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Response of Refractory Cement Based Composite to Gradual Temperature Loading

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RESEARCH ARTICLE

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Abstract

This paper deals with the experimental study of the response of refractory concrete mixture to gradual thermal loading, up to 1000 °C. A binding system based on calcium aluminate cement (CAC) modified by the partial replacement of metakaolin was used. Short ceramic fibers were applied in a dose of 4% by volume in the studied mixture. Material transformations due to thermal loading were monitored in terms of residual mechanical, fracture and basic physical properties-compressive strength, flexural strength, fracture energy, dynamic modulus of elasticity, and bulk density in the study. The results obtained corresponded well with the mineral transformations monitored using the thermogravimetric analysis performed on the binder paste. Residual values of compressive and flexural strength were approximately 40% of the initial values after exposure to a temperature of 1000°C; however, the dominant part of the total loss was monitored up to 400°C, due to decomposition of the hydrates. Fiber employment contributed considerably to the resistance against thermal loading up to 400°C, which was demonstrated by the fracture energy results (a loss of 25% was monitored). An additional increase of the temperature load led to micro crack propagation, which was obvious in the results of dynamic modulus of elasticity determination, in which the short fiber incorporation was noticeably limited.

Keywords

refractory composite, metakaolin, ceramic fibres, elevated temperatures

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1 Introduction

By refractories are usually presented as high utility composite materials, originally developed for industrial, technological, safety, or other special purposes, when the elevated temperatures take place. According to the location of the application, they protect various types of constructions, technologies, or how they can be used, e.g. for manufacture of monolithic furnaces in industrial production [1]. Due to the application in the environment with extreme conditions, the refractories consist of high utility and high quality raw materials, which are often expensive and very energy-intensive. Refractory materials can be characterized according to type of bond that provides a solid link between fillers and micro-fillers. Three main types of bonds in refractory composites are hydraulic bonds (created by hydration of aluminous cement), chemical bonds (replacement of aluminous cement by hydrated and reactive aluminium oxide), and ceramic bonds (formed by sintering filler and binder after an initial exposure up to 1000 °C) [2, 3]. Ceramic refractories exhibit the best resistance to increased temperatures, meaning that their testing is very laborious and instrumentally difficult. That is why, numerical instruments were developed to predict temperature dependent material changes [4, 5].

We can also classify refractories based on the method of production (shaped or unshaped). High-temperature refractory concrete, classified by their cement content, can be divided into conventional castables (CC), low cement castables (LCC), and ultra-low cement castables (ULCC) [6]. CC usually contains contents of more than 20% of CAC (calcium aluminate cement), LCC 6–15 %, and ULCC of less than 6% of CAC [7]. Several conventional castables can be characterized as self-flow [8] with similar workability parameters, as is to be expected for self-compacting concrete.

The interaction of building materials and an environment with elevated temperatures is usually connected with degradation, microstructure, chemical changes, a decrease of mechanical properties, and volume changes, etc. Every building material is characterized according to its fire safety (ability to resist fire), usually classified in standards [9]. Concrete, the most widespread building material in the world, has

satisfactory fire-resistance properties (compared with steel), that provides sufficient time for safe evacuation (tens of minutes). High strength concrete achieves lower fire-resistance, due to the explosive spalling phenomenon [10]. The explosive spalling took place between 300°C and 400°C, and it is caused by an increase of pressure in the pore structure of HPC (due to the volume expansion of water in pores). Composites based on Portland cement characterizes the gradual decomposition of CSH; at 400°C the portlandite $Ca(OH)_2$ is decomposed to quick-lime and CO_2. This process is accompanied by a decrease of mechanical properties, macroscopic cracks, and disintegration composite´s structure.

The experimental program, presented in this article, is based on previous research, focused on the experimental investigation of an optimal composition of a refractory calcium aluminate cement-based composite with an addition of metakaolin, what means refractory composite with hydraulic bond. Several ratios between metakaolin and calcium aluminous cement were analysed. On the basis of values of fracture and mechanical characteristics, and also workability, the optimal modification of the basic mixture formula consisted of 4% volume fraction of ceramic fibres and 20 % (of aluminous cement weight) of metakaolin. The process of the optimal composite composition development was published in [11]. Natural basalt aggregate was used in the described composite; therefore, the maximum temperature level was set at 1000°C.

The main goal of this study was to describe the gradual changes of refractory cement composite previously developed for high temperature applications. Several changes are connected with the evaporation of free and physically bounded water, gradual decomposition of hydration products, and the mineral transformation of used aggregate. These changes influenced the basic and mechanical properties; therefore, all parameters (bulk density, flexural strength, compressive strength, dynamic modulus of elasticity, and fracture energy) were investigated after the specimen´s exposure to various temperature loading levels (105°C, 200°, 300°C, …, 900°C and 1000°C) for 180 minutes).

2 Materials

2.1 Composite composition

The aim of this research was to determine and quantify gradual changes of basic and mechanical properties of refractory cement composite reinforced by ceramic (alumina-silicate) fibers due to temperatures loading from 100°C to 1000°C (in 100°C gradient). The composition of the studied composite (see Table 1) was based on previous research, in which the optimized mixture was researched. The total amount of fibers, aluminous cement, and metakaolin determined the optimal solution, which was 180kg/m^3 of metakaolin, 720kg/m^3 of aluminous cement, and 4% of volume of fibers. Superplasticizer Sika SVC1035 ensured a low water-binder ratio (0.25) and self-flow properties. The influence of elevated temperature on poly-carboxylate superplasticizer SVC had been investigated in [12], and with no negative implication detected. In this research, the various amounts of SVC were applied on the composite, and the mechanical properties were measured before and after exposure to 600°C and 1000°C. The mixing procedure took place in a horizontal laboratory mixing machine; the first 5-minute phase consisted of homogenization of aggregates and fine components (cement and metakaolin), 50% of water was added in the second phase (1 minute), the remainder of the water with the plasticizer was added in the third phase (2 minutes), and the last phase consisted of adding the full dosage of fibers and their homogenization in composite (2 minutes). This fresh mixture was embedded into the steel moulds, and specimens 40 × 40 × 160 mm were manufactured.

2.2 Basalt aggregate

According to the maximum temperature of application, the optimal type of aggregate, or filler, was chosen. For a technical application to 700°C, the natural aggregate is suitable, but only from selected types of rock (basalt, andesite, and diabase) [13]. For example, silica aggregates are unacceptable, due to volume expansion (approximately 0.5%) at 573°C, when α-silica is change to β-silica [14]. Granite has the opposite problem, shrinkage of volume due elevated temperatures. Alternativelly could be used artificial aggregates for application up to 700°C (usually chamotte fireclay, bauxite, chromite, corundum, carborundum, slag, and electro-porcelain) [15]. The main filler of investigated cement composite was a combination of two fractions (0/4 mm and 2/5 mm) of natural crushed basalt aggregated, also used in [13]. High utility properties of natural basalt could be used for specific severe applications [16].

2.3 High alumina cement

The well-known, and thoroughly described, phenomenon of conversion [17] eliminates the application of aluminous cement only for the special purposes of refractory composites, but not for load-bearing structures. The properties of conventional castables (CC) based on hydraulic bond are mostly dependent on the qualities and purity of the aluminous cement that was utilized. Aluminous cements are classified according to the total amount of Al_2O_3, and various studies

Table 1 Composition of used refractory composite

Ceramic fibers [kg/m^3]	Basalt aggregate [kg/m^3]		Fine components [kg/m^3]		Liquid components [kg/m^3]	
4% by volume	0/4 mm	2/5 mm	Cement Secar®71	Metakaolin MefistoL05	Plasticizer Sika SVC 1035	Potable water
80	880	220	720	180	22.75	224

have described the dependence of Al_2O_3 and suitable temperature ranges for their application [18]. The aluminous cement Secar71 used contained approximately 71% of Al_2O_3; therefore, it was suitable for utilization as a binder up to 1600°C. Table 2 shows the detailed chemical composition of used cement. The specific surface of the cement utilized, measured by a Blaine apparatus, achieved 381m²/kg. Refractory cement composites achieved the lowest strength in the range 800°C – 900°C; while the CAH was finally decomposed, although a ceramic bond was still not formed [19].

2.4 Metakaolin

Active pozzolanic materials (zeolite, metakaolin, ceramic powder, etc.) have a significant role in concrete and composite technology; this phenomenon has been confirmed by various studies focused on various topics [20, 21]. We can find several successful applications of metakaolin in the field of refractory cement composites [22]. An amount of 180kg/m³ of metakaolin MefistoL05 from České lupkové závody, a.s., Czech Republic, with a specific surface of 306 m²/kg was used for the purpose of this experimental program. Metakaolin is produced by controlled calcining of raw clay, at approximately at 800°C. The calcination temperature and quality of the input clay determine the metakaolin´s properties.

2.5 Fibers

Granulated alumino-silicate fibers ISOWAT 12G, with a maximal application temperature 1260°C were used for reinforcement. These fibers contain a bulk density of 200 kg/m3, with an average length ranging from 100 to 200 μm, and an average diameter of 10 μm. The surface treatment significantly influences behaviour during elevated temperature, because it provides the cohesion between fiber and the composite´s matrix [23]. These types of fibers are used even for ordinary concrete, when 0.2% of volume improved the dynamic characteristics of ordinary reinforced concrete [24]. However, ceramic fibres could be successfully used also for ceramic refractories for severe applications [25].

Table 2 Chemical composition of fibers, cement, and metakaolin (% of weight)

Components	Secar	MefistoL05	Fibers
Al_2O_3	70.8	41.9	44.0
SiO_2	0.58	52.9	53.7
K_2O	-	-	0.20
CaO	27.5	0.13	0.22
TiO_2	0.37	1.8	0.60
Fe_2O_3	0.42	1.08	0.66
SrO_2	-	-	0.01
ZrO_2	-	-	0.57
MgO	0.21	0.18	-

3 Used procedures

3.1 Thermal loading

Manufactured specimens were preconditioned by the drying process up to 105°C to suppress explosive spalling during thermal loading. Sets of samples were subjected to thermal loading using an automatic electrical furnace at a heating rate of 10°C/min, with the exception of one control set for each studied mixture. The level of thermal loading was gradually increased by units of 100°C up to 1000°C. The required thermal level was kept for an additional three hours, after which all samples were simultaneously cooled down to the room temperature. The process of thermal loading is illustrated on Fig. 1.

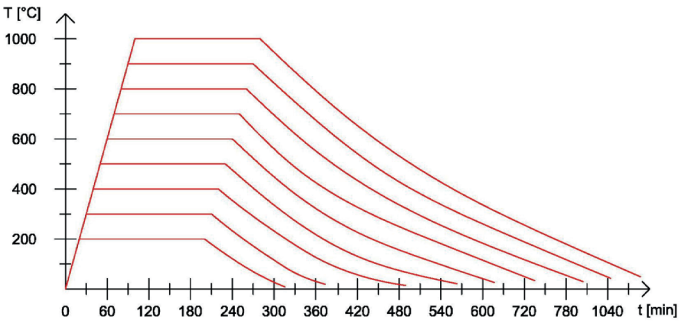

Fig. 1 Thermal loading cycles

3.2 Investigated parameters

Bulk density, flexural strength, compressive strength, and the dynamic modulus of elasticity were investigated on both the thermally loaded sets of samples and the control sets (dried to 105°C). Mechanical testing was performed perpendicularly to the direction of compacting, which is a standard arrangement for cement-based composites. Each set consisted of three prismatic specimens.

Bulk density (ρ_m) was determined by the base of the actual weigh dimensions of the specimens. Mineralogical transformations are closely related to the values of the bulk density [26].

Flexural strength (f_{tm}) determination was organized as a three-point bending test, with a support span of 100 mm, according to [27], where f_{tm} is calculated as a critical tensile stress of the rupture, using the theory of elasticity (1). A universal loading machine (MTS 100) and the operating system TestWorks4 were used to control the test by the deformation rate of 0.2 mm/min. Deflection of the samples was recorded by the couple of extensometers.

Compressive strength (f_{cm}) was determined using two fragments left after the flexural test. The loaded area of the samples was 40 × 40 mm². The value of f_{cm} was calculated according to [27] (2).

$$f_{tm} = \frac{3F_{t,max}}{2hb^2}, \quad (1)$$

$$f_{cm} = \frac{F_{c,max}}{bh}, \quad (2)$$

f_{cm} – compressive strength [MPa],
$F_{c,max}$ – maximal reached force during compression test [N],
b – width of the sample [mm],
h – height of the sample [mm],
f_{tm} – flexural strength [MPa],
$F_{t,max}$ - maximal reached force during flexural test [N].

The dynamic modulus (E_{cum}) of elasticity was measured using the ultrasonic pulse method, which presented the most frequently used non-destructive technique. The ultrasound speed (v_L), by the coupling of 54 kHz transducers and Pundit-Lab+ velocity test instruments produced by the Proceq company, was determined. The final values of the dynamic modulus of elasticity was then calculated according to (3).

$$E_{cum} = v_L^2 \cdot \rho_v \cdot \frac{1}{k^2} \cdot 10^{-6} \tag{3}$$

E_{cum} - dynamic modulus of elasticity [GPa],
v_L – velocity of the impulse [m/s],
k – coefficient of sample sizing [-],
ρ_v – bulk density [kg/m^3].

From the recording of the flexural strength determination, the fracture energy Gfm [J·m^{-2}] was calculated. For the determination of fracture energy, the formula given in RILEM recommendation was used [28] (4).

$$G_{fm} = \frac{1}{b \cdot (h-n)} \int_0^{\delta_{max}} F_{t,max}(\delta)\, d\delta \tag{4}$$

G_{fm} - fracture energy [J/m^2],
$F_{t,max}$ is force at failure in [N],
δ is deflection in mm, a is width of the beam in [m],
n is depth of the notch in [m].

3.3 Thermogravimetric analysis

Prismatic samples 40 × 40 × 160 mm were prepared for the thermogravimetric analysis by mixing used cement, with 25 % replacement of metakaolin and a water/binder ratio 0.25, which corresponds with the composite composition. Paste specimens were cured in similar condition as composite samples. They were dried at 105°C, and homogenized in the laboratory mill after 28 days of aging. Thermogravimetric measurement was performed on approximately 50 mg of the resulting powder by the monitoring of weight loss using temperature rate 10°C/min. The simultaneously applied DTA-TG apparatus (Schimadzu DTG-60H) used aerial condition in the measuring chamber. Three samples were measured for each studied material. Weight loss was monitored for specific temperature intervals matching the thermal loading levels.

4 Results and discussion

The gradual decline of bulk density value, due to the action of elevated temperatures and consequent transformations, can be observed from the chart in Fig. 2. The initial part of thermal loading is connected with the evaporation of both free and physically bounded water. The highest decline of bulk density took place in the temperature ranges of 300°C and 500°C, which is in accordance with the decomposition of the hydration products, which resulted in a gradual decrease of bulk density. The final value of bulk density at 1000°C is 6.8%, and less than at 100°C.

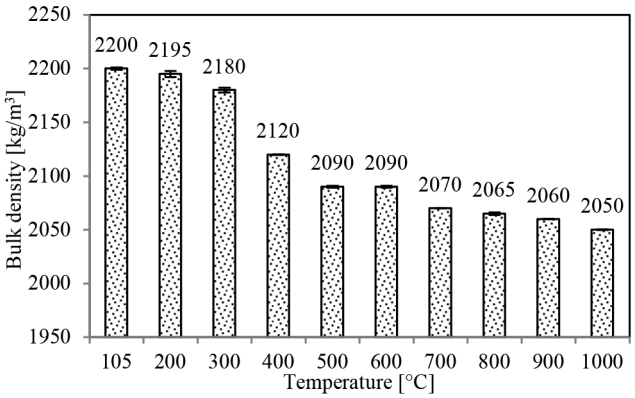

Fig. 2 Evaluation of bulk density

Compressive strength (Fig. 3) declined equally linearly due to the action of thermal loading with increasing temperature. The compressive strength of the specimen exposed to 300°C is at a 75.8% level of the reference value (105°C), 600°C corresponds to 61.2%, and 1000°C corresponds to 45% of the reference value.

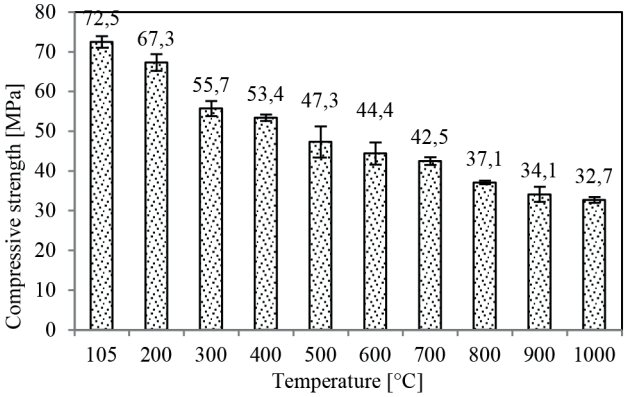

Fig. 3 Evaluation of compressive strength

A considerable decrease of flexural strength (Fig. 4) was monitored at 300°C (76.2% of origin value), after exposure to 400°C compressive strength declined to 55.4% of origin value, and the final compressive strength after exposure to 1000°C measured at the 41.5% level. The dynamic modulus of elasticity (Fig. 5) is also dependent on the bulk density; therefore, the trend follows bulk density values. The highest decline of fracture energy (Fig. 6) was realized between 200°C and 500°C (to 66.7% of original value). Further decline was successive without sudden changes.

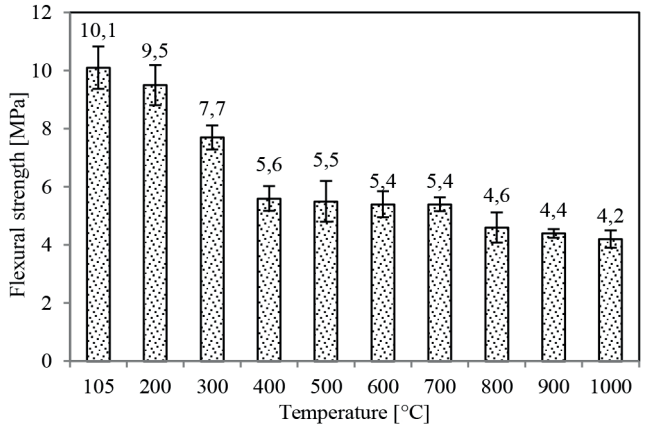

Fig. 4 Evaluation of flexural strength

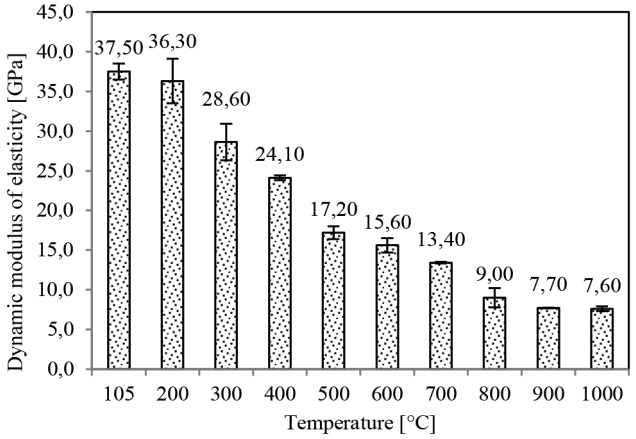

Fig. 5 Evaluation of dynamic modulus of elasticity

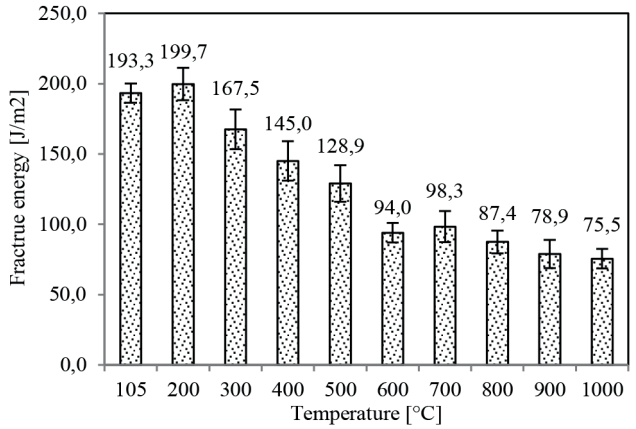

Fig. 6 Evaluation of fracture energy

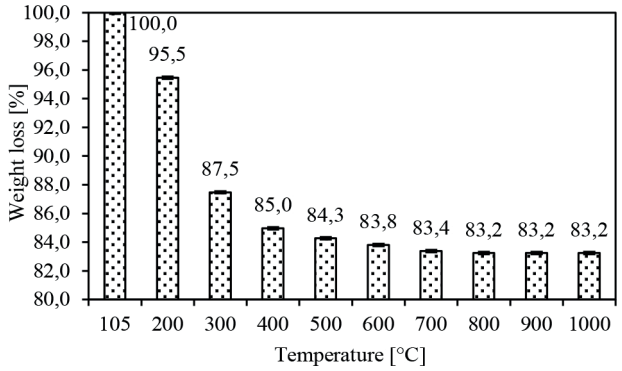

Fig. 7 Gradual weight loss determined using thermogravimetry

The resulting thermogravimetric measurement expressed by the cumulative weight loss of used binder corresponds well with the accompanied mineral changes. The dominant part of the decomposition of the formed hydrates took place up to 400°C, Fig. 7, where the weight loss of about 15% was monitored. The resulting thermal loading did not affect the studied material to any considerable degree. Total weight loss obtained after 1000°C exposure was shown to be only 16.8%.

5 Conclusions

The experimental work performed was focused on the detailed study of material transformations related to temperature loading. An investigation was carried out on the fiber composite mixture of optimized composition, which was developed in previous research. The studied composite was loaded by the elevated temperatures up to 1000°C, by 100°C increments.

In terms of the investigated mechanical properties, it can be concluded that the residual values correspond well with the mineral changes, and with binder decomposition respectively, during the gradual temperature loading. The most common of the formed hydrates decomposed up to 400°C, which was accompanied by the considerable decline of all the studied parameters that is specific to the calcium aluminate cement-based composites. The gradual increase of temperature load and consequent loss of mechanical properties was likely caused by the volume changes, accompanied by the initiation of cracks, resulting from the likely absence of siliceous component in the mixture, which decomposed after reaching a temperature of 573°C. Gradual deterioration was evident in the results of NDT measurement; however, the studied composite kept its integrity without any visual damage. Nevertheless, the application of longer fibers would be beneficial, due to a higher contact zone with respect to the maximum size of the grain that was studied.

The absence of secondary rehydration processes is a significant advantage of composites based on the calcium aluminate cements, which allows reuse of the material, although with reduced mechanical properties. Composites based on traditional Portland cement exposed to elevated temperature often suffer from internal stresses arising from the secondary hydration of quick lime after contact with water.

The production of calcium aluminate cement is very energy intensive; nevertheless, this binding system is indispensable to composite production for application at high temperatures. The research focused on the CAC-based composite confirmed the possibilities for the increased energy efficiency of these composites using metakaolin replacement.

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