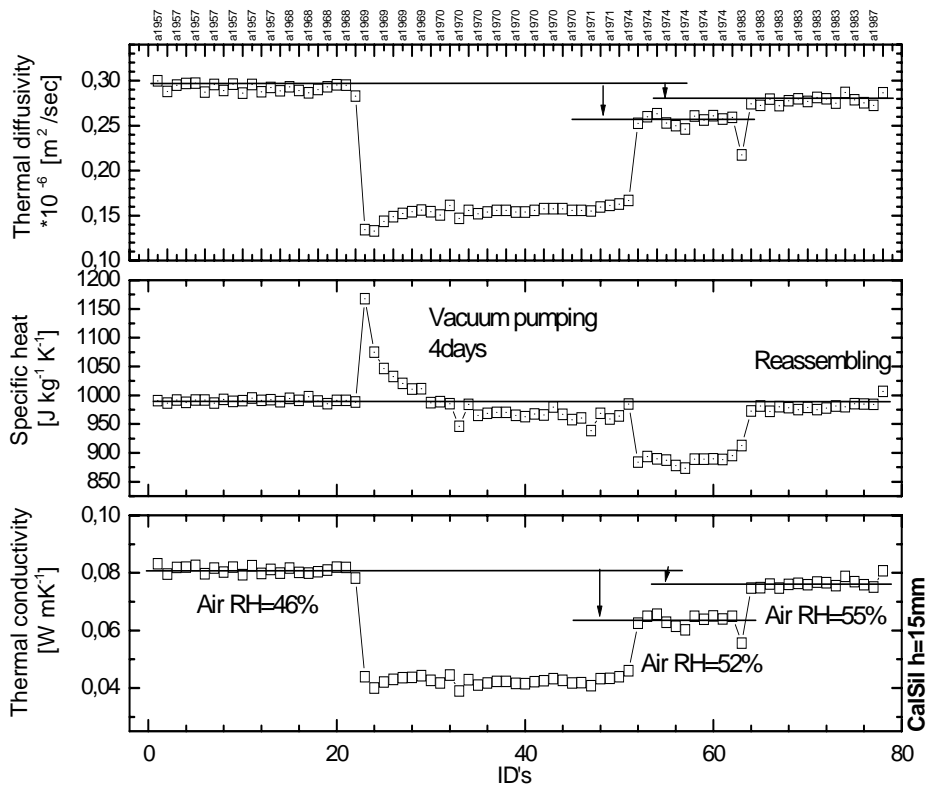
Fig. 3 The results of the ed (Equations 1 and 2) and pulse transient method method measured effective thermal conductivity for the calcium silicate with bulk density of 280 kg/m^3.

negligible. If the pressure of the gas in the material is reduced its thermal conductivity is given by [1]:

$$\frac{\lambda_e}{\lambda_0} = \frac{p \cdot d}{p \cdot d + 2.332 \cdot 10^{-5} \cdot T} \tag{3}$$

where: λ_e is the thermal conductivity at reduced pressure [W/m.K], λ_0 is the thermal conductivity at atmospheric pressure [W/m.K], p is the pressure [Pa], d is the characteristic dimension of a pore space [m], T is the absolute temperature [K].

According to the relation (3) the decrease of the pores dimension leads at a given pressure to the decrease of the thermal conductivity of the gas in a material. In the case of CS material the characteristic dimension of its pore space is represented by the median pore diameter equal to 574 nm. This value gives a tenfold reduction in the pore air thermal conductivity under the air pressure after the evacuation of 100 Pa.
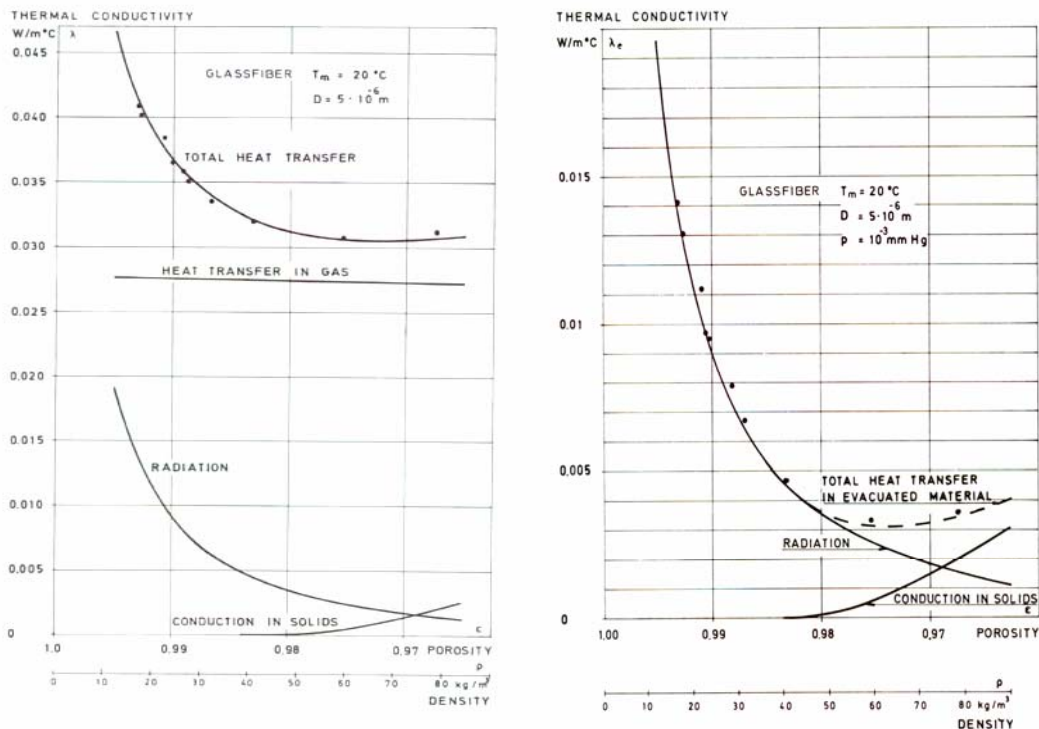

Fig. 4. The mechanisms of heat transfer in non-evacuated and evacuated material [1]

The influence of the evacuation to the thermal conductivity is effective mainly in smaller pores. However in given experiments that influence was not observed. The comparison of the results for non-evacuated and evacuated samples by the relation (2) gave that the thermal conductivity of the evacuated material is identical with the thermal conductivity of the skeleton of a dry material at atmospheric pressure.

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The only explanation here is the fact that the skeleton of the tested material was not evacuated. The same phenomenon was observed in a case of autoclaved aerated concrete (AAC) [7]. In the case of CS the non-evacuated skeleton pore volume represents 0.12% volume of the pores with radii lower than 100 nm compared with the evacuated ca 90% pore volume. In the case of AAC the 30% volume of the skeleton pores with radii lower than 0.1 mm was not evacuated in comparison with the evacuated 50% volume of macropores.

During the processes of evacuation and aeration the changes in a specific heat were noticed. The changes correspond to the effects of additional heat consumption and additional heat release during desorption and adsorption of air and water vapour molecules on the pores walls.

The thermal conductivity value of the aerated sample before reassembling corresponds to thermal conductivity of dry the material measured by GHPM. The relatively sudden aeration process did not enable to achieve the equilibrium moisture content corresponding to 50% relative humidity immediately. As the moisture content of the sample was not determined, it is not clear whether the lower thermal conductivity of the aerated sample was fully caused by the absence of water molecules in the pores of the material or by the combination of lower moisture content with some other effects connected with the evacuation-aeration process.

The values of thermal conductivity of aerated and reassembled sample were practically identical to thermal conductivity measured by GHPM at corresponding moisture contents.

4 Conclusion

The results of two methods - guarded hot plate method and pulse transient method - were compared with the aim to evaluate a reliability and to determine possibilities of the transient method in measurements of thermophysical parameters of highly porous materials.

The thermal conductivities of calcium silicate conditioned at 50% relative humidity obtained by the guarded hot plate method and the pulse transient method were practically identical.

The thermal conductivity of the evacuated sample measured by the pulse transient method was identical to the thermal conductivity of skeleton of a dry material at the atmospheric pressure obtained from the measurement by the guided hot plate method. Utilizing the evacuation process the pulse transient method enables to identify the thermal conductivities of the skeletons of porous materials.

Pulse transient method enables to monitor the effects of the heat consumption and release during gas desorption and sorption processes.

For a futher more detailed comparison of the methods the repeated measurement with the drying of the samples before evacuation, determination of actual moisture content of measured sample and the evacuation of the samples at lower pressures are recommended.

Acknowledgement

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