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Expanded glass in insulating monolithic refractories

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Abstract

Expanded glass has been in use for a wide variety of products as in dry mortar products, panels, lightweight concrete and fills. The following approach investigates the applicability of expanded glass produced by the company Dennert Poraver in an aluminate cement matrix for insulation monolithic refractories according to EN ISO 1927-1:2012. To examine the capability, the material was fired to certain temperatures. Mechanical properties were tested on standard beams before and after the heating. The chemical composition and mineral phases of the system were determined by XRF and XRD. To estimate the properties as insulating material, the thermal conductivity of the materials was investigated. Finally, the behaviour of the material during the heating process was monitored by heating microscopy.

Keywords: Expanded glass, Aluminate cement, Insulating, Monolithic, Refractory

SZKŁO EKSPANDOWANE W IZOLACYJNYCH MONOLITYCZNYCH MATERIAŁACH OGNIOTRWAŁYCH

Szkło ekspandowane jest wykorzystywane w szerokiej gamie produktów takich jak wyroby na bazie suchych zapraw, panele, lekkie betony i wypełniacze. Niniejsza praca bada zastosowanie szkła ekspandowanego wyprodukowanego przez Dennert Poraver w matrycy cementu glinowego przeznaczonej na ogniotrwałe izolacje monolityczne wg normy EN ISO 1927-1:2012. Aby zbadać tę zdolność, materiał był wypalany w pewnych temperaturach. Właściwości mechaniczne badano wykorzystując standardowe belki przed i po wypaleniu. Skład chemiczny i fazy mineralne układu zostały oznaczone za pomocą XRF oraz XRD. Aby ocenić właściwości izolacyjne, zbadano przewodność cieplną materiałów. Na koniec zbadano zachowanie materiału podczas procesu ogrzewania za pomocą mikroskopii grzewczej.

Słowa kluczowe: szkło ekspandowane, cement glinowy, izolowanie, monolit, materiał ogniotrwały

1. Introduction

In terms of energy management, insulation properties of temperature resistant materials are necessary to lower production costs e.g. for metal production. As aggregates in monolithic insulating refractories, ESCS (expanded shale, clay, slag) in combination with vermiculite are used widely [1-2]. For the highest demands, hollow ceramic spheres are typical. It is known that expanded glass offers high insulating properties for common building materials [3]. Optimization of a sieve line is possible with different available grain sizes. In addition with the mechanically stable grains and their spherical shape, the workability is positively influenced. One of the major problems of glass is the relatively low temperature before it softens at the glass transition (Tg).

This work is therefore focusing on the determination of the maximum applicable temperature in an aluminate cement matrix. According to EN ISO 1927 one key criteria is that the shrinkage of the material must not exceed 1.5% for monolithic insulating refractories fired to a distinct temperature. Application temperatures are depicted in Table 1. Beyond that, it is a standard for the declination of the material (e.g. based on type of product), method of placement and raw material base [4].

Table 1. Temperature classification of insulating monolithic refractories [4].

Application temperature
< 900°C not applicable
900°C
1000°C
1100°C
…
> 1700°C

2. Experimental

For this investigation beams 16 cm × 4 cm × 4 cm in size were produced using the aluminate cement Istra 40 (Co. Calucem) as binder, and a standard low Al_2O_3 chamotte and Poraver expanded glass (Co. Dennert Poraver) as aggregates. The expanded glass is made out of post-consumer recycled soda-lime-silicate glass. The water content was kept to a minimum to achieve a castable material by vibration without any plasticizing admixtures. A small amount of short cut polypropylene fibres (PB Eurofiber, 12 mm) were incorporated to minimize vapour pressure of water during heating as they are molten at 165°C and offer free capillary structures. The formulation is shown in Table 2 and chemical composition of aluminate cement Istra and Poraver expanded glass in Table 3 and 4, respectively.

2.1. Heating process

Before the firing process was started, the material was hardened for 24 h at room temperature and pre-dried for additional 24 h at 110°C. The specimens were heated up to a distinct temperature shown in Fig. 1 exemplary for 1000°C. The heating rate was 3 K/min. The temperature was hold for 2 hours at 400°C to completely desorb physical water. To guarantee a homogeneous temperature inside the material, it was heated for 4 hours at the temperature of interest.

2.2. Determination of properties

After slow cooling, to avoid crack formation, the mechanical properties of the specimens were investigated using a flexural and compressive strength testing device (Co. Form + Test). The thermal conductivity was measured with a Hot Disk M1 thermal analyser (Co. Hot disk) on the cooled specimen at room temperature.

Analysis of the chemical composition of the fired specimen was carried out by XRF. Mineral phases were determined by the Rietveld method (XRD) in cooperation with the Company Dorfner Anzaplan.

2.3. Heating microscopy

To determine the behaviour of pure expanded glass in aluminate cement matrix during the firing process, samples with only the Poraver expanded glass 0.1-0.3 mm as aggregate at 45 vol.% of total mix were investigated by heating microscopy. In a heating microscope (Co. Hesse Instruments) the shadow pane of the specimen is detected while heating. Therefore small cubes (5 mm × 5 mm × 5 mm) were prepared by sawing and grinding from the material pre-dried at 110°C. The small dimension of the samples made it necessary to use the fine Poraver grain size to ensure a homogenous specimen. The material was heated with 50 K/min up to 1350°C.

3. Results

Preliminary tests indicated the softening of the system between 1200°C and 1300°C with high shrinkage of it, beginning above 1100°C. Thus, the main investigation was for a temperature of 1000°C.

3.1. Chemical and mineral composition

Table 5 depicts the chemical composition in wt.% of the investigated formulation measured by XRF (DIN EN ISO 12677 2013-02).

Focusing on the ternary SiO_2-CaO-Al_2O_3 system without taking other components into account the main composition is by parts in weight: 35% SiO_2, 35% Al_2O_3 and 30% CaO. The area is illustrated in Fig. 2. lying between Anorthit and Gehlenite.

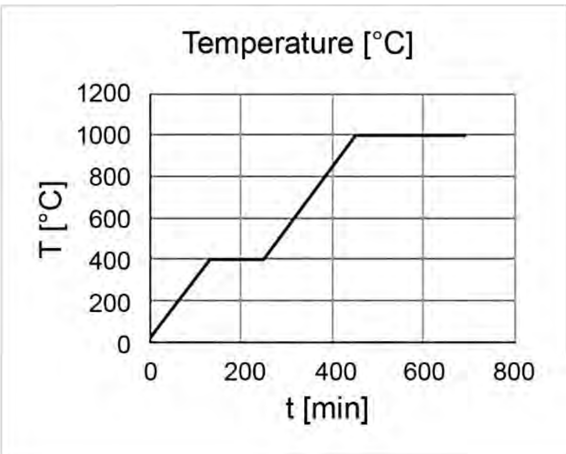

Fig. 1. Heating curve of specimen fired to 1000°C.

Table 2. Investigated formulation.

	Parts per weight [%]
Binder	
Aluminate cement	50.1
Aggregates	
Chamotte 0 - 0.2 mm	6.8
Chamotte 0 - 1 mm	11.3
Poraver® 0.5 – 1 mm	1.4
Poraver® 1 - 2 mm	6.5
Poraver® 2 - 4 mm	5.3
PP fiber	0.1
Water	18.8
Total	100
w/c ratio	0.37

Table 3. Chemical composition of aluminate Cement Istra [5].

Component	Content [wt.%]
SiO_2	≤ 6
Al_2O_3	38 - 42
Fe_2O_3	13 - 17
CaO	36 - 40
MgO	< 1.5
SO_3	<0.4

Table 4. Chemical composition of Poraver expanded glass [6].

Component	Content [wt.%]
SiO_2	70 - 75
CaO	7 - 11
MgO	0 - 5
Na_2O	10 - 15
K_2O	0 - 4
Al_2O_3	0.5 - 5

The XRD measurement was carried out on fired material. The test (DIN EN ISO 13925 2003-07/ Rietveld) Table 6 approved a high content of Gehlenite. Further high parts of calcium aluminate based on the cement and high part of others, mainly expected to be amorphous phase by the molten glass were detected.

3.2. Physical properties

As shown in Table 7, a typical decrease of the mechanical properties caused by heating took place. This is expectably due to the destruction of the hydrate phases between 400-900°C [6]. The ceramic transition over 900°C is not expected to be strongly developed while heating to a temperature of 1000°C for 4 hours. The shrinkage of the material was lower than 1.5% and met the requirements according EN ISO 1927. The measured thermal conductivity is ranging at values typical for lightweight concrete of this density [7].

During the heating, the glass is molten, coating spherical pores in the matrix. Picture Fig. 3 shows the fracture plane before and after firing. The melt partially infiltrated the matrix. After cooling, a thin glass lining of the pores remained forming a stable structure.

3.3. Heating microscopy

Figs. 4 and 5 display the material performance during the examination in the heating microscope. Starting at 709°C, an expansion of the glass grains is visible for particles on the outer surface which were exposed by the preparation of the specimen. This indicates that the glass softening is proceeding. A maximum expansion is visible at 823°C. At 952°C, the expansion is completely decreased and the glass is expected to be molten. Until 1200°C the complete system remains almost stable. Afterwards there is a softening of the complete material and at 1330°C the transition into molten state took place.

4. Conclusion

Expanded glass was investigated in an aluminate cement system. Despite the generally low softening point of glass, a specimen with a shrinkage of 0.8% could be manufactured for heating up to 1000°C. The thermal conductivity was lying in the expected range for this density. Compared to other expanded minerals for insulating refractories like

Table 5. Chemical composition of specimen.

Parameter	Unfired beam	Fired up to 1,000°C
	[wt.%]	[wt.%]
SiO_2	26.3	29.9
Al_2O_3	26.6	30.1
Fe_2O_3	8.59	9.55
TiO_2	1.15	1.28
K_2O	0.77	0.85
Na_2O	2.12	2.43
CaO	22.1	24.7
MgO	0.6	0.65
PbO	< 0.01	< 0.01
BaO	0.04	0.05
SO_3	< 0.01	< 0.01
MnO	0.04	0.05
P_2O_5	0.06	0.07
ZrO_2	0.04	0.05
LOI (1025°C)	11.3	0.3
Loss of water 105°C	0,5	0

Table 6. Mineral composition of specimen fired up to 1000°C.

Mineral phase	Content [wt.%]
Mullite $Al_6Si_2O_{13}$	3.5
Gehlenite $Ca_2Al_2SiO_7$	17
Quarz SiO_2	6
Heamatite Fe_2O_3	3.5
Wollastonite	6
CA $CaAl_2O_4$	19
Grossite $CaAl_4O7$	13
Brownmillerite Ca_2FeAlO_5	7
Other	25

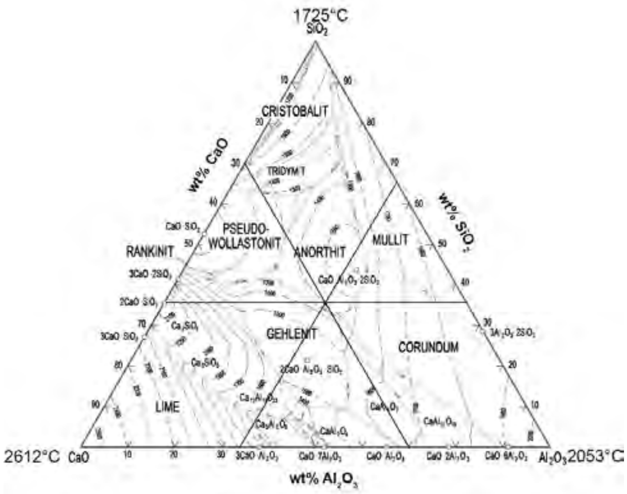

Fig. 2. Ternary system SiO_2-CaO-Al_2O_3 [5].

Table 7. Physical properties of specimen before and after firing up to 1000°C.

Specimen	Density [g/cm³]	Cold flexural strength [N/mm²]	Cold compressive strength [N/mm²]	Thermal conductivity [W/(m·K)]	Linear shrinkage [%]
Unfired	1.34 (dried at 110°C)	3.5 (24 h)	21,0 (24 h)	0.46	-
Fired to 1000°C	1.1	3.1	13.0	0.36	0.8

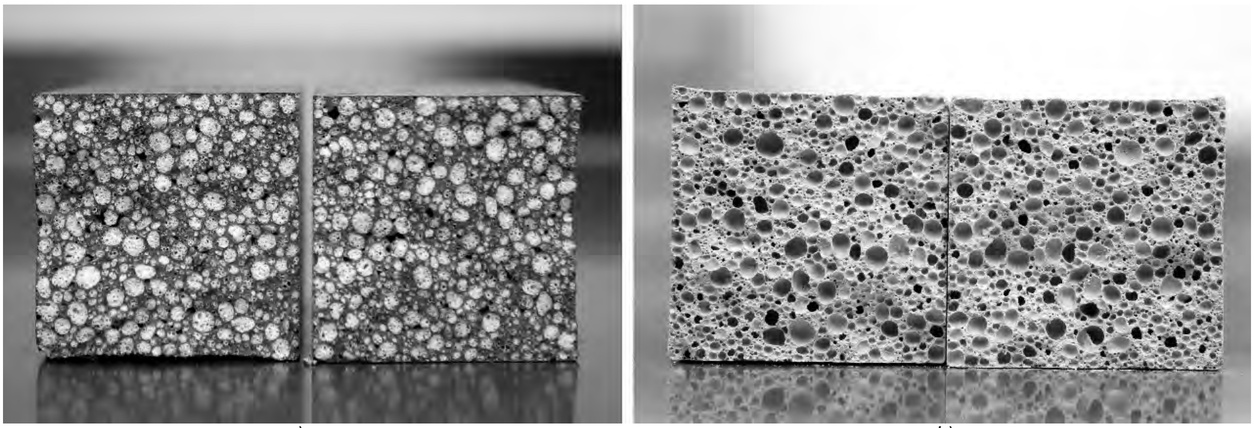

Fig. 3. Comparison of the fracture plane of unheated (a) and up to 1000°C fired (b) material.

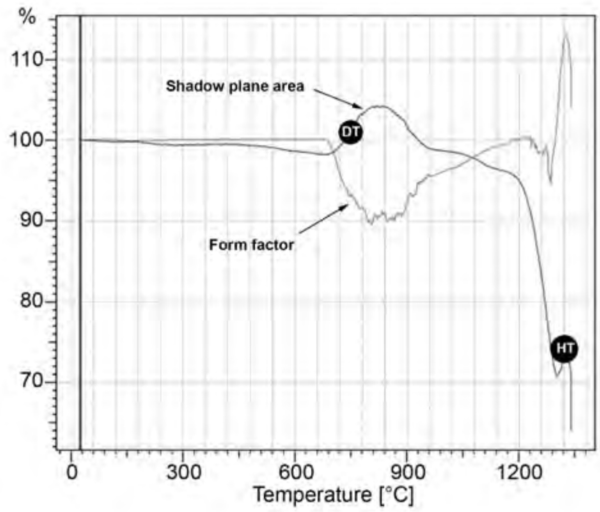

Fig. 4. Heating microscope plot illustrating the relative development of the shadow plane area.

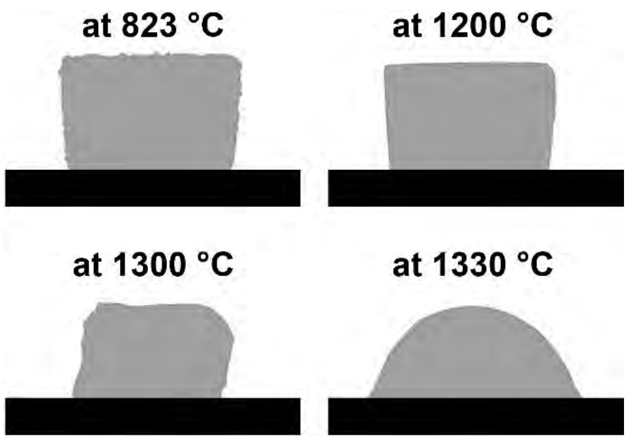

Fig. 5. Monitored pictures at certain temperatures in the heating microscope.

vermiculite, the expanded glass has an improved workability in the mixture due to its stable spherical grains. With the closed surface of the lightweight aggregates the water content of the formulation could be adjusted to a low level what is beneficial against crack formation during heating. Above temperatures of approx. 950°C, the glass is expected to be completely molten. Lining the voids with molten glass a stable closed spherical pore structure was formed. The stable structure is expected to have a positive effects on mechanical cold strength.

Acknowledgement

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