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CYCLIC TEMPERATURE LOADING: RESIDUAL FLEXURAL STRENGTH OF REFRACTORY SLABS

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Abstract. This paper describes the effect of cyclic elevated temperature loading on refractory slabs made from high performance, fibre reinforced cement composite. Slabs were produced from aluminous cement-based composites, reinforced by different dosages of basalt fibres. The composite investigated in this study had self-compacting characteristics. The slabs used were exposed to different thermal loading – 600 °C, 1000 °C, six times applied 600 °C and 1000 °C. Then, flexural strength was investigated in all groups of slabs, including group reference slabs with no thermal loading. The results show that the appropriate combination of aluminous cement, natural basalt aggregate, fine filler and basalt fibres in dosage 1.00 % of volume is able to successfully resist to cyclic temperature loading. Tensile strength in bending of these slabs (after cyclic temperature loading at 600 °C) achieved 6.0 MPa. It was demonstrated that it is possible to use this composite for high extensive conditions in real industrial conditions.

Keywords: refractory slabs; fibre composite; bending test; flexural strength; high aluminous cement; natural aggregate; cyclic thermal load.

1. Introduction

Refractories can be classified into many categories. According to the way of their production, refractory composites can be divided into shaped ones (bricks or similar precast elements) and non-shaped (monolithic) ones. In the last decades the monolithic refractories (refractory concrete) have been increasingly used due to their advantages over shaped refractories [1]. From the global point of view, refractory composites should be able to resist the action of elevated temperature. In general, refractory cement composites are characterized by excellent toughness, mechanical properties (compressive and flexural strength), thermal shock resistance, low permeability and higher porosity, etc. [2, 3]. Fibre reinforced cement composites suitable for high temperature applications have to satisfy several conditions. The binder, aggregate and fibres have to be stable in elevated temperature, and there has to be no weak part. Cohesion between the fibres' surface and hydration products must not be distorted by the effect of a thermal load [4]. These composites are suitable for industrial application in an environment with elevated temperature. We can also find a possible application in the field of building materials (e.g., chimney of fireplace bricks).

Motivation for experimental program. The main goal of the performed experimental program is the analysis of residual mechanical properties and macroscopic coherency of refractory slabs after cyclic elevated temperature loading. Several research works have investigated the effect of elevated temperature on mechanical properties, usually after one thermal load cycle [5]. In practical industrial application, the non-shaped refractory concrete is exposed to a high number of cyclic temperature changes. This specific type of thermal load occurs e.g., during technological breaks, accidents (emergency) or during planned shutdown of production. Long-term durability of refractory composite can be achieved by using high quality material. This solution will result in lower financial and environmental costs connected with more frequent repairs, replacement of damaged parts and service interruptions.

In the available literature we can find that some research deals with the cyclic load of refractories, usually dynamic or static, but none of these works focused on cyclic thermal loading. Exposure to tensile cyclic and static loading up to rupture constitutes a different approach of approximate quantification of the life-cycle of refractory concrete. This approach was used in [6] where commercial alumina refractory concrete was investigated after one heat treating at 900 °C level. The evaluation of cyclic loading in general can be investigated by X-Ray tomography [7]. A very common arrangement of large-scale tests is described in [8] where the fire-resistance of slabs made from reinforced concrete and pre-stressed concrete was tested.

The aim of this article is to investigate the effect of cyclic temperature loading at two levels – 600 °C and 1000 °C. The chosen size of slabs used in the performed experimental program was 300 × 200 × 38 mm. These dimensions better reflect real conditions in industrial application than ordinary laboratory-used specimens of 40 × 40 × 160 mm. The size effect of tested specimens can influence the final properties and durability, where some negative phenomena can occur on a larger slab area. Also, the shape of the

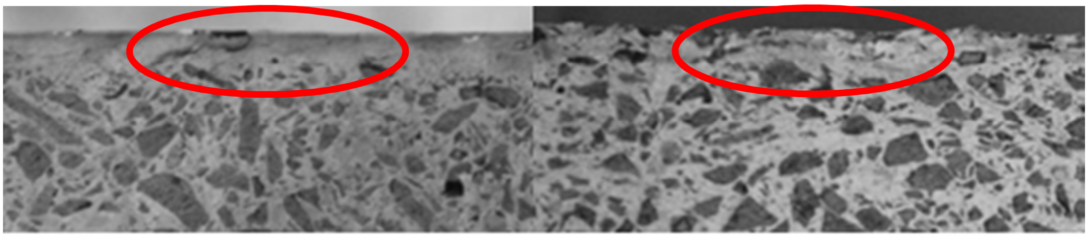

FIGURE 1. Specimen with segregation of fine fraction (0.25 % of fibres – left side) and without segregation of fine fraction (1.00 % of fibres – right side).

tested specimens (slabs) better reflects real elements made from refractory composites and used in practical applications. Slabs with similar dimensions of 400 × 250 × 13 mm reinforced by basalt fabrics were used in [9], where the authors investigated the direct tensile strength after submitting to different temperature.

At first, the sets of slabs with different dosage of basalt fibres were produced. At the age of 28 days all slabs were dried at 105 °C. One group of slabs was exposed to 600 °C for 3 hours and a different group was exposed to 1000 °C for 3 hours. Cyclic temperature loading was performed on different sets of slabs. There were three slabs in one group. One group was submitted to six loading cycles at 600 °C level and second group was submitted to six loading cycles at 1000 °C level. The influence of the performed temperature loading was investigated by three-points bending tests.

2. Materials and methodology

2.1. Composition of used mixtures

The mixtures used in the performed experimental program were investigated and tested in various modifications in previous research. The mixture used consists of basalt aggregate, fine ground ceramic powder, aluminous cement, basalt fibres, plasticizer and water. For the purpose of refractory composites, it is very common to supplement cement by another fine component. Application of metakaolin [10, 11] or microsilica in refractory composites is the generally used solution in technology of refractories.

Basalt aggregate. The combination of two fractions of basalt aggregate — 0/4 mm and 2/5 mm — was chosen in a ratio to create appropriate granulometric curve. The crushed aggregate comes from a local source (quarry Dobkovičky). The properties of the used natural aggregate limit the temperature range of application of final refractory composite. The approximate service temperature limit of basalt aggregate is 900 °C [12]. For higher temperature over 1000 °C it is necessary to use artificial aggregate.

Ceramic powder. Ceramic powder in dosage of 225 kg m^{-3} represents fine particles. This component shows pozzolanic properties and simultaneously has already gone through exposure to high temperature. Fine ground ceramic powder is a waste material produced during grinding of brick blocks. The philosophy of utilizating of waste materials in concrete or cement composite technology is an issue treated in several research works. We can find the application of waste materials in the form of crushed aggregate, or as fine parts [13]. The action of temperatures elevated to 500 °C was the objective of [14], where concrete with natural aggregate and a combination of natural and recycled aggregates had been studied. The concrete with recycled aggregate shows higher porosity. This concrete achieved better resistance to the action of elevated temperatures due to the pores' size distribution [15]. Even in the area of refractory cement composite, which is a very specific, expensive and closely profiled branch, different research deals with the possible application of recycled crushed refractory concrete as an aggregate for newly produced refractory concrete [16].

Aluminous cement. The hydraulic bond is provided by the aluminous cement Secar®71 with a total amount of Al_2O_3 over 70 %. The amount of Al_2O_3 determines the temperature range of application (for the purpose of application over 1000 °C, the minimum is 70 %). Modern aluminous cement is produced from high quality, pure, artificial bauxite. A modern method for producing high quality cement consists of melting raw materials (mixture of bauxite and limestone) at 1600 °C in an electric arc furnace [17]. Major clinker phases of this type of binder are CA ($CaO \cdot Al_2O_3$), CA_2 ($CaO \cdot 2\,Al_2O_3$) and $C_{12}A_7$ ($12\,CaO \cdot 7\,Al_2O_3$) [18].

Basalt fibres. The used mixtures vary in total amounts of fibres from 0.25 to 1.0 % of volume. Based on previous research we can conclude that the combination of two lengths of basalt fibres at a ratio of 1 : 9 on behalf of length of 12.7 mm shows the best mechanical properties after action of elevated temperatures. Density of used fibres of 2900 kg m^{-3} and tension strength of 2 GPa are the general characteristics of this material [19]. The slabs with the lowest dosage of fibres (0.25 % of volume) show segregation of technological water and cement (bleeding) (Figure 1). On the other hand, the fibres' dosage of 1 % lies at the limit of workability of fresh mixture on self-compacting level.

Components	**Mixture** [kg m^{-3}]		
	C_0.25_C5	C_0.50_C5	C_1.00_C5
Cement Secar®71	675	675	675
Ceramic powder	225	225	225
Basalt aggregate (0/5 mm)	1100	1100	1100
Water – potable	224	224	224
Plasticizer SVC	22.7	22.7	22.7
Basalt fibres 6.35 mm	0.73	1.46	2.92
Basalt fibres 12.7 mm	6.52	13.04	26.08

Table 1. Used mixtures – composition.

Mixture	**Temperature** [°C]	**Bulk density** [kg m^{-3}]	**Flexural strength** [MPa]	**Compressive strength** [MPa]	**Fracture energy** [J mm^{-2}]
C_0.25_C5	105	2425	12.6	97	160.4
	600	2275	6.5	62.6	303.8
	1000	2125	3.2	31.5	238.8
C_0.50_C5	105	2440	11.7	80.2	79.3
	600	2320	6.1	52	148.4
	1000	2125	3.2	35.2	94.7
C_1.00_C5	105	2410	10.7	89	77.9
	600	2275	4.1	48.7	70.6
	1000	2070	3.8	27.6	72.9

Table 2. Properties of the used mixtures after the action of various conditions.

Plasticizer. In accordance with principles of high performance concrete production [20–22], a high amount of super-plasticizer (based on polycarboxylether) was applied to reduce the water to binder ratio to 0.25. This high dosage of plasticizer can cause changes during its burning and evaporation after the first thermal loading cycle. Based on the study performed by Jogl et al., we can say that the high dosage of plasticizer does not negatively influence residual mechanical properties [23].

For detail information about the composition, see Table 1. In Table 2, the bulk density, flexural strength, compressive strength and fracture energy of used mixtures are shown (after exposure to 105 °C, 600 °C and 1000 °C). All mentioned properties were measured on specimens with dimensions of 40 × 40 × 160 mm and are taken from [24].

2.2. Cycling temperature loading

In previous research [11, 23, 24], the influence of elevated temperatures on various properties of refractory composites has been investigated after exposure to one loading cycle. In accordance with the goal of this research, the temperature loading scheme was modified. All specimens were dried for 72 hours at 105 °C at the age of 28 days. The major part of free and physically bounded water was evaporated. This is in line with using refractory elements in practice. The pore pressure formed during evaporation of this water can cause spalling from the surface, macroscopic cracks and total destruction.

Two different thermal loading cycles were performed – one with maximal temperature of 600 °C and the second with maximal temperature of 1000 °C. The temperature loading process consisted of an increase in temperature with a gradient of 10 °C/min till the selected final temperature level (600 °C or 1000 °C). When reached, the required temperature was maintained for three hours; then the electric furnace was turned off and the temperature naturally cooled down to laboratory conditions (see Figure 2). For the purpose of cyclic thermal loading, this temperature loading cycle was repeated six times.

The experimental program consisted of three reference specimens from each mixture (0.25 %, 0.50 %, 1.00 % of fibres) not exposed to elevated temperature level. Three specimens were exposed to 600 °C and the last three specimens were exposed to 1000 °C. The influence of cyclic thermal load was investigated in a similar way. From each mixture two groups of three specimens were selected. The first group was exposed to six thermal load cycles at a level of 600 °C. The second set was exposed to six thermal load cycles at a level of 1000 °C.

2.3. Destructive testing

The test arrangement of slabs was in accordance with three points bending test with clear distance of sup-

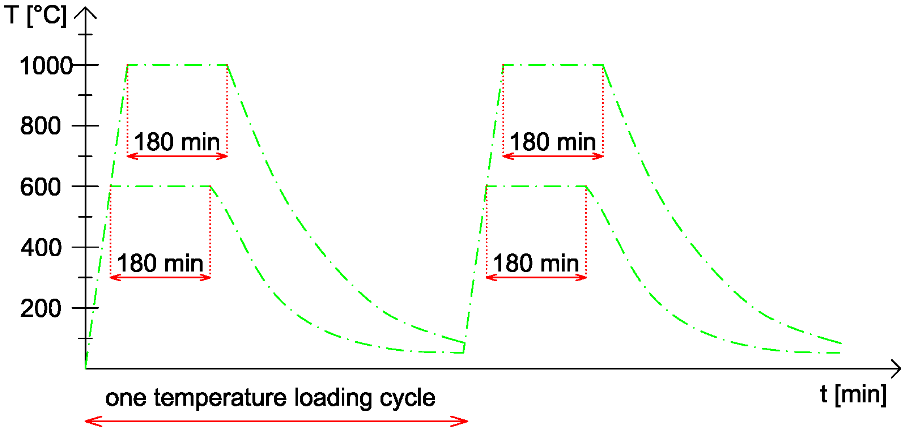

FIGURE 2. Description of thermal loading cycles.

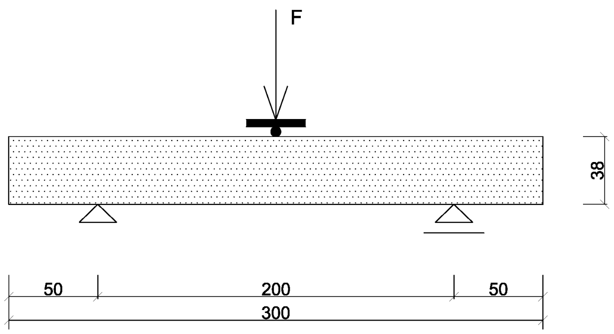

FIGURE 3. Slab bending test arrangement [mm].

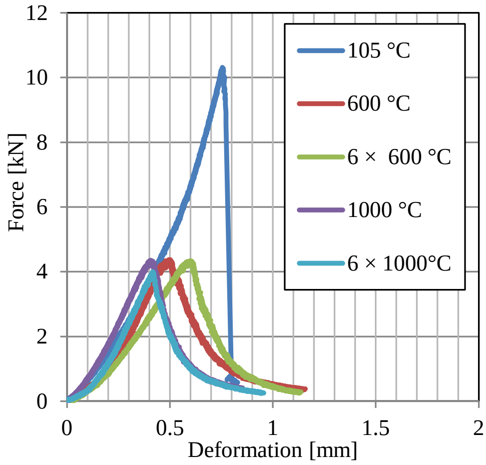

FIGURE 4. Load-deflection diagrams of slabs with 0.25 % of fibres.

ports of 200 mm and 50 mm overlap on both sides. Acting force was designed in the middle of specimen. The whole test was arranged by an increase of deformation of 0.2 mm/min controlled by the INOVA hydraulic system. Data from PS20 force sensor and DTA10 inductive displacement transducer were continuously recorded by the DEWETRON system. Due to the small and especially slim shape of specimens the inductive displacement transducer registered the displacement of the loading cell, but not the deformation in the middle of the span under action force. In the point of view of small achieved forces, this solution is acceptable and has its value. Figure 3 shows the arrangement of the bending test, together with all measures in [mm].

The final tensile strength in bending was calculated in accordance with the theory of plasticity:

$$f_t = \frac{3F_{\max} l}{4h^2 b}, \tag{1}$$

where f_t [MPa] is the tensile strength in bending, $F_{\max}$ [N] is the maximal force achieved during bending test, l [mm] is the clear span of supports, h [mm] is the height of slab, b [mm] is the length of slab.

3. Results and discussion

First, it is necessary to say that thermal loading started by drying at 105 °C (28 days old slabs). Therefore elevated temperature loading took place on specimens without the vast majority of free water. This solution is in accordance with real conditions of refractory composites in industrial application. In total, 45 slabs were manufactured and tested in this research.

3.1. Bulk density

The dimensions of all specimens were measured before drying. The weight was measured before and after drying and after each temperature loading cycle. Bulk density depends on gradual changes of weight, while the dimensions remain constant. Drying at 105 °C for 72 hours means a decrease of bulk density about 2 % in average (mostly free water). Exposure to one thermal load cycle on level 600 °C means a decrease in bulk density of about 9.4 % in average. This temperature range represents the most noticeable change in bulk density, which is connected with chemical changes and evaporation of the rest of physically bounded water. Repeating of thermal load on the level of 600 °C means an insignificant decrease in bulk density, which implies that no substantial changes take place. An approximately 1 % decrease of bulk density characterizes the action of 1000 °C in comparison with specimens

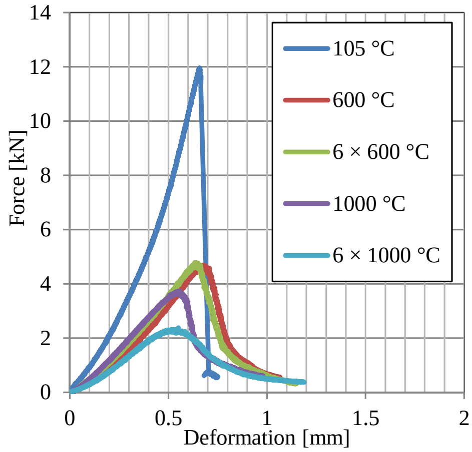

Figure 5. Load-deflection diagrams of slabs with 0.50 % of fibres.

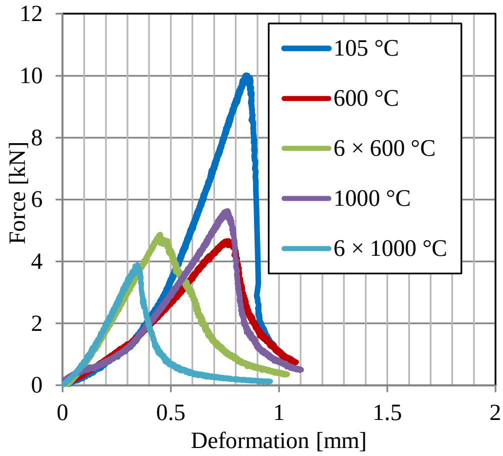

Figure 6. Load-deflection diagrams of slabs with 1.00 % of fibres.

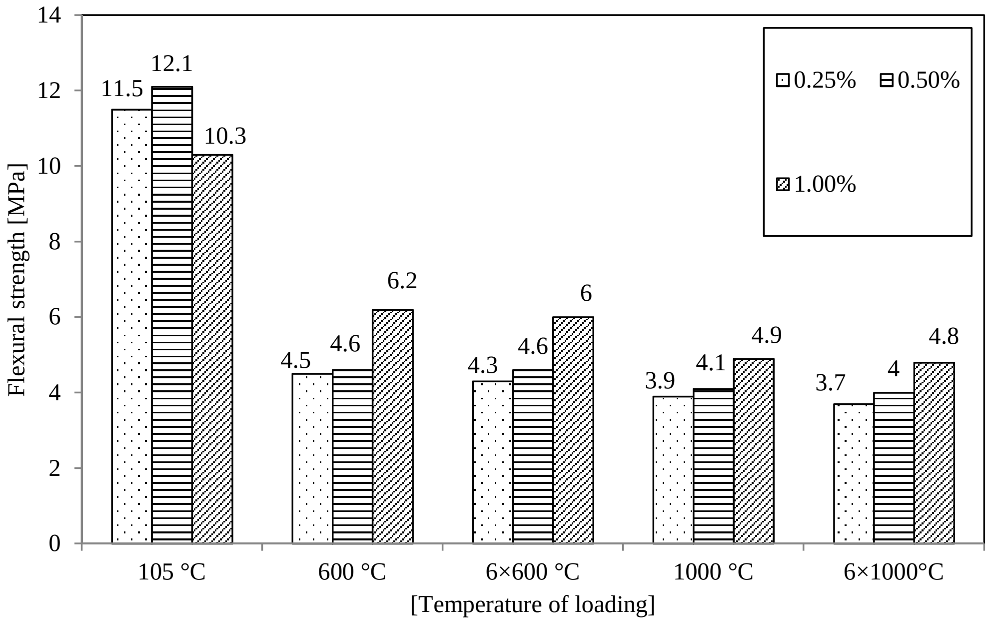

Figure 7. Evaluation of flexural strength.

exposed to six thermal loading cycles on the 600 °C level. A slightly higher decrease (approximately 1 %) describes the cyclic thermal load at 1000 °C in comparison with specimens exposed to one loading cycle on the 1000 °C level. Table 2 clearly shows all values of bulk density after performed thermal loading and evaluates the changes in percentage expression. The trend of gradual decrease of bulk density due to action of elevated temperature is summarized in Figure 7.

3.2. Flexural strength and deformation

Table 3 summarizes the values of deflection measured at the moment of maximal achieved force during the bending test. Flexural strength, listed in the same table, was calculated according to the theory of plasticity (1) from the dimensions and maximal achieved force. The listed values present the arithmetic average obtained from three specimens. Slabs dried at 105 °C were tested as the comparison level for quantification of flexural strength decrease. Action of 600 °C means decrease to 39.1 % of the original value (for 0.25 % of fibres), 38.0 % of the original value (for 0.50 % of fibres), while a dosage of 1.0 % means highest resistance expressed by 60.2 % of the original value. Slabs exposed to six thermal load cycles at 600 °C level decreased by 1.2 % on average in comparison with the values after one thermal load cycle. We can observe that the highest decrease of bending capacity of all tested slabs (independently on the total amount of fibres) takes place in the temperature range between 105 °C and 600 °C. This is in accordance with the available literature, where the temperature of 400 °C is usually mentioned (for refractory concrete with natural aggregate) [25] as the limit for decrease of basic and mechanical properties. Values of slabs with 0.25 % and 0.50 % of fibres are on a similar level of 33.9 % after 1000 °C, respectively 32.5 % after six thermal load cycles on a level of 1000 °C. We can observe the

Mixture	Thermal loading					
	21 °C	105 °C	600 °C	6 × 600 °C	1000 °C	6 × 1000 °C
	Bulk density [kg m^{-3}]					
A_0.25_C5	2410 100 %	2370 98.3 %	2185 90.7 %	2165 89.8 %	2150 89.2 %	2130 88.4 %
A_0.50_C5	2325 100 %	2280 98.1 %	2105 90.5 %	2095 90.1 %	2080 8940.0 %	2055 88.4 %
A_1.00_C5	2295 100 %	2240 97.6 %	2080 90.6 %	2070 90.2 %	2045 89.1 %	2025 88.3 %

Table 3. Values of bulk density after different thermal load.

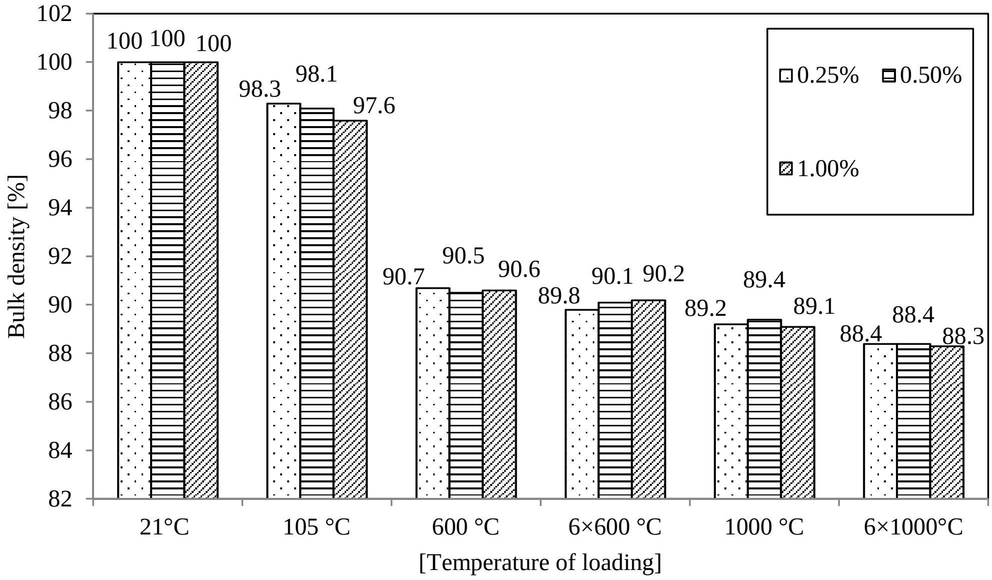

Figure 8. Evaluation of bulk density.

positive benefit of basalt fibres in the case of 1.00 % of volume dosage and its reaction to action of 1000 °C thermal load. The residual bending capacity is on 47.6 % of flexural strength achieved at 105 °C. Six thermal load cycles at 1000 °C level caused a decrease of only 1 % in comparison with one thermal load cycle. This decrease lies in the interval of statistic error.

3.3. Ductile behaviour

From the load-deflection diagrams (Figures 4–6) we can observe the behaviour of refractory slabs during bending and especially after reaching maximal achieved force. However, it should be noticed that each load-deflection diagram represents only one specimen from a group of three samples. Independently on the fibres' volume, all slabs without thermal loading achieved brittle breakdown after reaching the maximal force during bending. The action of fibres in the form of ductile behaviour did not show. Slabs exposed to thermal load, regardless of whether cyclic or not, showed ductile behaviour, due to the action of basalt fibres. The effect of elevated temperature is reflected by colour changes, which are illustrated in Figure 9.

4. Conclusions and further outlook

The bending capacity of refractory slabs made from fibre-reinforced cement composite was determined on a total number of 45 slabs. These slabs were produced with various volume amounts of fibres and tested after exposure to different thermal loads. Based on the results derived from the performed experimental program, we can present the following conclusions:

(1.) The highest decrease of bulk density, which is generally connected with the evaporation of free, physically and chemically bound water, took place after drying at 105 °C. The loss of all chemically and part of physically bound water is connected with higher thermal exposure up to 400 °C. Also, the highest decrease of bulk density after thermal loading (600 °C or 1000 °C) took place after the first thermal loading cycle. The other thermal loading cycles caused only an insignificant decrease of bulk density. This gradual decrease of bulk density could be caused by the release particles from the surface due to cyclic thermal loading.

Mixture	Physical quantity	Thermal loading				
		105 °C	600 °C	6 × 600 °C	1000 °C	6 × 1000 °C
A_0.25_C5	**Flexural strength** [MPa]	11.5	4.5	4.3	3.9	3.7
	Deflection [mm]	0.72	0.53	0.69	0.38	0.54
A_0.50_C5	**Flexural strength** [MPa]	12.1	4.6	4.6	4.1	4
	Deflection [mm]	0.76	0.66	0.73	0.51	0.58
A_1.00_C5	**Flexural strength** [MPa]	10.3	6.2	6	4.9	4.8
	Deflection [mm]	0.82	0.75	0.78	0.74	0.62

Table 4. Flexural strength and maximal deformation of tested slabs.

Figure 9. Colour changes of tested slabs after the action of elevated temperatures (from the left side: 105 °C, 600 °C, 1000 °C).

(2.) From a macroscopic point of view we can conclude that cyclic thermal loading at 600 °C level did not cause any visual cracks in the matrix or in the aggregate. The first exposure to 1000 °C did not cause any visible cracks. Approximately the third thermal loading cycle caused visual cracks in the corners of the tested slabs made from composite with fibre dosage of 0.25 %. These cracks did not develop throughout the entire cross-section. Maximal fibres dosage prevents this negative phenomenon, but the limit of operation temperature for natural basalt aggregate was expressed.

(3.) It has been specified that the deflection at maximal achieved force in the bending test increased with increasing fibre dosage independently on intensity of thermal load. This is connected with higher ductility in specimens with higher dosage of fibres. Thermal loading reflected its influence in different failure mode and behaviour during bending. The slabs dried at 105 °C collapsed fragile after reaching the maximal force. We can find the softening part of load-deflection diagrams only in the specimens after exposure to elevated temperature, independently on total dosage of fibres.

(4.) The values of achieved flexural strength from bending tests describe the benefit of basalt fibres in various dosages. The maximal fibres' dosage of 1.00 % of volume resulted in highest flexural strength of slabs after thermal loading. Especially this dosage shows excellent resistance to cyclic thermal loading (after 600 °C and 1000 °C). This dosage is also the highest acceptable one in accordance with the workability of fresh mixture.

(5.) In addition it was found that the tested cement composite with a dosage of 1.00 % volume of basalt fibres is suitable for application in refractory slabs or similar element production. Even after exposure to cyclic thermal loading (6 × 600 °C), slabs made from this composite showed excellent mechanical properties and ability to resist external mechanical load, without any visual cracks. The limit of tested composites is in the properties of natural basalt aggregate, which was predicted and expected.

Further experiments will deal with the possible application of artificial aggregate (fire clay, liaver, etc.) for the production of refractory slabs. Usage of this aggregate will allow the application at temperatures over 1000 °C. Due to how highly time-consuming thermal loading in an electric furnace is, only six cycles were performed. In further experiments an increased number of thermal loading cycles will be performed.

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