



Univerzitet u Zenici
University of Zenica
Bosnia and Herzegovina
FAKULTET ZA METALURGIJU I MATERIJE
FACULTY OF METALLURGY AND MATERIALS SCIENCE
FACHHOCHSCHULE GELSENKIRCHEN
Gelsenkirchen
Germany



VII Naučno/stručni simpozijum
sa međunarodnim učešćem

7th Scientific/Research Symposium
with International Participation

METALNI I NEMETALNI MATERIJALI
proizvodnja – osobine – primjena

METALLIC AND NONMETALLIC MATERIALS
production – properties – application

ZBORNIKA RADOVA

PROCEEDINGS

Zenica, Maj 2008.

UREDNIK/EDITOR
Dr Fuad Begovac

IZDAVAČ/PUBLISHER
Univerzitet u Zenici
Organizaciona jedinica Fakultet za metalurgiju i materijale
Travnička cesta 1, 72000 Zenica
Tel: ++ 387 401 831, 402 832, Fax: ++ 387 406 903

KOMPJUTERSKA OBRADA TEKSTA
TECHNICAL ASSISTANCE AND DTP

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Elektronsko izdanje/Electronic Edition

TIRAŽ/ISSUE: 200 primjeraka/copies CD

CIP - Katalogizacija u publikaciji
Nacionalna i univerzitetska biblioteka
Bosne i Hercegovine, Sarajevo

666/669:502/504](063)(082)

NAUČNO/stručni simpozij sa međunarodnim učešćem Metalni i nemetalni materijali (7 ; 2008 ; Zenica) Metalni i nemetalni materijali : proizvodnja, osobine, primjena : zbornik radova / VII naučno/stručni simpozij sa međunarodnim učešćem Metalni i nemetalni materijali, Zenica, [22.-23. maj 2008.] = Metallic and nonmetallic materials : production, properties, application : proceedings / 7th Scientific/Research Symposium with International Participation Metallic and Nonmetallic Materials, Zenica, [22.-23. May 2008.]. - Elektronski tekstualni podaci. - Zenica Fakultet za metalurgiju i materijale = Faculty of Metallurgy and Materials Science, 2008. - 1 elektronski optički disk (CD-ROM) : tekst, slike ;12 cm

Elektronski izvor. - Nasl. s naslovnog ekrana. - Bibliografija uz sve referate

ISBN 978-9958-785-10-8

COBISS.BH-ID 16628486

ZAHVALNICA

Organizacioni odbor VII Naučno/stručnog simpozijuma sa međunarodnim učešćem pod nazivom „METALNI I NEMETALNI MATERIJALI“ zahvaljuje se:

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na ukazanom povjerenju i finansijskoj pomoći. Zahvaljajući vama uspeli smo da organizujemo Simpozijum koji je od izuzetne važnosti za nas.

Hvala!

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***NEMETALNI MATERIJALI – NONMETALLIC
MATERIALS***

HIGH-TEMPERATURE CONCRETES, EFFECT OF POROSITY AND PHASE-CONTENT ON ITS MECHANICAL PROPERTIES

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Key words: refractory concrete, bauxite, corundum, micro-structure, mechanical properties, silica fume

ABSTRACT

Two types of refractory concrete were prepared: first group of samples containing corundum and calcium-aluminate cement, and second group containing bauxite, calcium-aluminate cement (CAC) and chamotte. Each group of samples was prepared in 3 different variations: without admixture, with admixture and with silica fume as additive. Samples were dried at 110°C, and afterwards heat-treated at 110, 800, 1300 and 1500°C. Porosity was investigated using program for image analysis (Image Pro Plus) and, afterwards, effect of porosity on the mechanical properties (cold strength, strength at high temperatures, etc.) was observed. It was noticed that corundum concrete shows better characteristics than bauxite concrete. It is caused by pores and pore-size distribution, and also due to the formation of new mineral phases.

1. INTRODUCTION

Refractory materials are known for centuries. Their application in metallurgy, in steel and metal industry, as well as in cement and glass fabrication, is irreplaceable. However, the quality and requirement, as well as brand new possibilities, of refractory materials are ever increasing. Nowadays „shaped“ refractory materials are almost replaced by „unshaped“ products such is refractory concrete. Advantages of unshaped refractory material are numerous: easy application to unreachable sections (footer of a furnace), possibility of producing complex concrete shapes or filling out extremely thin sections. It is, also, possible to repair damaged concrete lining of a furnace with special reparation mortar or concrete, which couldn't be performed before in the case of shaped products. The most important thing is probably aspect of economy and decreasing of expenses connected to manufacturing of final product – refractory concrete ^[1, 2].

This paper is concerned with self-flowing concrete, prepared with standard portion of refractory high-aluminate (calcium-aluminate) cement – CAC. In order to obtain the best possible performances, mixture is carefully designed. Particle size has been limited for high-packing

density to be reached. Thus, size and portion of coarse aggregate is also reduced to minimum. CAC has been applied as binder, because of its standard use in manufacturing of the refractory concrete and thus it is easier to compare properties of commercial refractory concrete with experimental one – varying only type of aggregate and additive.

2. CHARACTERIZATION AND COMPOSITION OF CONCRETES

Experiments were performed on two different compositions of concrete (shown in table 1). Composition of the first type of concrete (B – concrete) is bauxite + chamotte + CAC and it is „commercial“ concrete. Composition of „experimental“ concrete (K – concrete) is corundum + CAC. Each type of concrete has 3 different sub-types: B1 and K1 are without additives, B2 and K2 are with admixture added (chemical bonding agent Litopix P-56), B3 and K3 are prepared with addition of silica fume.

Table 1. Compositions of investigated commercial and experimental concretes.

TYPE OF CONCRETE	COMPOSITION	MARK
commercial concrete I	bauxite (40 %), (0-4; 0-6 mm); chamotte (30 %; 0-1 mm); CAC (25 %); water (12 - 14 % on 100 %)	B1
commercial concrete II	bauxite (40 %; 0-4; 0-6 mm); chamotte (30 %; 0-1 mm); CAC (25 %); chemical additive (1 % on 100 %); water (12 - 14 % on 100 %)	B2
commercial concrete III	bauxite (40 %; 0-4; 0-6 mm); chamotte (30 %; 0-1 mm); CAC (25 %); silica fume (1 % on 100%); water (12 - 14 % on 100%)	B3
experimental concrete I	corundum (80 %; 0-1, 1-3, 3-5 mm); CAC (20 %); water (12 - 14 % on 100%)	K1
experimental concrete II	corundum (80 %; 0-1, 1-3, 3-5 mm); CAC (20 %); chemical additive (1% on 100 %); water (12 - 14 % on 100 %)	K2
experimental concrete III	corundum (80 %; 0-1, 1-3, 3-5 mm); CAC (20 %); silica fume (1% on 100 %); water (12 - 14 % on 100%)	K3

Chemical analyses of raw materials, which are used as components for refractory concretes are shown in table 2. Chemical analyses of concretes B and K are presented in table 3.

Table 2. Chemical analysis of raw materials.

content, (%)	Al ₂ O ₃	SiO ₂	CaO	MgO	Fe ₂ O ₃	Na ₂ O	K ₂ O	TiO ₂	L.I.
raw material									
CAC	68,85	0,107	29,73	0,1368	0,058	0,285	0,0078	<0,01	0,6
Bauxite	81,4	10,70	1,05	0,473	1,80	0,046	0,309	4,14	0,35
Chamotte	31,0	56,2	1,36	0,41	2,86	0,177	1,45	1,26	5,02
Corundum	99,82	0,06	0,025	-	0,07	0,2	-	0,009	0,01
Silica fume	0,227	94,62	0,35	0,108	2,86	0,446	1,32	0,033	2,76
Litopix p-56	Al ₂ O ₃ = 4	SiO ₂ = 24			P ₂ O ₅ = 19		Na ₂ O = 33		20

Table 3. Chemical analysis of B and K concrete.

content, (%)	Al ₂ O ₃	SiO ₂	CaO	MgO	Fe ₂ O ₃	Na ₂ O	K ₂ O	TiO ₂	L.I.
raw material									
B concrete, (B1, B2, B3)	62,88	21,17	8,26	0,35	1,57	0,059	0,56	2,03	2,58
K concrete, (K1, K2, K3)	93,62	0,07	5,97	0,03	0,066	0,21	-	0,007	0,12

3. METHODS USED IN EXPERIMENT

Bauxite-based and corundum-based samples of concrete are prepared and cured in exactly the same manner: both samples are mixed for 8 minutes in laboratory RILEM-Cem mixer. Afterwards, the mixture is poured into mould and cured for 24 hours at ambient temperature 20°C and relative humidity $\phi = 60\%$. After 24 hours, when samples gained enough mechanical strength, they are taken out from the molds. During following 7 days they are cured at exactly the same temperature and humidity as when they were prepared. After 7 days, samples are dried at 110°C for 24 hours. Dried samples are fired at 800, 1300 and 1500°C for 4 hours. Dimensions of the samples are 10x10x10 cm.

DTA was performed on the samples of concrete. Porosity was investigated using standard laboratory testing method and using program for image analysis (Image Pro Plus). Mechanical properties (cold strength, strength at high temperatures) were investigated using standard laboratory methods and devices (hydraulic pressure device). Thermo-microscopy was performed on the concrete samples.

4. RESULTS AND DISCUSSION

DTA analysis was performed with DTA-50 Shimadzu device on the samples of K and B concrete. Diagrams are presented in the figure 1. Temperature was increased from ambient temperature up to 1100°C, with the rate of 10°C/min.

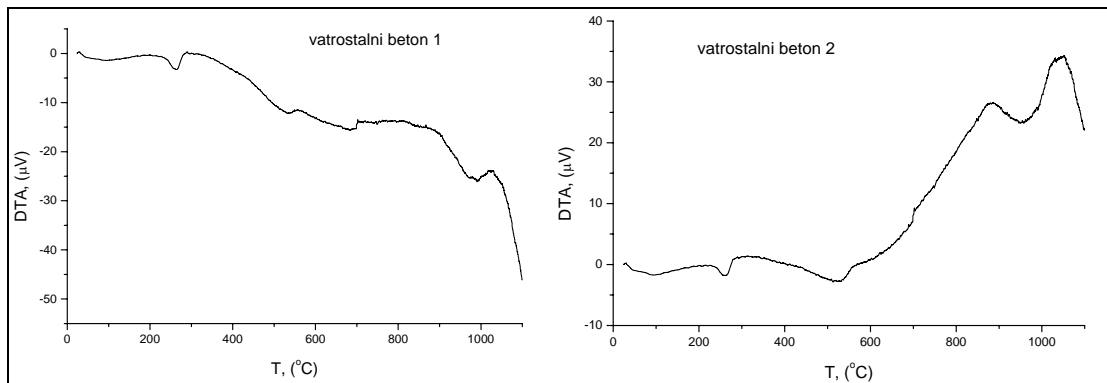


Figure 1. DTA of B and K concrete.

Both samples show endothermic effects at the following temperatures:

- Sample of B concrete: $T_1 = 246,65^\circ\text{C} \rightarrow Q_1 = -60,34 \text{ J/g}$; $T_2 = 531,77^\circ\text{C} \rightarrow Q_2 = -71,20 \text{ J/g}$; $T_3 = 681,63^\circ\text{C} \rightarrow Q_3 = -87,24 \text{ J/g}$; $T_4 = 990,62^\circ\text{C} \rightarrow Q_4 = -0.17 \text{ kJ/g}$

- Sample of B concrete: $T_1 = 261,21^\circ\text{C} \rightarrow Q_1 = -31,67 \text{ J/g}$; $T_2 = 512,75^\circ\text{C} \rightarrow Q_2 = -37,25 \text{ J/g}$; $T_3 = 957,73^\circ\text{C} \rightarrow Q_3 = -0,29 \text{ kJ/g}$

Endothermic effects correspond to the phase transformations of cement minerals: the first endothermic effect (in both cases) corresponds to dehydration of $\text{CaO}\cdot\text{Al}_2\text{O}_3$.

Apparent porosity is investigated with, both, laboratory method and Image Pro Plus (PC program for analysis of picture of a testing sample). Results were approximately the same (table 4).

Table 4. Apparent porosity of concrete samples.

temperature, (°C)	CONCRETE B			CONCRETE K		
	P _{B1}	P _{B2}	P _{B3}	P _{K1}	P _{K2}	P _{K3}
20	8,6	11,1	11,3	3,7	4,1	3,9
110	18,5	19,9	20,5	6,9	6,4	6,9
800	25,1	30,2	35,1	10,0	9,9	10,2
1300	33,6	33,26	33,1	10,6	10,7	10,6
1500	33,2	32,9	32,7	10,1	10,3	10,0

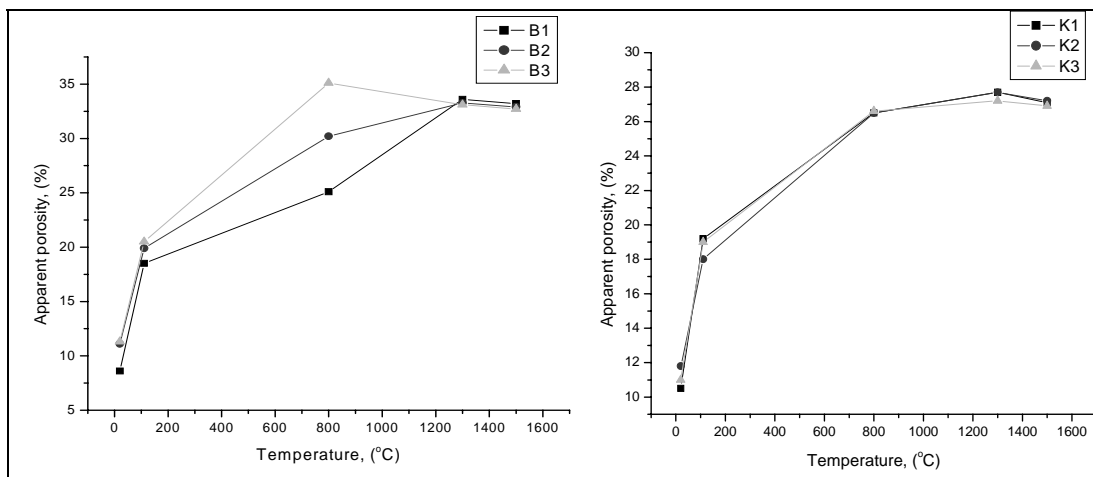


Figure 2. Apparent porosity of B and K concrete.

Due to the thermal decomposition of hydrate phases, samples which are fired at temperatures above 1000°C have lower bulk density than dried samples of concrete. As for apparent porosity, at first, it shows trend of increasing in both cases. Process of sintering starts at the temperature of cca 900°C . Effect of the sintering would be increasing of the density and decreasing of the porosity. In first case (B concrete) porosity starts decreasing at 800°C , and in second case (K concrete) porosity shows trend of increasing up to 1300°C . Beside that, apparent porosity of samples of K concrete is lower than porosity of samples of B concrete [5].

Mechanical compressive strength of concrete samples was investigated on standard laboratory hydraulic press. Pressure was disposed vertically (axial force) on one a side of the prismatic sample. Mechanical compressive strength is presented in table 5.

Table 5. Compressive strength of concrete samples.

temperature, (°C)	CONCRETE B			CONCRETE K		
	$\sigma_{p,B1}$	$\sigma_{p,B2}$	$\sigma_{p,B3}$	$\sigma_{p,K1}$	$\sigma_{p,K2}$	$\sigma_{p,K3}$
20	58.2	45	58.3	59.2	45.9	66.4
10	51.6	43.4	39.1	31.4	27.4	39.9
800	39.5	31.3	26.6	42.4	47.9	37.8
1300	22.3	23.6	17.3	24.9	26	30
1500	20.1	21.4	16.9	24.1	25.3	29.2

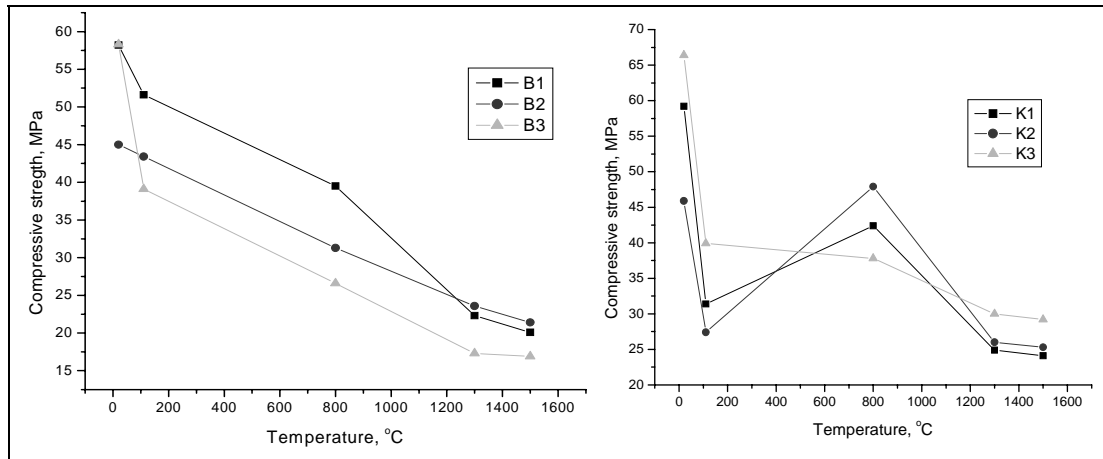


Fig 3. Compressive strength of B and K concrete.

Due to the structural properties of corundum and possible better compaction, K concrete has higher mechanical strength than B concrete. However, B concrete shows trend of continual decreasing of the strength. As for K concrete, increasing of strength at 800°C (K1 and K2) was noticed. Reason for it can be explained with difference in structure of pores and, also, with 3 times lower porosity at 800°C of K concrete [6].

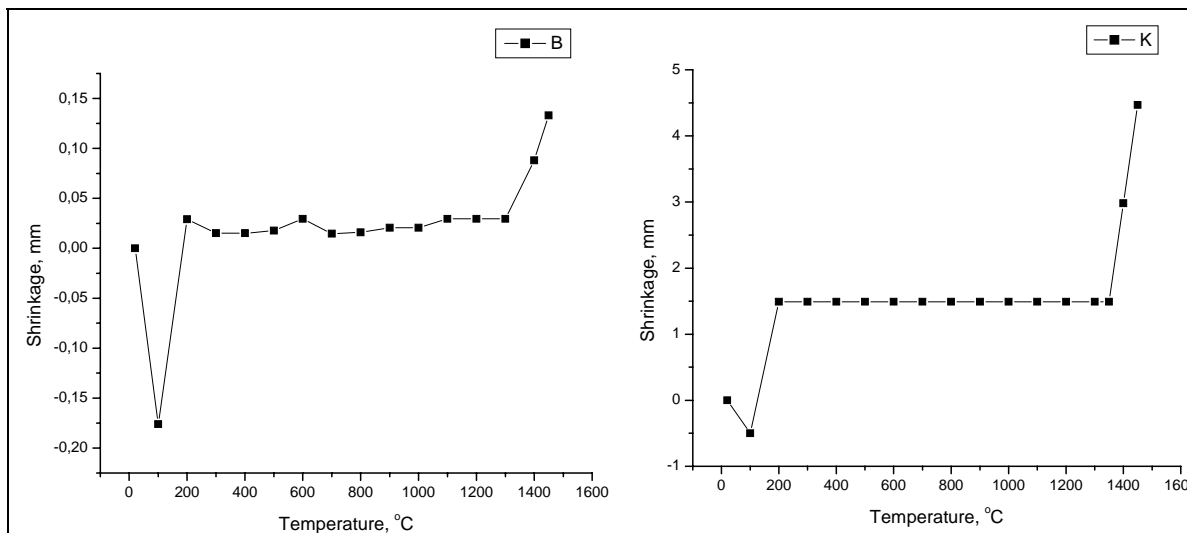


Fig 4. Shrinkage of B and K concret.

Thermo-microscopy was performed on the samples of B and K concrete. Results are presented on figure 4.

As it is presented on diagrams, shrinkage of the samples of both B and K concrete is insignificant. At the temperature of 110°C process of expanding is noticed (due to the hydration of cement). After that, samples show no activity until high temperatures are reached (1300°C for B concrete, and 1400°C for K concrete), which means that both types of concrete poses extremely high temperature of application. Temperature of application for K concrete is higher due to its microstructure and lesser porosity.

5. CONCLUSION

Cordierite-based refractory concrete (and its sub-types with admixture and silica fume) show better results than bauxite-based concrete: apparent porosity is lower, mechanical compressive strength is higher, temperature of application is higher. Reason for such behavior can be found in more favorable pore structure and pore-size, as well as in formation of different phases. Conclusion is that “experimental” concrete show better final results and better properties. However, bauxite “commercial” concrete is not significantly falling behind. In both cases (K and B concrete) results are improved by adding admixture and silica fume.

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