

## THERMAL CONDUCTIVITY OF GYPSUM PLASTERBOARD BEYOND DEHYDRATION AND ITS CORRELATION WITH THE PORE STRUCTURE

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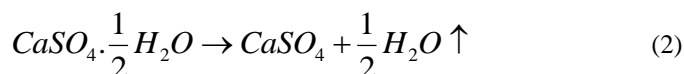
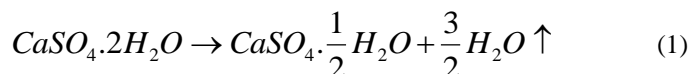
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### ABSTRACT

Gypsum plasterboard is a material used in the building industry for its low weight (porosity 50-65%) and its high resistance to fire due to the endothermic dehydration taking place between 150 and 200°C. Its thermal conductivity which is a decisive thermal property regarding reaction to fire drops by 50% of its initial value after dehydration due to the loss of water (20 mass %) but starts to rise again with rising temperature and reaches its initial value around 750°C. The present study shows that this rise is not due to the increasing radiative or conductive heat transfer but to changes in the bimodal pore structure which leaves the overall structural dimensions nearly unchanged (dilatation of around 2%). Different methods such as mercury intrusion porosimetry, scanning electron microscopy and in-situ X-ray diffraction up to 1000°C were carried out to investigate the correlation between pore structure and thermal conductivity of this material.

### INTRODUCTION

Gypsum plasterboards are used worldwide in the building industry due to the availability of the primary material and the variability of the final products properties [1]. The reaction to elevated temperatures, i.e. in case of fire is a crucial property of gypsum plaster boards responsible for its widespread usage as a protecting material for both light weight wall constructions and steel columns [2]. The main reason this performance is a two-step dehydration procedure induced by the rising temperature during which gypsum loses two molecules of bound water and turns into anhydrite [3]. In case of fire this endothermic reaction absorbs a substantial amount of heat [3] and delays the increase in temperature of the deeper material layer.



The temperature at which this reaction starts and ends depends strongly on the heating rate and vapor pressure [2, 4 and 5] and hence on the type of fire scenario to which the gypsum plaster board is subjected to [6]. The corresponding temperature interval for the dehydration of gypsum plaster board at fire conditions is between 100°C and 200°C.

### NOMENCLATURE

$a$	[m <sup>2</sup> /s]	Thermal diffusivity
$c_{eff}$	[J/kgK]	Effective specific heat capacity
$T$	[K]	Temperature

Special characters

$\rho$	[kg/m <sup>3</sup> ]	density
$\lambda$	[W/mK]	Thermal conductivity

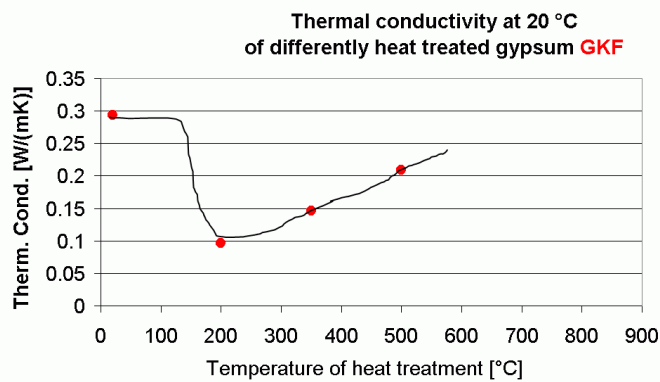
Another effect which influences positively the reaction to fire is the decrease in thermal conductivity by around 50% induced by the loss of water constituting 20% of the original mass. The thermal diffusivity responsible for the movement of the temperature front depends on thermal conductivity, density and the apparent specific heat capacity hence, summarizing the changes these three properties undergo when the gypsum plaster board is exposed to fire.

$$a = \frac{\lambda}{\rho \cdot c_{eff}}$$

As fire temperatures can reach around 1000°C depending on the occurring scenario, the thermal behavior of the dehydrated gypsum plaster board (anhydrite board) is also of basic importance especially when modeling the temperature evolution and predicting time of failure of the gypsum protection. The present paper deals with the thermal conductivity of the dehydrated gypsum up to 500°C and provides some insight into the responsible effects by using different investigation methods.

## THERMAL CONDUCTIVITY

Three gypsum samples of 250mm × 250mm × 10mm were conditioned in an oven at three different temperatures (200, 350 and 500°C). In order to prevent heat shocks to the brittle plates the treatment started with a heating rate of 5°C/min until the prescribed temperature was reached. The sample remained for 24 hours at that constant temperature and was cooled down with the same heating rate. The thermal conductivity of all heat treated samples was measured in a Guarded Hot Plate at ambient temperature (Figure 1). By doing so the contributions of the convective and the radiative heat transfer which increases at elevated temperatures could be excluded.



**Figure 1** Thermal conductivity of gypsum plaster board (GKF) as a function of the heat treatment temperature (measured values in red, the line is only an approximation)

The sharp decrease in the thermal conductivity during the dehydration process is due to the bound water molecules which are good thermal conductors, leaving the crystal in vapour form. The consecutive increase after complete dehydration is due to the enhancement of the conductive heat transfer and hence to be induced by a change in the structure of the dehydrated (anhydrite) gypsum. A similar increase in thermal conductivity has been reported by [7] too.

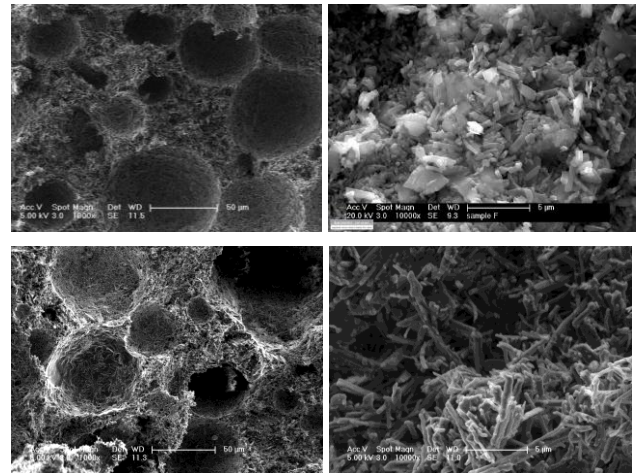
## PORE STRUCTURE

To get a first idea about the pore structure of gypsum and its possible changes due to different heat treatments Scanning Electron Microscopy (SEM) was applied to a number of gypsum samples.

Figure 2 shows two different magnifications of two gypsum samples, on the top the one before and on the bottom the one after complete dehydration at 200°C. The surfaces shown were produced by breaking up prisms of 10mm × 10mm × 50mm.

The investigated gypsum plaster board can be defined as a material with a bimodal porosity. Large spherical and mostly disconnected pores of around 50 microns in diameter are embedded in some kind of loose fill material of crystal needles. The dehydration process does not seem to change the overall structure but to soften the crystal needles edges after bound water escaped out.

In order to get information on the porosity and the pore size distribution of non-heated and dehydrated gypsum, mercury intrusion porosimetry MIP was applied to respective samples of about 10mm<sup>3</sup>. Due to the high brittleness of gypsum at temperatures around and above 200°C MIP could not be carried out on those.



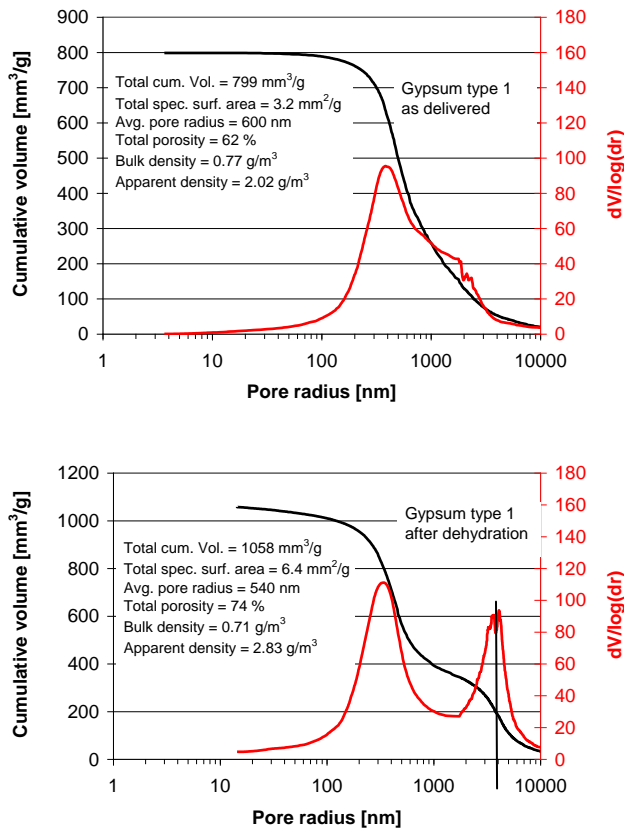
**Figure 2** Structure of gypsum (GKF) by scanning electron microscopy at two magnification levels for a non-heated sample (top) and a sample after complete dehydration at 200°C (bottom)

According to the MIP the total porosity of gypsum changes from 62% before dehydration into 74% after dehydration. These measurements were carried out 3 times and the values averaged. The values for the bulk densities 0.77 g/cm<sup>3</sup> for the unheated/as delivered and 0.71 g/cm<sup>3</sup> for the dehydrated case were determined by using the large samples of the thermal conductivity measurement to have a good average and used as input information for the MIP. The resulting apparent densities of 2.02 g/cm<sup>3</sup> for gypsum CaSO<sub>4</sub>·2H<sub>2</sub>O and 2.82 g/cm<sup>3</sup> for anhydrite CaSO<sub>4</sub> are in good accordance with literature values (2.32 and 2.96 g/cm<sup>3</sup> respectively).

Figure 3 shows the pore size distribution (red) and the cumulative volume accessible to mercury intrusion (black) for an unheated sample (top) and a completely dehydrated one (bottom). It is interesting to note that the cumulative volume reaches zero already at a pore radius of 10 microns for the non-heated as well as for the dehydrated sample. This means that MIP is not able to detect the large pores visible in the SEM images on the left of Figure 2. The reason is the ink-bottle

effect occurring due to the disconnectedness of these large pores which are only accessible for the penetrating mercury by small ink-bottle-neck type pores [8]. Their necks are around 0.4 microns for the non-heated as can be seen at the top of Figure 3. The dehydration process leading to the loss of the bound water molecules seems to enable a large number of them to coalesce to wider ink-bottle-neck openings of around 4 microns visible as a second peak at the bottom of Figure 3.

As already mentioned the MIP is not applicable to the highly brittle gypsum samples which endured a heat treatment beyond 200°C due to their implosion caused by the mercury pressure.



**Figure 3** Pore size distribution of gypsum by mercury intrusion porosimetry non-heated (top) after complete dehydration (bottom)

For temperatures between 200°C and around 550°C there are no chemical reactions occurring according to the thermo gravimetric analysis (TGA) and differential scanning calorimetry (DSC) carried out on different gypsum board materials as stated in earlier publications [2,3]. Beyond 550°C disintegration of some ingredients such as  $MgCO_3$  or  $CaCO_3$  will occur but this is not the topic of this paper.

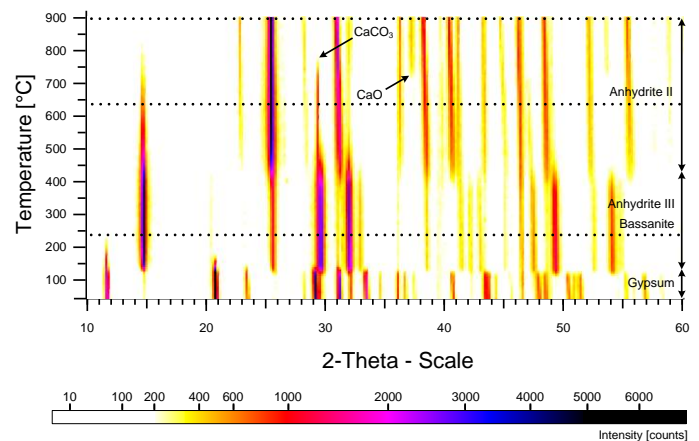
Nevertheless, it has been reported [7] that a weakly exothermic reaction takes place around 450°C which is attributed to the transition of anhydrite III (soluble) to Anhydrite II (nearly insoluble). There is a change in the crystalline structure assigned to this transition according to [9]

and [10]. A non-destructing method which would be able to distinguish between these two phases is X-ray diffraction.

## X-RAY DIFFRACTION (XRD)

Figure 4 shows the X-ray diffraction (XRD) analysis of gypsum as a function of temperature. On the X-axis is the diffraction angle at which the reflexes of the different minerals are detected. Typical for XRD-measurements is that a pattern of one substance shows reflexes at many different angles. On the y-axis is the temperature range of the measurements reaching from 40 °C to 900°C. The intensities are shown by different colours. There are several phase changes happening in the gypsum while heating up. This becomes obvious in the sudden signal changes at 120 °C and 440 °C. The initial sample consists mainly of gypsum  $CaSO_4 \cdot 2H_2O$  and a small amount of  $CaCO_3$ . Gypsum converts to  $CaSO_4 \cdot 0.5H_2O$  (Bassanite) at 120 °C which is subsequently dehydrated to  $CaSO_4$  anhydrite III. These phases are almost the same and therefore only a minor signal change happens. Anhydrite III is then converted to anhydrite II at 440 °C. For more clarity, the temperature range for the different chemical phases is indicated by the arrows on the right hand side.

Differences between the measured reflexes and the position of the pattern are due to the fact that the database patterns were measured at room temperature. As anisotropic thermal expansion takes place during heating to 900 °C, a shift of certain signals to lower angles, which means a larger inter atomic distance, is unavoidable.



**Figure 4** 2D-plot of the X-ray diffraction analysis of Gypsum (GKF) as a function of temperature

The transition from anhydrite III to anhydrite II occurs in a narrow temperature interval around 440°C and hence cannot be responsible for a steady increase of thermal conductivity from 200°C up to 500°C and presumably beyond as indicated in Figure 1.

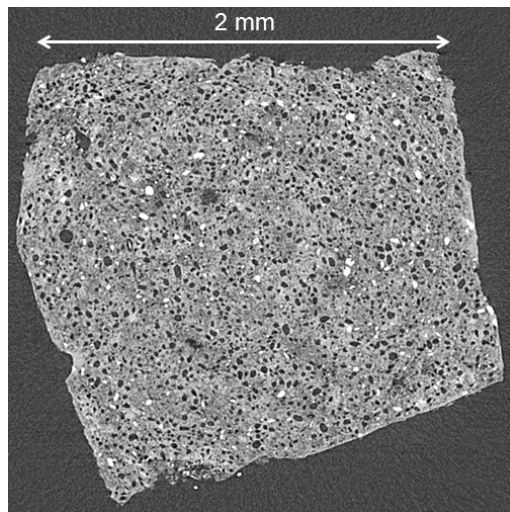
## CONCLUSIONS

An increase in the thermal conductivity of differently heat treated plaster board samples between 200°C and 500°C was measured which is attributable to the conduction heat transfer only. To understand this phenomenon the structure of gypsum plaster board was investigated by different methods (SEM, MIP, XRD). Based on these findings it can be concluded that the mentioned increase in the thermal conductivity is not due to the transition of anhydrite III into anhydrite II but most probably due to a kind of “sintering” process taking place in the dehydrated gypsum plaster board which enhances the thermal contact between the single anhydrite crystals. To verify this, the structural behaviour of dehydrated gypsum and mainly the changes in the pore size distribution should be investigated between 200°C and 1000°C by a quantitative method. MIP is not appropriate due to both the ink-bottle effect which neglects the large pores and the brittleness of the material with rising temperature.

## OUTLOOK

X-ray tomography is a non destructive method to determine the size and the distribution of the large pores. By using a heating device which brings the whole probe to around 1000°C the evolution of these pores within the structure can be imaged and the anticipated “sintering” eventually confirmed.

Figure 5 shows a partial result of a preliminary test at the TOMCAT beam line of the PSI on a non-heated gypsum specimen of approximately 2mm × 2mm × 2mm. The large pores (dark) invisible for MIP are clearly visible here. White dots are additives to the plasterboard such as clay particles etc.



**Figure 5** Horizontal cross section of tomographic scan of gypsum made at the TOMCAT beam line at PSI using the UPLAPO4x objective (beam energy: 32keV)

X-ray tomography measurements during heating-up of gypsum samples to 1000°C have recently been carried out at

the TOMCAT beam line of the PSI using a laser heating device and the results will soon be processed by imaging software, analysed and published.

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