Laser-Flash Measurements on Moist Porous Materials [1]

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ABSTRACT

The influence of temperature, moisture and ice content on the thermal diffusivity of a clinker was measured using the laser-flash technique. For this a special modified laser flash apparatus with a HgCdTe-detector for temperatures from -50 °C to +230 °C was used. The results have been compared with measurements of the thermal conductivity by the heat flow meter apparatus and measurements of the specific heat by a Calvet-DSC as a function of the temperature and of the moisture content.

It was found a good agreement between this techniques. The expenditure needed for the characterization of the influence of the water content and of the ice content on the thermal diffusivity could be considerably reduced.

KEY WORDS: thermal diffusivity; thermal conductivity; specific heat; moisture dependence; porous ceramic material, laser flash technique

1. INTRODUCTION

The influence of the moisture and the ice content on the “apparent“ thermal conductivity of porous material is of interest because of the additional heat transport by vapor diffusion of water and heat conduction along the ice or moisture film. The measurement of the thermal conductivity of such materials with guarded hot plate or heat flow meter instruments is complicated by the moisture migration inside of the specimen, initiated by the temperature field necessary for the measurement. The vapor diffusion of the moisture is combined with adsorption and desorption processes with corresponding latent heat.

For the quantitative understanding and modeling of such processes it is necessary to separate the heat transport by diffusion during the transient hygric phase from the additional heat conduction under hygric steady state conditions via the liquid film. For measurements with the guarded hot-plate method, the typical time to reach hygric steady state conditions in a specimen of (10...50) mm thickness by a prescribed temperature profile, is between one day and two weeks. Therefore such measurements are expensive and time consuming. Contrary to that thermal measurements by the laser flash technique need a much shorter measuring time (0,1...10) s and smaller specimens with (1...3) mm thickness.

2. EXPERIMENTAL

The thermal diffusivity ***a*** of the clinker was measured in an inert atmosphere (dry nitrogen) using a modified Netzsch Model 427 laser flash diffusivity apparatus. The instrument used in this work was equipped with a liquid-cooled furnace and a HgCdTe-detector capable of operation from -50°C to +230°C. For the data analysis the theoretical equation of Cape & Lehman [1] with finite pulse correction and heat loss

correction (Dusza/Netzsch software) was used. The diameter of the specimens was 12,5 mm. Because of the inhomogeneous character of the material the influence of the surface roughness (resulting from the porosity) has been tested by variation of the specimen thickness (1,8...2,3) mm and of the coating (graphite, SiC, no coating,). A systematic influence of the thickness of the specimen and of the coating was not observed. For the measurement of the specific heat c_p a Setaram Calvet-DSC II was used. The measurements were carried out in the temperature range from -20 °C to +100 °C at a heating rate of 0,2 K / min.

The thermal conductivity l of the moist material was determined by a two-heat-flow-meter apparatus described in [2] at specimen dimensions of 85 mm diameter and 20 mm thickness. The heat flow meter calibration was performed by direct calibration of a “transfer“ heat flow meter with a guarded hot plate instrument and with Pyrex glass as reference material [3]. The measurement of the thermal conductivity of the dry clinker as a function of temperature has been performed with a Holometrix TCHM-LT heat flow meter apparatus at a specimen diameter of 50 mm and 15 mm thickness.

3. RESULTS

First measurements (Fig.1.) were performed on dry material as a function of temperature. The specimens for laser flash and heat flow meter investigations have been cut out of the same piece of clinker.

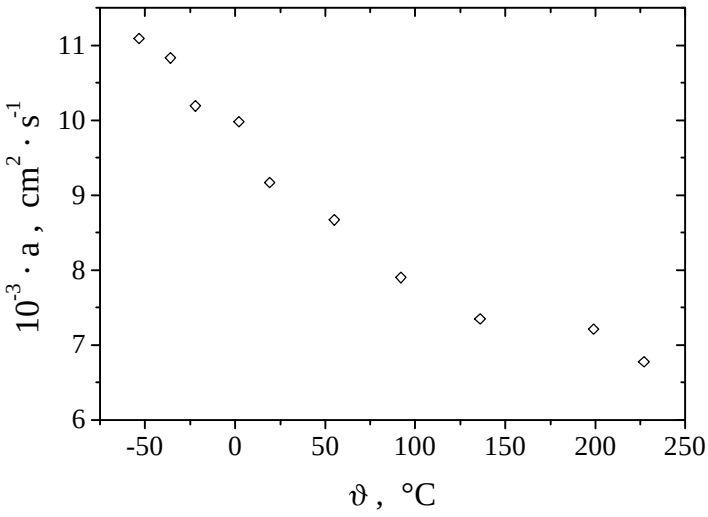

Fig. 1. Thermal diffusivity a of clinker (ρ = 2,1 g / cm^3) as a function of temperature

For the comparison of the laser flash data with the data measured by the heat flow meter apparatus the specific heat c_p and the density r of the specimens must be known. The thermal conductivity l can be calculated in according to $\lambda = a \cdot \rho \cdot c_p$.

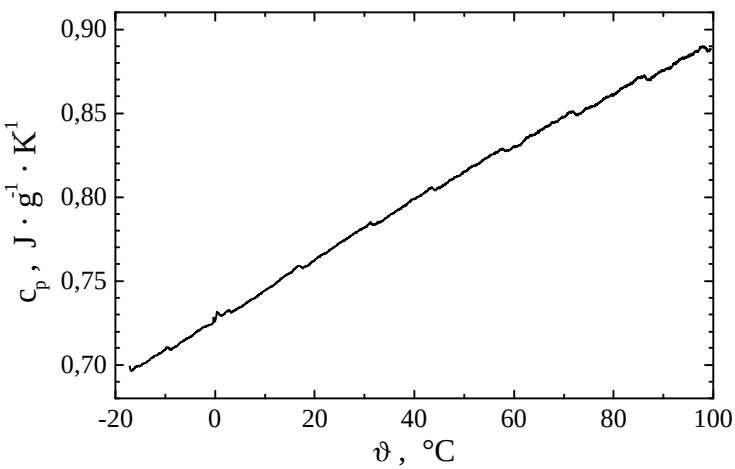

Fig. 2. Specific heat of clinker as a function of temperature

The measurements of the specific heat Fig. 2. have been performed on dry material. For moist material the specific heat was calculated by the caloric mixing rule and the well known specific heat of water. In contrast to a direct measurement of the specific heat of moist material on dry material no desorption of water during the caloric experiment occur and the variation of the pressure inside of the specimen container is negligible. The comparison of the laser flash data with the data from the heat flow meter apparatus (Fig. 3.) shows a satisfactory agreement if one takes into account that the measurements has been performed on different specimens.

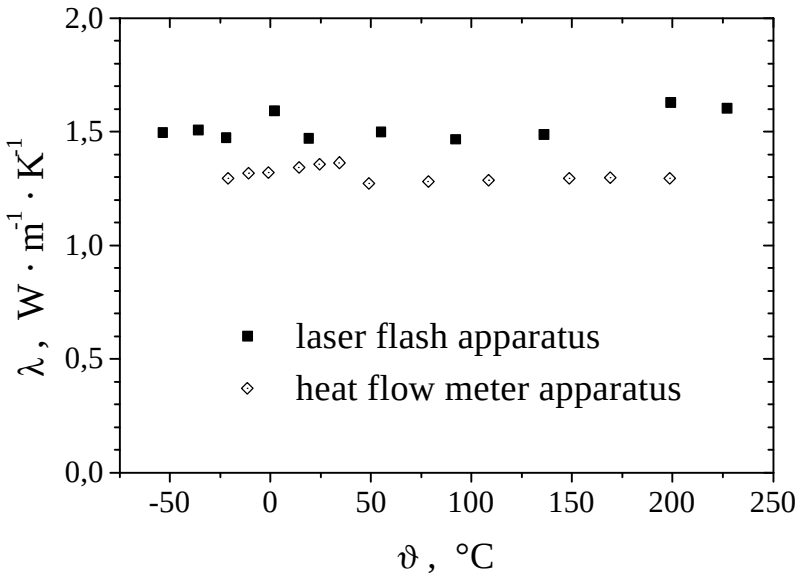

Fig.3. Thermal conductivity of the clinker as a function of temperature, Comparison of laser flash data with the data from the heat flow meter technique

The thermal diffusivity measurement of the moist specimens has been performed by a dynamic method. At the beginning of the experiment the specimens has been moistened up to saturation. The moisture content $\boldsymbol{u_m}$ (moisture content per mass in percent) of the

specimen has been monitored by weighting before and after the laser flash experiments (Fig. 4.). The time interval between the initial weighting of the specimen, three laser flash experiments and the final weighting was 300 s.

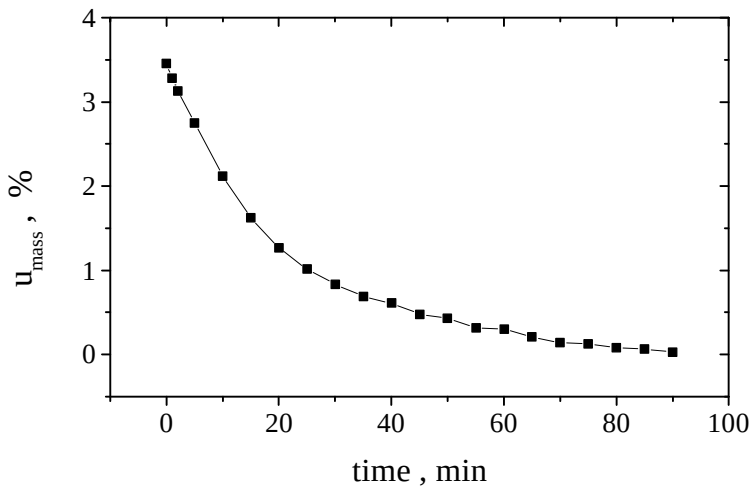

Fig. 4. Moisture content $\boldsymbol{u_m}$ of the clinker specimen as a function of time

The moisture measurements show no influence of the laser flash experiment on the rate of moisture loss of the specimen. It should be noted that the first part of the drying curve (e.g. high moisture content of the specimen) is characterized by a higher speed of drying du / dt. For measurements at temperatures below the freezing point of water the specimen has been moistened at room temperature, shock cooled with liquid nitrogen and inserted into the pre-cooled laser flash apparatus. A drying of the specimen during the measurement at -20°C was not observed.

As can be seen from Fig. 5. the change of the thermal diffusivity of the clinker with the moisture and ice content shows a similar behavior. The difference of the absolute values in the dry state is caused by the temperature dependence of the thermal diffusivity of the clinker (see Fig. 1.).

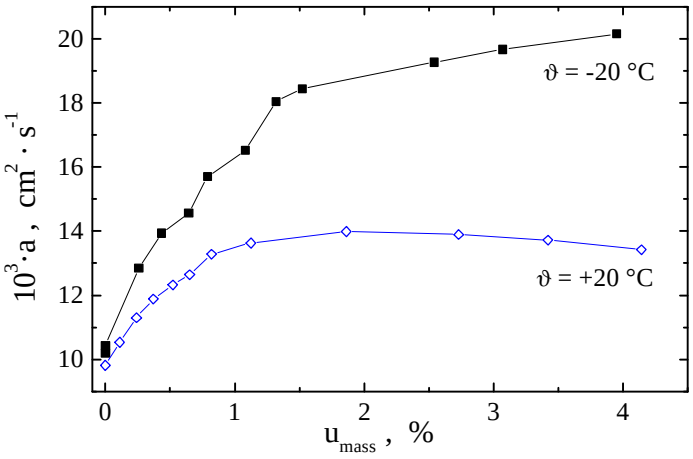

Fig. 5. Thermal diffusivity of clinker as a function of the moisture and the ice content

The comparison of the data from the laser-flash technique with the data from the heat flow meter technique Fig. 6. is shown as a function of the moisture content.

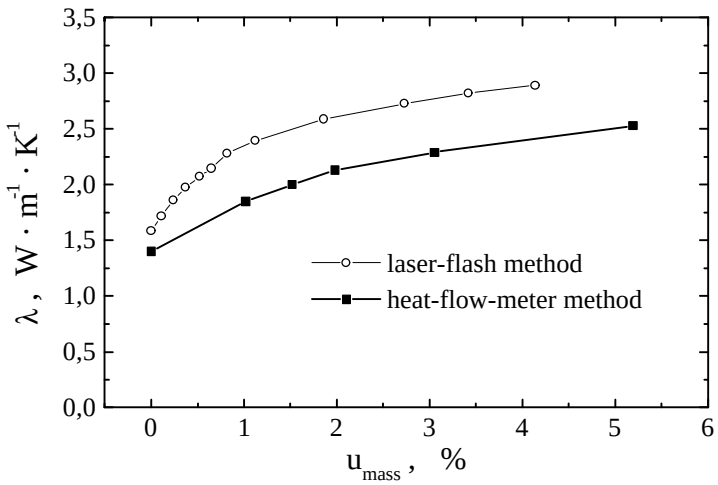

Fig. 6. Thermal conductivity of clinker as a function of the moisture content

For the measurement of the thermal conductivity of the moist specimen by the heat flow meter method the specimen has been enclosed into vapor tight foil after the preparation and during the measurement. Although there is a systematic difference between both techniques again, the slope of both curves shows the same behavior e.g. both techniques deliver the same moisture dependence of the thermal conductivity.

4. CONCLUSIONS

A technique for the measurement of the thermal diffusivity of moist clinker has been presented. The results have been compared with data from measurements of the thermal conductivity by the heat flow meter method and of the specific heat by a Calvet-DSC. The comparison of both techniques shows a satisfactory agreement which is typical for measurements on such systems. The expenditure needed for investigations of this kind could be considerably reduced. It seems the laser flash technique offers various new aspects for a better understanding of heat and moisture transport in such materials. The results are encouraging for further systematic investigations.

4. LITERATURE

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