ABSTRACT

In the study of the mechanical behaviour of building materials in presence of fire, the knowledge of the correlation of the thermal conductivity and of the specific heat with temperature is of fundamental importance. The numerical verifications of fire resistance of structures are often carried out using computer codes that solve differential equations of thermal exchanges. For the correct prediction of the evolution with the temperature, the knowledge of the above thermal properties as a function of temperature is required.

The measurement of thermal conductivity and thermal diffusivity has been made with the Hot Disk Thermal Constants Analyser equipment. This apparatus, that operates in variable speed, generates a thermal constant power in a nickel sensor placed in contact with two specimens of the same material. It allows the detection of temperature increase on the surfaces of these specimens.

Assuming as infinite the specimens size, the solution of the general equation of the heat conduction allows to express the temperature increase in of the specimen as a linear dimensionless time function; the simultaneous determination of thermal diffusivity and conductivity of the test material becomes easy.

The experimental environment suitable to achieve high temperatures consists of a special controlled temperature oven in which measurements are made with sensors enclosed in two layers of refractory material (mica) suitable for working with acceptable accuracy in a range of temperatures between 500-1000 K.

This paper presents the results of experimental measurements of thermal conductivity and of volumetric specific heat, \( \rho \cdot c_p \), carried out at high temperature on autoclaved aerated concrete (AAC) specimens at different densities.

The results are also compared with the directions on the material provided by national and international standards.

INTRODUCTION

Aerated autoclaved concrete, AAC, is a material commonly used in buildings for its good properties of thermal insulation, mechanical strength and resistance to high temperatures, which may be the ones that occur during fires.

In computational testing of fire resistance, the evolution of the temperature field with time must necessarily be determined by solving differential equations of heat transfer. The proper integration of these equations, however, requires knowledge of the thermal properties such as the thermal conductivity and the specific heat of the material, both depending on temperature.

To experimentally measure these properties at high temperatures, the most appropriate ways are in dynamic regime. In this paper, we used the so called Transient Plane Source method (TPS) for the determination of the above properties, in the temperature range between 300-900 K.

NOMENCLATURE

- \( a \) [m\(^2\)/s]: Thermal diffusivity
- \( c_p \) [J/kg K]: Specific heat
- \( m \) [-]: Number of turns of the sensors
- \( P \) [W]: Thermal power
- \( \rho \) [-]: Porosity
- \( q_r \) [W/m\(^2\)]: Radiative heat transfer
- \( r \) [m]: Porosity mean radius
- \( R \) [m]: Sensor radius
- \( s \) [-]: Deviation
- \( t \) [°C]: Temperature
- \( T \) [K]: Absolute temperature
- \( V \) [m\(^3\)]: Total volume

Special characters
- \( \beta_R \) [m\(^{-1}\)]: Rosseland extinction coefficient
- \( \varepsilon \) [-]: Emissivity
DESCRIPTION OF THE MEASUREMENT AND EXPERIMENTAL PROCEDURE

The common steady-state methods for measuring material thermal conductivity, such as guarded hot plate and heat flow meter methods, are not easily applicable in high-temperature measurements. In these cases it is possible to use transient methods. In this study the so called transient plane source method will be used [1-3].

This technique involves the generation of a constant thermal power in a disc-shaped sensor (suitably chosen radius $R$), composed of a number $m$ of concentric coils, placed in contact with two specimens of the same material, in this study they are samples of AAC (see Figure 1).

The Hot Disk method is based on the use of a transiently heated plane sensor that consists of an electrically conducting pattern in the shape of a double spiral, which has been etched out of a thin nickel foil. This spiral is sandwiched between two thin sheet of insulating material kapton, mica etc., the latter particularly suitable for operating with sufficient sensitivity and accuracy in a range between 500-1000 K.

During the measurements the Hot Disk sensor is fitted between two pieces of the specimen, each one with a plane surface facing the sensor. By passing an electrical current, high enough to increase the temperature of the sensor between a fraction of degree up to several degrees and at the same time record the resistance increase as a function of time, the Hot Disk sensor is used both as a heat source as a dynamic temperature sensor.

This sensor, in addition to providing thermal power, works as a resistance thermometer and allows to measure the temperature increase of the specimen active surfaces, as a result of the thermal power applied.

The solution of the thermal conductivity equation is based on the assumption that the Hot Disk sensor is located in an infinite medium, which means that the transient recording must be interrupted as soon as any influence from the outside boundaries of the two sample pieces is being recorded by the sensor.

The method requires the measurements in times of trial such as to render negligible the influence of the boundary conditions applied to a sample of finite size: thermal analysis, indeed, is done by taking the sample as an infinitely extended one. This means that the test should end as soon as the sample outline influences the measurements.

In this case, applying the solution of the general equation for conduction and working iteratively starting from the Hot Disk measured temperatures, it is possible to calculate the thermal conductivity and the diffusivity of the tested material in an independent way.

\[ \Delta t = \frac{P}{\pi^{1/2} \lambda R m^2 (m+1)^2} \cdot F(\sigma) \]  

(1)

where:

- $\Delta t$ is the average temperature increase of the specimen active surfaces at the general time;
- $P$ is the thermal power generated by the sensor;
- $\lambda$ is the thermal conductivity;
- $R$ is the sensor radius;
- $m$ is the number of sensor turns.

Figure 1 Hot disk sensor and its placement between the two specimens

Figure 2. Thermal response in thermal conductivity and diffusivity measured with the Hot Disk method for AAC 450 at 473 K.

Temperature increase measured during the heating of the specimen active surfaces are put in relation to a dimensionless time function, $\sigma$, obtaining in this way the linear trend predicted by Equation 1. In this way it is possible to evaluate the thermal conductivity $\lambda$ and thermal diffusivity $\sigma$ of the tested material as it is possible to see from Figure 2 [4].
The function $F(\sigma)$ is defined as:

$$F(\sigma) = \frac{\sigma}{\int_0^1 u^2 du \sum_{i=1}^m \sum_{k=1}^m \left( \frac{k^2 + k^2}{4\pi^2 m^2} \right) I_0 \left( \frac{lk}{2\mu^2m^2} \right)}$$

(2)

where:

- $I_0$ is Bessel amended function of order 0;
- $\sigma$ is the dimensionless time, defined as $\sigma^2 = \tau / \Theta$;
- $\Theta$ is the time $\Theta = R^2/a$;
- $a$ is the thermal diffusivity $a = \lambda / (\rho c)$.

Equation (1) is linear:

$$\Delta t = \frac{C}{\lambda} \cdot F(\sigma)$$

(3)

where we indicated with $C$ a constant depending on the instrument characteristics and on the thermal power generated by the sensor.

The regression of temperature increase experimental data versus time allows to evaluate the thermal conductivity and diffusivity best values as soon as the choice of parameters verifies the linear equation (3).

In this study the results of thermal conductivity experimental measurements carried out on autoclaved aerated concrete specimens with a nominal density 350 kg/m$^3$ and 450 kg/m$^3$ are reported. Measurements were performed at different temperatures in a range between 300-900 K. To prevent any experimental error due to the nickel solid state transition we avoided measurements in the temperature range between 573-673 K.

For each selected temperature, 15 measurements for the density $\rho = 350$ kg/m$^3$ and 6 measurements for the density $\rho = 450$ kg/m$^3$ have been carried out. In both cases a power of 0.1 W for a range of 80 s was applied.

The used sensors were of two different types: the first covered with kapton and radius $R = 9.869$ mm, up to a temperature of 500 K, the second coated with mica and radius $R = 9.719$ mm for the higher temperatures (see Figure 3).

**FORECAST MODEL FOR DETERMINING THE THERMAL CONDUCTIVITY**

The studied material can be considered as a porous medium consisting of a solid matrix crossed by a network of spaces occupied partly by moist air and partly by liquid water. In our model it is assumed that any liquid water is trapped inside the porous solid matrix. The material heat transfer mechanisms are, therefore, shown as conduction in the solid matrix and thermal conduction and radiation in the pores; we also consider convection negligible due to the small size of the pores (about 0.8 mm).

Several models have been proposed to express the equivalent thermal conductivity. They were usually obtained as a combination of two elementary models which provide the material distribution in series and in parallel to the heat flow.

For example, the following established and relatively simple model suggested by Russell [5], can be used for this type of material:

$$\lambda_{eq} = \lambda_s - \frac{\lambda_{por}}{\lambda_{por} + 1} \left[ Por - Por^2/3 - Por^2/3 \right]$$

(4)

where:

- $\lambda_s$ is the solid matrix conductivity;
- $\lambda_{por}$ is the porous apparent conductivity (it includes both the conductive gas exchange and the radiative one);
- $Por$ is the porosity of the medium, defined as $Por = V_v/V$ where $V_v$ is the voids volume and $V$ is the total volume.

In the study of the radiative exchange, the medium can be considered optically thick and, therefore, Rosseland diffusive model is applicable. This model describes the radiative exchange, $q_r$ [W/m$^2$], as [6]:

$$q_r = -\frac{4\sigma_p T^3}{3} \frac{\partial T}{\partial x} = -\lambda_r \frac{\partial T}{\partial x}$$

(5)

where:

- $\sigma_p$ is Stefan Boltzmann constant: 5.67·10$^{-8}$ W/(m$^2$·K$^4$);
- $T$ is the temperature;
- $\beta_r$ is Rosseland extinction coefficient;
- $\lambda_r$ is the quantity, dimensionally equivalent to a thermal conductivity, defined as radiativity:

$$\lambda_r = 4\sigma_p T^3 \left( \frac{3}{4\beta_r} \right)$$

In the event that the porosity can be considered spherical, the Rosseland extinction coefficient may be related to the radius of...
the pores, \( r \), and to the cavity surface emissivity, \( \varepsilon \); \( \beta_r = 1/(r \varepsilon) \) [6].

Ultimately, heat exchange inside porosity can be expressed by an equivalent conductivity:

\[ \lambda_{\text{por}} = \lambda_s + \lambda_r. \]

For the evaluation of the thermal conductivity of the air as a function of temperature, the following expression which approximates the data reported in [8] within the 0.5\% has been used:

\[ \lambda_a = A + BT + CT^2 + DT^3 \]

where:

\( A = -1.4109119 \times 10^{-3} \text{ W/(mK)}; \)
\( B = 1.1038605 \times 10^{-4} \text{ W/(mK}^2); \)
\( C = -6.9545118 \times 10^{-8} \text{ W/(mK}^3); \)
\( D = 3.1611 \times 10^{-11} \text{ W/(mK}^4). \)

RESULTS AND CONSIDERATIONS

Tables 1, 2 and Figures 4, 5 show the thermal conductivity and diffusivity measurement results carried out with the Hot Disk equipment on the autoclaved aerated concrete specimens of density \( \rho = 350 \text{ kg/m}^3 \) and \( \rho = 450 \text{ kg/m}^3 \). The error bars, shown in the figures, refer to a confidence interval of 2 on the measurements repeated for each experimental point.

It is possible to see the increment of the thermal conductivity with temperature that reaches respectively the value of 0.27 \text{ W/(m-K)} for the density of 350 kg/m\(^3\) and 0.32 \text{ W/(m-K)} for the density of 450 kg/m\(^3\) at a mean test temperature of 900 K. These values are about two times the thermal conductivity at room temperature.

Thermal behaviour of aerated concrete in function of temperature is therefore in contrast to ordinary concrete for which, on the contrary, thermal conductivity decreases with the increasing of the temperature. At a mean test temperature of 900 K in fact, the thermal conductivity is only 60\% of the measured value at room temperature [9].

This behaviour is due to the increasing of the thermal conductivity inside the cells of the material and mainly to the increasing of the radiative effect inside the cavities.

The experimental results were then interpolated using the above described model of Russel.

The best regression was obtained using a conductivity \( \lambda_s = 0.48 \text{ W/(m-K)} \) for the solid matrix with a porosity \( \text{Por} = 0.74 \) for the material with density \( \rho = 350 \text{ kg/m}^3 \) and a porosity \( \text{Por} = 0.67 \) for the material with density \( \rho = 450 \text{ kg/m}^3 \). An extinction coefficient of radiation \( \beta_r = 1389 \text{ m}^{-1} \) was also used, corresponding to a cavity mean radius \( r = 0.00080 \text{ m} \) with an emissivity \( \varepsilon = 0.9 \).

All these values agree perfectly with those known for this kind of material and as it is possible to see from Figure 4 and 5, the model interpolates experimental data with a good accuracy.

| Table 1. Thermal conductivity, \( \lambda \), and thermal diffusivity, \( a \), experimental measurements for AAC 350 kg/m\(^3\). |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| \( T \) \[\text{°C}\] | \( \lambda \) \[\text{W/(mK)}\] | \( s_a \) \[\text{W/(mK}^2\)] | \( a \times 10^6 \) \[\text{m}^2/\text{s}\] | \( s_a \times 10^6 \) \[\text{m}^2/\text{s}\] |
| 20 | 0.125 | 0.000254 | 0.356 | 0.000843 |
| 50 | 0.128 | 0.00102 | 0.349 | 0.0115 |
| 90 | 0.135 | 0.000462 | 0.343 | 0.00360 |
| 150 | 0.141 | 0.000329 | 0.347 | 0.00379 |
| 200 | 0.145 | 0.000341 | 0.351 | 0.00326 |
| 250 | 0.186 | 0.000331 | 0.385 | 0.0173 |
| 300 | 0.193 | 0.000482 | 0.414 | 0.0264 |
| 450 | 0.274 | 0.00776 | 0.523 | 0.0471 |
| 500 | 0.262 | 0.00519 | 0.490 | 0.0684 |
| 550 | 0.287 | 0.00649 | 0.516 | 0.0512 |
| 600 | 0.304 | 0.00804 | 0.555 | 0.0857 |

| Table 2. Thermal conductivity, \( \lambda \), and thermal diffusivity, \( a \), experimental measurements for AAC 450 kg/m\(^3\). |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| \( T \) \[\text{°C}\] | \( \lambda \) \[\text{W/(mK)}\] | \( s_a \) \[\text{W/(mK}^2\)] | \( a \times 10^6 \) \[\text{m}^2/\text{s}\] | \( s_a \times 10^6 \) \[\text{m}^2/\text{s}\] |
| 20 | 0.147 | 0.00300 | 0.354 | 0.0103 |
| 50 | 0.152 | 0.000347 | 0.339 | 0.00216 |
| 90 | 0.159 | 0.000965 | 0.336 | 0.00423 |
| 150 | 0.164 | 0.000786 | 0.336 | 0.00579 |
| 200 | 0.166 | 0.00121 | 0.337 | 0.00529 |
| 250 | 0.211 | 0.00163 | 0.375 | 0.00419 |
| 300 | 0.213 | 0.00391 | 0.376 | 0.0154 |
| 450 | 0.288 | 0.00564 | 0.384 | 0.0168 |
| 500 | 0.279 | 0.00825 | 0.460 | 0.0364 |
| 550 | 0.302 | 0.00952 | 0.443 | 0.0424 |
| 600 | 0.316 | 0.0134 | 0.469 | 0.0721 |

Figure 4. Thermal conductivity of autoclaved aerated concrete with density 350 kg/m\(^3\) versus temperature.
Table 3, finally, shows the thermal conductivity values for various densities of aerated concrete as a function of the temperature as suggested by European standard EN 12602. It is possible to use these values to evaluate the thermal field evolution versus time in computational testing of structure of good fire resistance.

We can see that the standard values are significantly lower than those measured with the Hot Disk device, making them just precautionary for the fire resistance calculation.

Table 3. Thermal conductivity \( \lambda \) expressed in W/(m·K) at high temperature according to EN 12602.

<table>
<thead>
<tr>
<th>Temperature [K]</th>
<th>Density kg/m(^3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>0.08</td>
</tr>
<tr>
<td>400</td>
<td>0.10</td>
</tr>
<tr>
<td>500</td>
<td>0.12</td>
</tr>
<tr>
<td>600</td>
<td>0.14</td>
</tr>
</tbody>
</table>

CONCLUSION

Autoclaved aerated concrete thermal conductivity is temperature dependent. In particular, although cement paste thermal conductivity decreases with temperature, aerated concrete thermal conductivity increases with temperature because of the air thermal conductivity increase and, mainly, due to the increase of radiative heat transfer inside the cavity.

In fact, while at room temperature, thermal radiation present in the material pores is a small fraction (<5%) of the total heat exchange, it increases with increasing temperature according to the fourth power of absolute temperature becoming, at a temperature of 900 K, about 55% of the total.

It was also verified that thermal conductivity values at high temperatures, suggested by the standard EN 12602, are significantly lower than those experimentally measured with the Hot Disk equipment in this study and thus they are not precautionary checks for computational fire resistance. This means that it would be necessary to investigate on the source of the suggested data and probably it will be necessary to ask a revision of the above mentioned standard.

REFERENCES