

# VEZA IZMEĐU MEHANIČKIH SVOJSTAVA I MIKROSTRUKTURE VATROSTALNIH KOMPOZITA ODREĐENA NEDESTRUKTIVNIM METODAMA ISPITIVANJA

## CORRELATION BETWEEN MECHANICAL PROPERTIES AND MICROSTRUCTURE OF REFRACTORY COMPOSITES DETERMINED USING NON-DESTRUCTIVE TESTING METHOD

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### 1 UVOD

Za pravljenje obloga metalurških peći i sličnih termičkih postrojenja koriste se "oblikovani" ili "neoblikovani" vatrostalni proizvodi. Primena neoblikovanih vatrostalnih materijala (vatrostalnog betona, vatrostalnog maltera, vatrostalnih torkret betona, itd.) ima brojne prednosti: pojednostavljeni način ugradnje vatrostalnih obloga, ekonomski aspekt, tj. ušteda pri procesu proizvodnje obloga, mogućnost reparacije oštećene obloge, itd. [1] Vatrostalni beton je najznačajniji i najčešće primenjivani materijal iz grupe vatrostalnih kompozita. Mehanička čvrstoća vatrostalnog betona je svojstvo koje određuje njegove performanse pri različitim namenama i definiše se kao pritisna sila koju beton može da izdrži na različitim temperaturama. Kada je beton podvrgnut režimu povišene temperature i opterećenju, dolazi do promena u mikrostrukturi: povećava se poroznost, pore postaju krupnije, javljaju se prsline unutar strukture materijala. Prethodno navedene promene dovode do smanjenja pritisne čvrstpcje i degradacije kompozita. Nastanak prslina u betonu i povećanje ukupne poroznosti utiču na smanjenje gustine i degradaciju elastičnih svojstava materijala. Dakle, merenjem bilo kojeg od ta dva svojstva (gustina, elastična svojstva) može se pratiti tok promena koje se dešavaju u mikrostrukturi betona. Prethodno može biti praktično sprovedeno primenom metode merenja brzine rasprostiranja ultrazvučnih talasa ( $v_p$ ) kroz uzorak vatrostalnog betona [2, 3].

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### 1 INTRODUCTION

Lining of metallurgical furnaces and other plants operating at high temperature are made of either shaped or unshaped refractory composites. Application of unshaped refractory composites (i.e. refractory concrete, refractory mortar and shotcrete) has numerous advantages: simplified building of refractory linings, economic aspect i.e. cheaper process of manufacturing, possibility of damaged lining reparation, etc [1]. Refractory concrete is the most important and commonly used represent of refractory composites group. Mechanical strength of refractory concrete determines its performance in different applications and it is measured in terms of applied compressive load which concrete can withstand at various temperatures. When refractory concrete is subjected to increasing compressive load and temperature, microstructure of the material changes: apparent porosity increases, pores become bigger and cracks within structure occur. It results in loss of strength and composite degradation. The formation of cracks and increasing porosity decrease density and elastic properties of the material. Therefore, measuring either of these properties can directly monitor the development and change of microstructure. Such thing can be performed by measuring the velocity of ultrasonic pulses ( $v_p$ ) traveling through the refractory concrete specimen [2, 3].

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Nedestruktivne metode ispitivanja vatrostalnih betona su zbog svojih evidentnih prednosti isplativije za primenu nego konvencionalne, destruktivne metode ispitivanja. Metoda ultrazvuka je jednostavna i brza, nema potrebe za razaranjem uzorka pri ispitivanju, pa se uzorak može koristiti i u narednim ispitivanjima, itd. Primena metode brzine ultrazvuka u nedestruktivnoj evaluaciji kvaliteta betona se izučava već decenijama. Ova nedestruktivna ispitna metoda je pokazala nezanemarljiv značaj pri utvrđivanju kvaliteta betona koji je ugrađen u obloge metalurških peći. Evaluacija pritisne čvrstoće, kao jednog od najznačajnijih svojstava vatrostalnog betona, pomoću metode brzine ultrazvuka zasniva se na empirijskoj vezi između čvrstoće i nedestruktivnih parametara. Dalje, pritisna čvrstoća je u direktnoj korelaciji sa poroznošću uzorka betona i stepenom degradacije istog. Proizvođači uređaja za ispitivanje metodom ultrazvuka obično, uz opremu, dostavljaju i dokumentaciju koja sadrži empirijske obrazce i korelacije za različite parametre karakteristične za taj ispitni sistem. Ovakve veze nisu primenjive za svaki materijal, pa se za različite mešavine betona mora raditi kalibracija [4, 5].

Pomenute matematičke relacije su izvedene pomoću regresione analize. Numeričke vrednosti i korelacija između čvrstoće ( $S$ ) i brzine ultrazvuka ( $v_p$ ) za betonski uzorak su date formulom: [6]

gde je:  $a$  i  $b$  – empirijski parametri,  $S$  – pritisna čvrstoća vatrostalnog betona i  $v_p$  – brzina longitudinalnih ultrazvučnih talasa.

Većina faktora koji utiču na pritisnu čvrstoću betona utiču i na brzinu ultrazvučnih talasa, ali ne uvek na isti način i u istoj meri. Na primer, primenjena količina agregata utiče na odnos između brzine ultrazvuka i čvrstoće betona: beton sa najvećim udelom agregata će najverovatnije imati najveću brzinu ultrazvučnih talasa [7]. Izabrani tip cementsa, takođe, utiče i na pritisnu čvrstoću betona i na brzinu prolaza ultrazvučnih talasa. Viši vodo-cementni faktor utiče na promenu brzine prostiranja ultrazvučnih talasa tako što se brzina smanjuje sa povećanjem udela vode u mešavini [8]. Međutim, Bernardo [9] je u svojim istraživanjima došao do suprotnog zaključka: viši vodo-cementni faktor doprinosi bržem rasprostiranju ultrazvučnih talasa kroz beton. Sa druge strane, utvrđeno je i da viši vodo-cementni faktor daje beton sa manjom pritisnom čvrstoćom [10]. Ovakve neusklađenosti ponekad onemogućavaju jednoznačno tumačenje rezultata dobijenih ultrazvučnim ispitivanjima.

Ipak, može se sa sigurnošću tvrditi da je metoda ultrazvuka jednostavna, precizna i pouzdana nedestruktivna metoda ispitivanja. Pregled dostupne i aktuelne literature je potvrdio da se merenje brzine ultrazvučnih talasa uglavnom koristi u cilju predviđanja pritisne čvrstoće betona. Međutim, ova metoda se može primeniti i za detekciju raznih unutrašnjih defekata betona kao što su pukotine, prsline, mrežasti sistemi pukotina, delaminacije, poroznost, itd. [11, 12]. U ovom radu su predstavljene mogućnosti metode ultrazvuka u određivanju mikrostrukturnih promena u betonu.

Metoda ultrazvuka se često kombinuje sa nekom drugom nedestruktivnom metodom, kao što su programi

Non-destructive testing method of refractory concrete is preferred due to its evident advantage over conventional compression testing. The method is simple and rapid, there is no need for destruction of specimen, thus specimen can be used afterwards, etc. The application of ultrasonic pulse velocity (UPV) in the non-destructive evaluation of concrete quality has been investigated for decades. This non-destructive testing method has proved to be of real importance as a useful tool for inspection of concrete quality in metallurgical furnaces. The evaluation by non-destructive methods of the actual compressive strength of concrete in existing structural element is based on empirical relations between strength and non-destructive parameters. Furthermore, mechanical strength is in direct relationship with porosity of concrete sample and its level of degradation. Manufacturers of UPV devices usually provide empirical relationships for their own testing system. Such relationships are not suitable for every kind of concrete. Therefore, they need to be calibrated for different mixtures [4, 5].

Mentioned mathematical relationships can be derived by means of regression analysis. Numerous data and the correlation relationships between strength ( $S$ ) and ultrasonic pulse velocity ( $v_p$ ) of concrete have been proposed and presented [6]. Commonly used formula is:

$$S = a \cdot \exp(b \cdot v_p) \quad (1)$$

where:  $a$  and  $b$  are empirical parameters determined by the least squares method,  $S$  is concrete compressive strength and  $v_p$  - the ultrasonic pulse velocity of longitudinal waves.

Most factors that influence concrete strength also influence pulse velocity, though not necessarily in the same way or to the same extent. The presence of aggregate affects the relationship between pulse velocity and the compressive strength of concrete: concrete with the highest aggregate content will probably have the highest pulse velocity [7]. Cement type influences pulse velocity and it influences the compressive strength of concrete, too. Higher water content affects the propagation velocity approximately in proportion to the change of the water content in concrete [8]. The results of experiments, performed by Bernardo [9], also show that higher water content leads to a higher pulse velocity through concrete. On the other side it was found that higher water content leads to a lower compressive strength of concrete [10]. Such dissimilarities sometimes may create ambiguity in the interpretation of the ultrasonic results.

However, it can still be stated that UPV is simple, sensitive, and reliable non-destructive testing method. A reviewing of the literature indicated that ultrasonic waves were used mainly to predict concrete strength. However, this method can also be used to detect the internal defects of concrete such as cracks, delaminating, and/or honeycombs, porosity i.e. for characterization of microstructural defects [11, 12]. UPV capabilities for monitoring microstructural change are presented and demonstrated in this work.

UPV technique can be accompanied with other monitoring non-destructive method, such is for example program for image analysis. [13, 14] Application of

za analizu slike [13, 14]. Primena optičkog mikroskopa povezanog na PC sa instaliranim programom za analizu slike otvara novi spektar mikrostrukturnih karakteristika koje mogu biti ispitane: broj pora smeštenih unutar površinskih pora, oblik i veličina pora ili pukotina, sferičnost pora, itd. U ovom radu je ispitivana poroznost pre i nakon svakog termičkog tretmana primenom *Image Pro Plus* (IPP) programa za analizu slike, a rezultati su doveđeni u korelaciju sa rezultatima ultrazvučnih merenja.

Cilj ovog rada je da se istaknu prednosti primene nedestruktivnih metoda (brzine ultrazvuka i analize slike) i da se objasne njihove mogućnosti u predviđanju ponašanja betonskih kompozita izloženih pritisku i povišenoj temperaturi.

## 2 EKSPERIMENTALNA PROCEDURA

### 2.1 Materijal

Ispitane su dve serije uzoraka vatrostalnih betona različitog sastava ( $2 \times 60$  uzoraka) označenih kao betoni C i B. Vatrostalni betoni su imali različiti procentualni deo i različitu vrstu agregata (Tab. 1.). Prva grupa betonskih uzoraka (B uzorci) su sadržali boksit kao agregat i šamot kao punioci. Ova vrsta betona je komercijalno dostupna. Druga grupa betonskih uzoraka, eksperimentalni beton C, je spravljen na bazi korundnog

optical microscope connected to PC with image analysis program enables entirely new specter of properties to be described: number of pores within surfacial pores, shape and size of pores or cracks, roundness of pores, etc. In this paper, apparent porosity level was monitored before and after each thermal treatment using Image Pro Plus (IPP) program for image analysis, and results were correlated with results of ultrasonic measurement.

The goal of this work is to use nondestructive testing method (UPV) and image analysis (IPP) and their advantages to predict the behavior of composite material submitted to compressive load and increased temperature.

## 2. EXPERIMENTAL PROCEDURE

### 2.1 Material

Two series of refractory concrete samples of different composition ( $2 \times 60$  samples), hereafter indicated as C and B, were investigated. Refractory concrete samples contained different volume fraction and different type of refractory aggregate (Tab. 1.). First type of concrete (B sample) contained bauxite and chamotte as aggregate and it can be indicated as commercially available concrete. Other type of concrete (C sample) was prepared with corundum as aggregate and it can be indicated as

*Tab. 1. Parametri projekta betonskih mešavina C i B*  
*Tab. 1. Mix design parameters for C and B refractory concrete*

Mešavina Mixtures	C beton C concrete	B beton B concrete
w/c	0.5	0.6
Zapreminska masa Green bulk density, (g/cm <sup>3</sup> )	2.92	2.54
pH	9.5	9
Cement Cement, (%)	20	30
Voda Water, (%)	(12 – 14 at 100 %)	(12 – 14 at 100 %)
Agregat Aggregate, (%)	80	40 + 30
Granulometrijski sastav korunda Corundum aggregate size, (mm)		
- 5 +3	28	
- 3 + 2	22	
- 2 +1	28	
- 1 + 0,5	12	
- 0,5+ 0	10	
Granulometrijski sastav boksita Bauxite aggregate size, (mm)		
- 6 + 4		15
- 4 + 1		55
- 1 + 0		30
Granulometrijski sastav šamota Chamotte aggregate size, (µm)		
- 74	7.56	
- 74 + 44	18.23	
- 44 + 33	17.59	
- 33 – 23	7.93	
- 23 + 15	17.53	
- 15 - 0	31.16	

agregata. Agregati su imali različit granulometrijski sastav. Obe vrste betona su spravljene sa visokoaluminatnim cementom SECAR 70 (*Lafarge*). Hemski sastav ispitivanih betona je: B beton ( $\text{Al}_2\text{O}_3$  – 62,88 %,  $\text{SiO}_2$  – 21,17 %,  $\text{CaO}$  – 8,26 %) i C beton ( $\text{Al}_2\text{O}_3$  – 93,62 %,  $\text{SiO}_2$  – 0,07 %,  $\text{CaO}$  – 5,97 %).

## 2.2 Čvrstoća pri pritisku

Čvrstoća pri pritisku vatrostalnih betona C i B je određena u skladu sa standardnom laboratorijskom destruktivnom metodom [15]. Ispitano je šezdeset uzoraka kockastog oblika svake serije (po 10 kocki za svaku temperaturu: 20, 110, 800, 1000, 1300 i 1500 °C) dimenzija (10 cm x 10 cm x 10 cm). Nakon 7 dana provedenih u klimatizovanoj komori (na 20 °C), uzorci su izvađeni iz kalupa i čuvani u istim uslovima narednih 21 dan. Posle 28 dana, uzorci su osušeni na 110 °C (u trajanju od 24 časa). Zatim su uzorci premešteni u električnu peć gde su termički tretirani na sledećim temperaturama: 800, 1000, 1300 i 1500 °C. Termički tretman je sproveden u grupama od po 10 uzoraka sa zadrškom od 4 sata na određenoj temperaturi za svaku grupu. Svaka grupa uzoraka je, zatim, ispitana destruktivnom metodom na standardnoj laboratorijskoj hidrauličnoj presi u cilju određivanja pritisne čvrstoće. Uzorci su prethodno ispitani nedestruktivnim metodama merenja brzine ultrazvuka i IPP analizom.

## 2.3 Prividna poroznost

Prividna poroznost betonskih uzoraka je određena pomoću optičkog mikroskopa (Olympus, CX31-P) povezanog sa PC programom za analizu slike. Korišćen je Image Pro Plus (IPP) program (Materials Pro Analyzer, Version 3.1, Media Cybernetics, Silver Spring, MD, USA). Snimljene su digitalne fotografije površine betonskih uzoraka pre i posle termičkih tretmana i nakon ispitivanja pritisne čvrstoće). Uzorci su pre ispitivanja pokriveni slojem krede u cilju formiranja tankog filma. Na taj način je neoštećena površina prekrivena tankim praškastim slojem (filmom), a oštećena površina je zadržala prirodnu boju vatrostalnog betona. Film je omogućio ostvarenje boljeg kontrasta između oštećene i neoštećene površine. Različite površine su na snimku pomoću IPP "alata" označene zeleno, odnosno crveno, čime je postignuta bolja rezolucija i oštiji kontrast između ispitivanih površina. Označavanjem izabrane površine, tj. odabirom boje, bilo je moguće kvantitativno odrediti odnos između oštećene i neoštećene površine, kao i stepen "oštećenja", tj. poroznost. Analizirano je najmanje 10 fotografija po uzorku da bi se dobila relevantna karakterizacija mikrostrukture. Površinska poroznost za svaki betonski uzorak je izračunata tako što je određen odnos oštećene i neoštećene površine.

experimental concrete. Aggregates had different granulations. Both types of concrete were prepared with high aluminate cement SECAR 70 (*Lafarge*). The chemical compositions of the investigated concretes are as follows: B sample ( $\text{Al}_2\text{O}_3$  – 62.88 %,  $\text{SiO}_2$  – 21.17 %,  $\text{CaO}$  – 8.26 %) and C sample ( $\text{Al}_2\text{O}_3$  – 93.62 %,  $\text{SiO}_2$  – 0.07 %,  $\text{CaO}$  – 5.97 %).

## 2.2 Mechanical compressive strength

The mechanical compressive strength of refractory concretes C and B was experimentally determined according to standard laboratory procedure [15]. Sixty cubic samples of each series (10 cubes for each temperature: 20, 110, 800, 1000, 1300 and 1500 °C) with identical dimensions (10 cm x 10 cm x 10 cm) were investigated. After 7 days of curing in climate chamber (at 20 °C), samples were demoulded and stored for another 21 days under the same conditions as in the climate chamber. After 28 days, samples were dried at 110 °C for following 24 hours. Afterwards samples were transferred into electric furnace and fired at following temperatures: 800, 1000, 1300 and 1500 °C in groups of ten samples and with delay of 4 hours on each temperature for each group of samples. Each group of concrete specimens was tested on mechanical compressive strength using conventional laboratory hydraulic pressure device. Same samples were previously tested with UPV method and screened for IPP analysis.

## 2.3 Apparent porosity

Apparent porosity of the refractory concrete samples was investigated with optical microscope (Olympus, CX31-P) accompanied with PC program for image analysis. The Image Pro Plus (IPP) program (Materials Pro Analyzer, Version 3.1, Media Cybernetics, Silver Spring, MD, USA) was used in the experiment. Digital photographs of the samples surface were taken before each thermal treatment and then after compressive strength testing (same samples from compressive strength testing). The specimens were covered with thin chalk-powder film before surface damage was investigated. The area without damage is colored by the chalk powder, while the damaged areas keep the basic color of the refractory materials. The film provided better contrast and differentiation of damaged and non-damaged surfaces. Different (damaged and non-damaged) surfaces of the samples were marked with different colors using IPP tools - green and red. Thus, higher resolution and sharper difference in damaged and non-damaged surfaces on the specimens could be obtained. When the appropriate color is selected, it is possible to quantitatively measure the ratio and level of damaged and non-damaged areas by means of image analysis using a statistical approach. At least 10 photographs per sample were analyzed in order to obtain a reliable characterization of the microstructure. The ratio between sample surface area and damaged surface area was calculated for each refractory concrete sample and, thus, surfacial apparent porosity was determined.

## 2.4 Nedestruktivna metoda ispitivanja – metoda brzine ultrazvuka

Metoda merenja brzine ultrazvuka služi za nedestruktivni monitoring i karakterizaciju svojstava industrijskih materijala. U ovom eksperimentu je primjenjen komercijalni ultrazvučni aparat PUNDIT plus PC1006, CNS Farnell Ltd., Hertfordshire, England. Instrument je opremljen sa generatorom ultrazvučnih talasa, osciloskopom za merenje vremena i dve merne sonde (220 kHz) koje su ručno postavljene na suprotne krajeve svakog od uzoraka. Sonde su na svojim dodirnim površinama imale 2 mm debeli gumeni "poklopac" radi boljeg naleganja merne sonde na hrapavu površinu uzorka betona. Longitudinalni ultrazvučni talasi se grenerišu u elektro-akustičnom provodniku koji je smešten u okviru jedne od sondi. Talasi koji putuju kroz uzorak na kraju puta bivaju "pretvoreni" u električnu energiju pomoću prijemnika smeštenog u drugoj sondi. Standardi predlažu tri načina dispozicije mernih sondi: (1) sonde su na suprotnim, paralelnim stranama uzorka – direktna transmisija, (2) sonde su smeštene dijagonalno jedna u odnosu na drugu, u suprotnim uglovima paralelnih strana uzorka – dijagonalna transmisija i (3) sonde se nalaze na istoj strani uzorka na određenom rastojanju – indirektna transmisija. U okviru ovog eksperimenta primenjena je metoda direktne transmisije kao najpreciznija i najpouzdanija.

Sonde su smeštene na dve paralelne strane betonskog uzorka. Ispitano je 60 uzoraka svake serije betona istih dimenzija (10 cm x 10 cm x 10 cm). Svaki uzorak vatrostalnog betona je prozvučavan i po dužini i po visini. Zbog postizanja adekvatne preciznosti svako ultrazvučno merenje je ponovljeno bar pet puta. Uzorci, na kojima su izvršena ultrazvučna merenja, dalje su poslužili u eksperimentu određivanja pritisne čvrstoće destruktivnim putem. Ultrazvučna merenja su obavljena u skladu sa aktuelnim standardima [16].

Brzina ultrazvuka kroz uzorak ( $v$ ) je izračunata iz poznatog rastojanja između mernih sondi i poznatog tranzitnog vremena ultrazvuka određenog pomoću osciloskopa:

$$V = \frac{l}{t} \quad (\text{m/s}) \quad (2)$$

gde je:  $l$  – rastojanje između sondi, (m) i  $t$  vreme prolaska ultrazvučnih talasa, (s).

Dinamički modul elastičnosti se može odrediti iz poznate zapreminske mase uzorka density (ASTM C-134), Poasonovog koeficijenta za ispitivani materijal i poznate brzine ultrazvuka na sledeći način:

$$E_{dyn} = v^2 \cdot \rho \frac{(1 + \mu_{dyn}) \cdot (1 - 2\mu_{dyn})}{(1 - \mu_{dyn})} \quad (\text{GN/m}^2) \quad (3)$$

gde je:  $v$  brzina ultrazvuka, (m/s),  $\rho$  zapreminska masa, ( $\text{kg}/\text{m}^3$ ) i  $\mu_{dyn}$  dinamički Poasonov koeficijent.

Pritisna čvrstoća se može aproksimativno izračunati na osnovu dobijenih vrednosti brzine ultrazvuka kako je to prikazano empirijskom jednačinom (4):

## 2.4 Nondestructive testing method - Ultrasonic Pulse Velocity

Ultrasonic pulse velocity (UPV) is important method because it can characterize and monitor the properties of an industrial material nondestructively. A commercial ultrasonic testing instrument of transmission type (PUNDIT plus PC1006, CNS Farnell Ltd., Hertfordshire, England) was used in the experiment. The instrument is equipped with a pulse generator and timing circuit coupled to two transducers (220 kHz) that were positioned manually at opposite ends of each specimen. Each transducer had a 2 mm thick rubber tip to help overcome measurement problems due to the roughness of the refractory surface. Pulses of longitudinal elastic stress waves are generated by an electro-acoustical transducer which is held in direct contact with the surface of the testing sample. The pulses are traveling through the material and at the end of the path they are received and converted into electric energy by a second transducer. Most standards describe three possible arrangements for the transducers: (1) the transducers are located directly opposite to each other (direct transmission), (2) the transducers are located diagonally to each other, that is, the transducers are across corners (diagonal transmission), and (3) the transducers are attached to the same surface and separated by a known distance (indirect transmission). Direct transmission is the most sensitive and it was applied in this experiment.

The transducers were placed on two parallel surfaces of specimens. Sixty refractory specimen of each series with identical dimensions (10 cm x 10 cm x 10 cm) were investigated. For each refractory specimen, measurements of ultrasonic pulse velocity through the length and thickness on direct transmission disposition were performed. Each test was run at least five times to correctly validate the ultrasonic velocity. Same specimens as in compressive strength investigation were used in UPV analysis. Testing was performed according to valid standards [16].

The ultrasonic pulse velocity ( $v$ ) is calculated from the distance between the two transducers and transit time of the pulse measured by an oscilloscope as:

where:  $l$  is the stress wave path length, (m) and  $t$  is the transit time, (s).

Dynamic modulus of elasticity can be determined if bulk density (ASTM C-134), the Poisson's ratio and ultrasonic velocity of a refractory material are known:

where:  $v$  is the pulse velocity, (m/s),  $\rho$  the bulk density, ( $\text{kg}/\text{m}^3$ ) and  $\mu_{dyn}$  is the dynamic Poisson ratio.

Mechanical compressive strength can also be approximately calculated from obtained values of ultrasonic velocity as it is shown by equation (4):

$$S = S_0 \left( \frac{v}{v_0} \right)^n \text{ (MPa)} \quad (4)$$

gde je:  $S_0$  - pritisna čvrstoća materijala pre izlaganja termičkom tretmanu;  $v$  - brzina ultrazvuka nakon ispitivanja čvrstoće, (m/s);  $v_0$  - brzina longitudinalnih ultrazvučnih talasa pre ispitivanja čvrstoće, (m/s) i  $n$  - konstanta materijala ( $n = 0,488$ ) usvojena kao srednja vrednost za obe vrste betona na osnovu literature [17, 18].

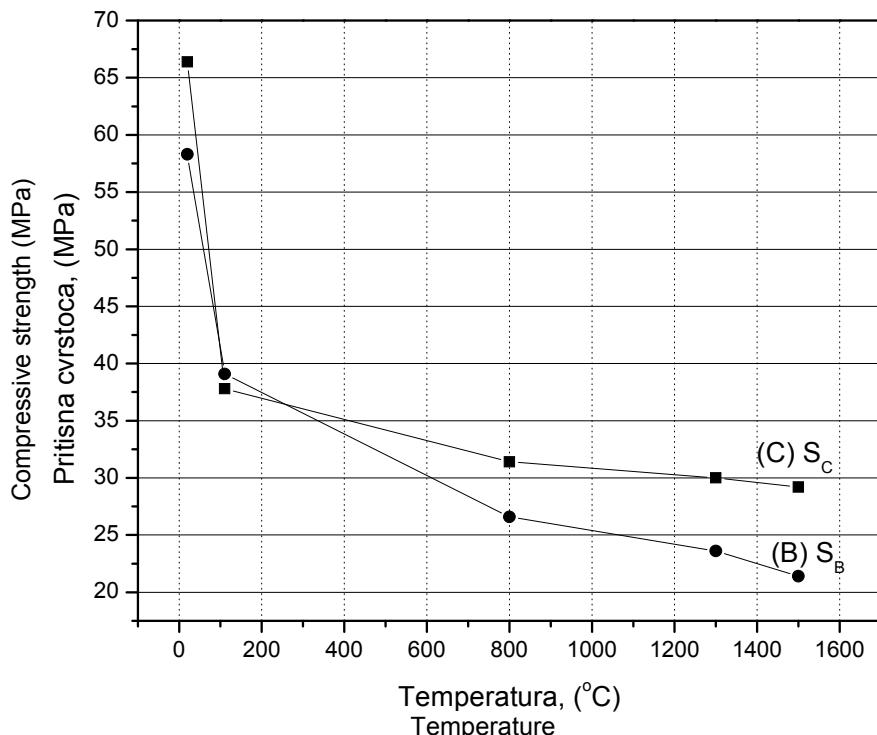
### 3 REZULTATI I DISKUSIJA

Srednje vrednosti čvrstoće pri pritisku -  $S$ , (MPa) određene destruktivnom metodom (opisanom u poglavljiju 2.2) na uzorcima betona C i B prikazane su na slici 1.:

where:  $S_0$  is compressive strength before exposure of the material to thermal treatment, (MPa);  $v$  is longitudinal ultrasonic velocity after testing, (m/s);  $v_0$  is longitudinal ultrasonic velocity before testing, (m/s) and  $n$  is the material constant ( $n = 0,488$ ) accepted from literature for and taken as average for both materials [17, 18].

### 3 RESULTS AND DISCUSSION

Average values of the mechanical compressive strength -  $S$ , (MPa) for C and B samples, obtained by destructive method described in section 2.2 are presented in Fig.1.:



Slika 1. (C) Degradacija pritisne čvrstoće SC vatrostalnog betona C sa porastom temperature; (B) Degradacija pritisne čvrstoće SB vatrostalnog betona B sa porastom temperature

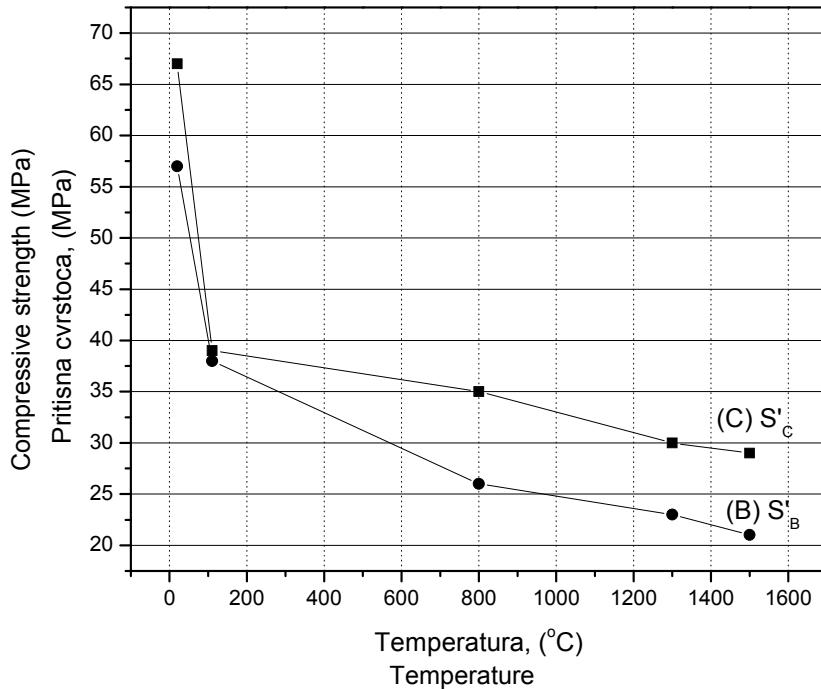
Fig. 1. (C) Average values of mechanical compressive strength degradation -  $S_C$ , (MPa) of refractory concrete C; (B) Average values of mechanical compressive strength degradation -  $S_B$ , (MPa) of refractory concrete B

Dijagram prikazan na slici 1. prikazuje degradaciju (smanjenje) pritisne čvrstoće betona koja je prouzrokovana povećanjem temperature. Kao što se može uočiti na dijagramu, uzorak C ima veću početnu čvrstoću pri pritisku (na 20 °C), ali i veću finalnu pritisnu čvrstoću (na 1500 °C) nego beton B. Razlike u pritisnoj čvrstoći na 20 i 1500 °C su 12,2 i 26,7 %, respektivno. Degradacija pritisne čvrstoće je upravo posledica promene mikrostrukture kompozita sa povećanjem temperature i ukazuju na postojanje defekata u istoj. Očigledno je da se kod uzorka betona C degradacija sporije odvija u intervalu termičkog tretmana od 110 °C do 1500 °C, nego što je to slučaj kod betona B. Dakle, postoji razlika na mikrostukturalnom nivou kod ova dva tipa betona, a samim tim javlja se razlika u toku razvoja i promene u

Diagram presented on Fig. 1. describes mechanical compressive strength degradation caused by increasing temperature. As it can be seen, C sample shows higher initial mechanical compressive strength (at 20 °C) and also higher final strength (at 1500 °C) than B concrete. Difference in strength values (at 20 and 1500 °C) are 12.2 and 26.7 %, respectively. Existence of compressive strength degradation points out to a microstructural change occurring within composites. It is evident that C sample shows slower rate of strength degradation than B concrete during thermal treatment (from 110 °C to 1500 °C). Thus, there are differences in the development of microstructure of these two concrete samples, probably due to the better choice of grain-size distribution in case of C sample.

mikrostrukturi. Razlog boljeg "ponašanja" betona C može biti bolje izabran tip agregata i granulometrijski sastav agregata.

Slika 2. prikazuje srednje vrednosti pritisne čvrstoće određene nedestruktivnom metodom merenja brzine ultrazvuka. Pritisna čvrstoća  $S'$ , (MPa) je izračunata na osnovu poznate vrednosti  $v_p$ , (m/s) za svaki od uzoraka betona (opisano u poglaviju 2.4, jednačina (4)). Vrednosti dobijene za pritisnu čvrstoću na ovaj način su aproksimativno iste kao i vrednosti pritisnih čvrstoća određenih destruktivnom metodom.



*Slika 2. (C) Degradacija pritisne čvrstoće  $S'_C$  vatrostalnog betona C sa povećanjem temperature; (B) Degradacija pritisne čvrstoće  $S'_B$  vatrostalnog betona B sa povećanjem temperature. Čvrstoće su određene metodom brzine ultrazvuka*

*Fig. 2. (C) Average values of mechanical compressive strength degradation -  $S'_C$ , (MPa) of refractory concrete C; (B) Average value of mechanical compressive strength degradation -  $S'_B$ , (MPa) of refractory concrete B. Both group of values are calculated using UPV testing method*

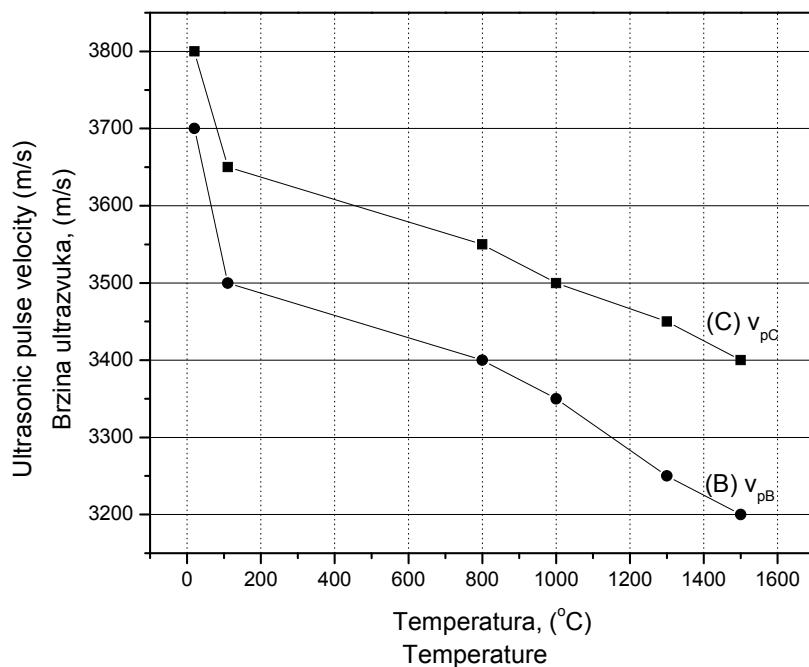
Slika 3. prikazuje zavisnost između srednjih vrednosti brzine ultrazvuka -  $v_p$ , (m/s) merenih kroz različite uzorce vatrostalnih betona (C i B) i povećanja temperature -  $T$ , (°C).  $v_p$  je izračunata primenom jednačine (2) za svaki od uzoraka, a potom su određene srednje vrednosti.

Ako se dijagrami na slikama 1. i 2. uporede sa dijagrom na slici 3., može se zaključiti sledeće: manja vrednost pritisne čvrstoće odgovara manjoj vrednosti brzine ultrazvuka. Razlog smanjenja čvrstoće pri pritisku i brzine ultrazvuka je upravo u degradaciji koja se dešava na mikrostrukturnom nivou u betonu. U ovom slučaju dolazi do povećanja poroznosti. Dakle, metoda brzine ultrazvuka se može upotrebiti za praćenje promene poroznosti u betonskim uzorcima umesto standardne laboratorijske metode (na primer živinog porozimetra), a naročito u onim slučajevima kada za eksperiment nije neophodno znati tačnu vrednost poroznosti.

Fig. 2. presents average values of mechanical compressive strength -  $S'$ , (MPa) calculated from  $v_p$ , (m/s) – ultrasonic pulse velocity, which was obtained using non-destructive testing method (UPV -described in 2.4 and formula (4)). Values of mechanical strength obtained by both methods are approximately same and they lay close to one another which justify application of UPV. It provides data which are approximately same as regular destructive testing method.

Fig. 3. shows dependence between calculated average values of ultrasonic pulse velocity -  $v_p$ , (m/s) measured through various concrete specimen (C and B) and increasing temperature -  $T$ , (°C).  $v_p$  was calculated using formula (2) for each specimen and average value was determined afterwards.

If diagrams on Fig. 1. and Fig. 2., were compared with diagram on Fig. 3., conclusion could be made about correlation between composites compressive strength and ultrasonic pulse velocity: lower value of compressive strength means slower rate of ultrasound pulse. Reason for decreasing of mechanical compressive strength and ultrasonic pulse velocity is degradation which occurs within concrete microstructure, i.e. increasing level of porosity. Thus, UPV method can be used as a means of the monitoring of changes in porosity instead of classic laboratory methods (for example Mercury porosimeter), when precise level of apparent porosity is not necessary to be known for an experiment.

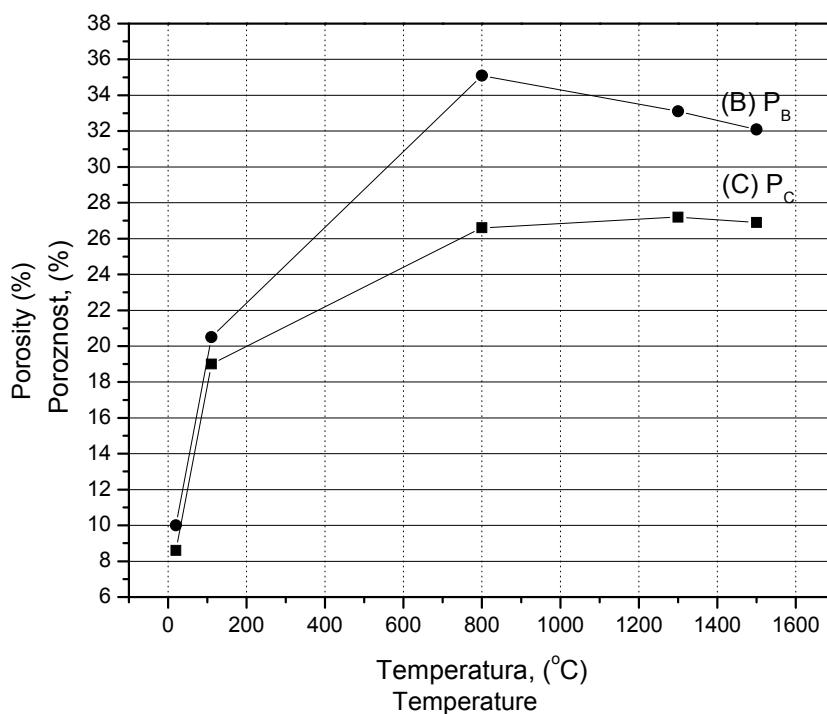


Slika 3. (C) Promena brzine ultrazvuka  $v_{pc}$  kroz beton C sa povećanjem temperature; (B) Promena brzine ultrazvuka  $v_{pb}$  kroz beton B sa povećanjem temperature

Fig. 3 (C) Average values of ultrasonic pulse velocity -  $v_{pc}$ , (m/s) of refractory concrete C; (B) Average values of ultrasonic pulse velocity -  $v_{pb}$ , (m/s) of refractory concrete B

Rezultati promene srednje vrednosti pravidne poroznosti -  $P$ , (%) u funkciji povećanja temperature, za uzorke betona C i B, dobijeni primenom IPP metode (postupak objašnjen u poglavlju 2.3.) su prikazani na slici 4.

Results of change of average apparent porosity -  $P$ , (%) in function of increasing temperature, for C and B samples, obtained by IPP method (described in chapter 2.3.) are given in Fig. 4.



Slika 4. (C) Promena poroznosti PC za beton C; (B) Promena poroznosti PB za beton B. Poroznost određena IPP metodom

Fig. 4. (C) Change of average apparent porosity -  $PC$ , (%) of refractory concrete C; (B) Change of average apparent porosity -  $PB$ , (%) of refractory concrete B. Both diagrams obtained by applying IPP metod

Beton C ima manju vrednost poroznosti nego beton B na svim temperaturama ispitivanja (od 20 do 1500 °C). Ovakav odnos poroznosti je najverovatnije posledica bolje izabranog granulometrijskog sastava i boljih parametara projekta betonske mešavine (vodočementni faktor, hemijski sastav, itd.). Razlika u konačnim vrednostima poroznosti nije zanemarljiva: prividna poroznost uzorka betona B je 16.2 % veća nego adekvatna vrednost poroznosti kod betona C. Ovakav odnos vrednosti poroznosti opravdava pretpostavku o uzrocima većeg smanjenja pritisne čvrstoće u slučaju betona B, tj. veća poroznost uslovljava manju pritisnu čvrstoću.

Na slici 4. se na oba dijagrama (i u slučaju betona B i betona C) uočava pik (ekstremum) na 800 °C. Pomenuti pik odgovara početku procesa sinterovanja u betonu. Naime, kada vatrostalni beton prolazi kroz termički tretman dolazi do sinterovanja na određenoj temperaturi. Ovo se obično dešava u temperaturnom intervalu od 800 do 900 °C. Posledice sinterovanja su: smanjenje poroznosti, denzifikacija materijala, povećanje pritisne čvrstoće kao posledica manje poroznosti i veće gustine, rekristalizacije i polimorfne transformacije, itd. U slučaju ispitivanih vatrostalnih betona C i B, vatrostalnost je visoka, tako da se značajno smanjenje poroznosti i povećanje pritisne čvrstoće ne mogu uočiti ispod 1500 °C.

IPP metodom su određeni parametri kao što su: maksimalni, minimalni i srednji prečnik pore ( $D_{\max}$ ,  $D_{\min}$ ,  $D_{av}$ ), sferičnost pora (R) i broj pora (N) smeštenih unutar površinski vidljivih (otvorenih) pora. Rezultati su prikazani u tabeli 2. Prema IPP analizi, zaključeno je: srednji prečnik pore se povećava od 0,0067 mm (za beton C) i 0,003 mm (za beton B) do 0,0089 mm (C) odnosno 0,004 mm (B) mereno na 1300°C. Nakon toga dolazi do skupljanja pora, što je posledica procesa sinterovanja i reakcija u čvrstoj fazi. Na uzorku B je utvrđena manja vrednost srednjeg prečnika pore, iako je poroznost betona B veća nego betona C na svim temperaturama ispitivanja. Ovo je posledica izbora granulometrijskog sastava agregata, tj. u slučaju betona B je primenjen veoma sitan agregat – punioc (šamotno bračno). Idealna sferičnost pora je 1,00, a za ispitivanje betone sferičnost pora se kreće od 1,07 do 1,17, što znači da su pore prilično pravilnog oblika. Vrednost N je manja u slučaju betona C, što takođe indicira da je prividna poroznost betona C manja od poroznosti betona B i da je većina pora otvorenog tipa.

Porosity of C concrete is lower than porosity of B concrete at all investigated temperatures (from 20 to 1500 °C), probably due to better grain-size distribution and better mix design parameters of C concrete. Difference in final apparent porosity value is significant: apparent porosity of B sample is 16.2 % higher than adequate apparent porosity of C sample. This justifies and explains assumption about cause of higher degradation of compressive strength of B samples, i.e. higher porosity means lower mechanical strength.

A peak on both diagrams (in case of C and B samples) at 800 °C can be noticed on Fig. 4. The peak corresponds to the beginning of the sintering process. Namely, when refractory concrete undergoes a thermal treatment, the sintering process occurs at certain temperature. That usually happens in temperature interval from 800 to 900 °C. Consequence of sintering are: decreasing of porosity, material getting thicker, compressive strength increasing as a result of lower porosity and higher density, etc. However, in this case, both composites (C and B) have high refractoriness, thus significant downfall of porosity and increment of compressive strength are delayed for thermal interval above 1500 °C.

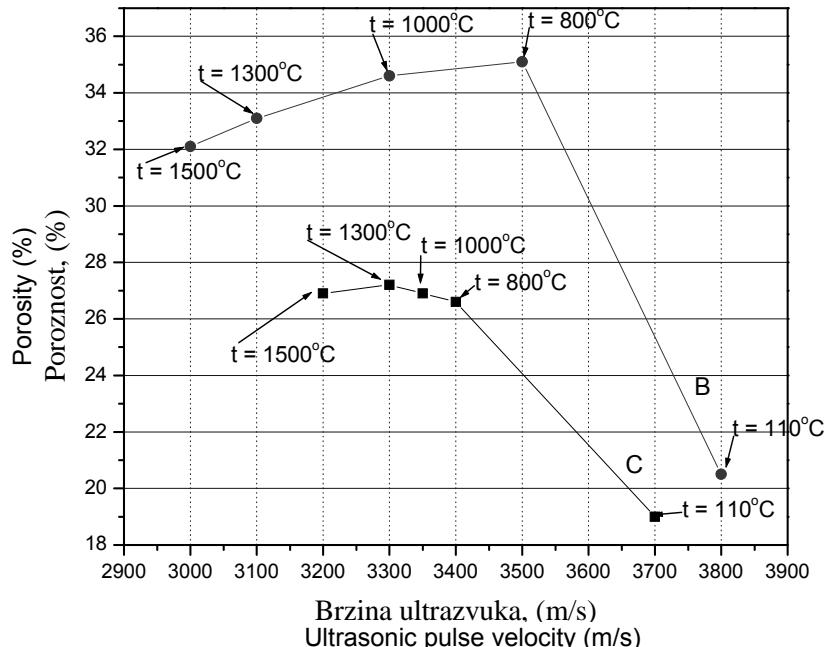
IPP also provides other parameters such as maximal, minimal and average pore diameter ( $D_{\max}$ ,  $D_{\min}$ ,  $D_{av}$ ), pore roundness (R) and number (N) of pores situated within superficial pores. Results are presented in Tab. 2. According to IPP analysis: average pore diameter increases from 0.0067 mm (for C concrete) and 0.003 mm (for B concrete) up to 0.0089 mm (C) and 0.004 mm (B) at 1300°C temperature. Afterwards pore shrinkage occurs, which is consequence of sintering process. B sample has smaller average pore diameter although its apparent porosity is higher on all temperatures of investigation. It is consequence of choice of aggregate granulation, i.e. in case of B concrete very fine chamotte aggregate (often referred as "chamotte flour") was used as the filler. Ideal pore roundness would be 1.00. For investigated concretes pore roundness is 1.07 to 1.17, which means that pores are almost spherical. N is smaller for C sample, which also indicates that apparent porosity of C concrete is lower than in case of B concrete, i.e. most of pores are superficial.

Tab. 2. Rezultati Image Pro Plus analize betona C i B  
Tab. 2. Results of Image Pro Plus analysis for C and B concrete samples

T (°C)	beton: concrete:	$D_{\max}$ (mm)	$D_{\min}$ (mm)	$D_{av}$ (mm)	N	R
110	C	0.046	0.0042	0.0067	9	1.08
800		0.057	0.00448	0.0077	14	1.10
1000		0.072	0.0045	0.0084	22	1.13
1300		0.089	0.0046	0.0089	26	1.14
1500		0.084	0.00455	0.0086	24	1.09
110	B	0.056	0.00129	0.003	51	1.07
800		0.073	0.00137	0.0035	74	1.12
1000		0.079	0.00138	0.0037	81	1.14
1300		0.085	0.00138	0.004	80	1.22
1500		0.082	0.00130	0.0038	75	1.17

Metoda brzine ultrazvuka je primenjena pri ispitivanju uzoraka vatrostalnih betona da bi se utvrdili mogući strukturni defekti i prisustvo pora i da bi se potvrdili parametri kvaliteta betona kao što je pritisna čvrstoća. S obzirom na određenu temperaturu ispitivanja može se zaključiti koji od vatrostalnih betona ima manju količinu mikrostrukturnih defekata i manju vrednost poroznosti. U pitanju je uvek beton koji za koji je dobijena veća brzina ultrazvuka – u ovom slučaju beton C. Zavisnost poroznosti i brzine ultrazvuka je prikazana na slici 5.

UPV non-destructive testing method was applied on concrete samples in order to investigate possible structural defects and presence of pores and to confirm parameters such as mechanical strength. Regarding specific temperature of investigation, it can be concluded which type of concrete has smaller amount of defects and lower porosity on the specific temperature of the investigation. It is the sample with higher rate of ultrasound – in this case C concrete sample. Function porosity – ultrasound pulse velocity is given in Fig. 5.



Slika 5. Zavisnost poroznosti i brzine ultrazvuka za betone C i B u funkciji temperature

Fig. 5. Change of apparent porosity and ultrasonic pulse velocity in function of temperature of refractory concrete C and B

Kao što se može zaključiti sa slike 5., veća poroznost uzorka ukazuje na manju brzinu ultrazvuka. Veza koja postoji između pritisne čvrstoće i brzine ultrazvuka je zapravo obrnuta korelacija poroznost – brzina ultrazvuka. Prethodno navedeno potvrđuje rezultate dobijene IPP metodom i metodom brzine ultrazvuka i ukazuje da su ove dve metode kompatibilne sa standardnim laboratorijskim metodama. Dakle, IPP metoda i metoda brzine ultrazvuka su pouzdane i mogu se primeniti pri ispitivanju poroznosti i pritisne čvrstoće, kao i za praćenje i predviđanje ponašanja vatrostalnog betona.

#### 4 ZAKLJUČCI

U ovom radu su ispitivana mehanička svojstva i karakteristike mikrostrukture vatrostalnih betona. Metoda brzine ultrazvuka je primenjena u cilju praćenja promene pritisne čvrstoće vatrostalnih betona sa povećanjem temperature. Prividna poroznost, raspored pora i veličina pora su određeni pomoću Image Pro Plus programa. Rezultati koji su izloženi u okviru ovog rada doprinose ideji o uključivanju drugih metoda u ispitivanje standardnih mehaničkih svojstava betona, kao što su nedestruktivne metode. Treba napomenuti da se metoda ultrazvuka, iako se već dugo primenjuje u različitim oblastima

As it can be concluded from Fig. 5., higher porosity of a sample implicates lower ultrasound velocity. Correlation between mechanical strength and ultrasonic velocity is reverse correlation between porosity and ultrasonic velocity to certain extent. Regarding the fact that results for porosity are obtained by IPP and result for mechanical strength with UPV (compatible with laboratory method) it can be concluded that using these two methods mentioned correlation between mechanical strength and porosity is confirmed and these methods can be used in monitoring and predicting behavior of a material.

#### 4 CONCLUSIONS

Mechanical properties and microstructure of refractory composites were investigated in this paper. UPV method was used to determine ultrasonic velocity and to monitor the compressive strength of refractory composite. Apparent porosity, pore distribution and pore size of concrete samples were investigated using Image Pro Plus program. Results presented in this paper contribute to the idea of including other testing methods in investigation of mechanical properties such are non-destructive methods instead of commonly used standard laboratory procedures. It should be noted that non-destructive testing methods, such as UPV, were rarely

nauke, vrlo retko koristila za ispitivanje svojstava vatrostalnih betona. Prednosti ove metode su: nedestruktivna metoda, jednostavna, brza i pouzdana, isti uzorci se mogu koristiti i za dalja ispitivanja jer ne bivaju oštećeni, postoji finansijska ušteda u materijalu i vremenu spravljanja uzorka, itd. Rezultati dobijeni ultrazvučnom metodom se mogu dovesti u vezu sa rezultatima drugih metoda: prividna poroznost, čvrstoća pri pritisku, itd. Metoda brzine ultrazvuka se može primeniti za predviđanje i praćenje ponašanja konstrukcionih elemenata od betona i vatrostalnih obloga u metalurškim pećima. Prednost ove metode je u tome što ne postoji potreba za oštećivanjem vatrostalne obloge peći radi vađenja kernova. Prednosti primene analize slike su sledeće: IPP analiza omogućava ispitivanje novog spektra svojstava i dolazak do informacija do kojih nijedna druga metoda prethodno nije mogla doći – strukturalni defekti, površinska poroznost, precizan prečnik pore, oblik pore, broj pore u unutar otvorenih pora, itd. Kada je poznat nivo površinskog oštećenja vatrostalnog betona, ti podatci se mogu iskoristiti za predviđanje ponašanja uzorka u toku daljeg ispitivanja ili, pak, ponašanja materijala ugrađenog u metaluršku pet ili termo-oblogu. U toku IPP analize i primene ultrazvučne metode zaključeno je da eksperimentalni korundni vatrostalni beton (C) ima bolje mehaničke i termičke karakteristike nego komercijalno dostupni beton sa boksim (B) usled boljih parametara mešavine i bolje izabranog granulometrijskog sastava.

#### Zahvalnost

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used in area of refractory concrete investigation, thus it represents a new method, although UPV has a long history of application in different researches. Benefits from using ultrasonic measurements are: it is non-destructive, simple, fast and reliable method; same samples could be used for further tests; there is financial benefit in minimizing number of samples for testing – saving in material and in time; obtained results and parameters like for example ultrasonic pulse velocity can be correlated with the results obtained by other methods: apparent porosity, mechanical strength, etc. UPV can be used for prediction of refractory concrete behavior and also in monitoring of behavior of structural concrete elements and refractory linings of metallurgical furnaces. Advantage of this method is in the fact that there is no necessity to damage the lining of furnace in order to investigate requested parameters. Benefits from using image analysis are also numerous: it provides entirely new and important information about structural damages and surface porosity like, for example precise diameters of pores, pore roundness, number of pores in a section, etc.; As surfacial damage level is measured, results could be useful for prediction of sample behavior during further testing or application in a metallurgic furnace or in thermal insulation. During UPV and IPP testing it was also concluded that experimental refractory concrete (in text referred as C – corundum concrete) possesses better mechanical and thermal characteristics than commercially available bauxite concrete, due to its better aggregate properties and granulation and better mix parameter design.

#### Acknowledgement

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## REZIME

### VEZA IZMEĐU MEHANIČKIH SVOJSTAVA I MIKROSTRUKTURE VATROSTALNIH KOMPOZITA ODREĐENA NEDESTRUKTIVNIM METODAMA ISPITIVANJA

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Ljubica PAVLOVIĆ  
Zagorka RADOJEVIĆ

Cilj ovog rada je da se utvrdi veza između važnih mehaničkih svojstava i karakteristika mikrostrukture pomoću nedestruktivne ispitne metode – metode merenja bezine ultrazvuka, na uzorcima korundnih i boksitnih vatrostalnih betona. Ispitivani betoni se razlikuju po hemijskom i mineraloškom sastavu. Standardnom destruktivnom laboratorijskom metodom je određena mehanička čvrstoća pri pritisku na uzorcima koji su bili prethodno izloženi termičkom tretmanu na temperaturama: 110, 800, 1000, 1300 i 1500 °C. Kada je uzorak vatrostalnog betona izložen uticaju povišene temperature i statickog opterećenja dolazi do smanjenja pritisne čvrstoće i do sveukupne degradacije materijala (gustina i elastična svojstva se smanjuju). Određivanjem mehaničkih svojstava betona može se ukazati ili se mogu pratiti promene koje se dešavaju unutar mikrostrukture materijala. Nivo površinske degradacije uzorka, nakon termičkih tretmana, je utvrđen pomoću optičkog mikroskopa i Image Pro Plus – kompjuterskog programa za analizu slike. Nedestruktivna metoda merenja brzine ultrazvuka je primenjena pri praćenju promene poroznosti unutar uzorka vatrostalnog betona. Ultrazvučna metoda i analiza slike su pouzdane metode za karakterizaciju mikrostukturalnih promena i defekata i veoma su korisne kada je potrebno utvrditi koji je tip vatrostalnog betona najpogodniji za određenu primenu.

**Ključne reči:** mehanička svojstva, vatrostalni beton, poroznost, nedestruktivne ispitne metode.

## SUMMARY

### CORRELATION BETWEEN MECHANICAL PROPERTIES AND MICROSTRUCTURE OF REFRACTORY COMPOSITES DETERMINED USING NON-DESTRUCTIVE TESTING METHOD

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Aim of this paper is to establish the correlation between important mechanical properties and characteristics of microstructure using non-destructive testing method, i.e. ultrasonic pulse velocity, on the example of corundum and bauxite based refractory concretes. Investigated concretes are varying in chemical and mineralogical composition. Mechanical compressive strength of concrete samples after thermal treatment at various temperatures (110, 800, 1000, 1300 and 1500 °C) was investigated using standard laboratory procedure. When refractory concrete sample is subjected to increased temperature and compressive load loss of strength and material degradation occurs (density and elastic properties of material are decreasing). Measurement of mechanical properties can indicate and monitor the changes in the microstructure. Level of surface deterioration after thermal treatment was determined using optical microscope and Image Pro Plus - program for image analysis. Nondestructive ultrasonic measurement was used as a means of monitoring of increasing porosity in refractory specimens. Ultrasonic pulse velocity technique and image analysis are reliable non-destructive methods for characterization of micro-structural defects and can be useful when type of refractory concrete is to be chosen for an application.

**Key words:** mechanical properties, refractory concrete, porosity, non-destructive testing methods.