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# Thermal stability of cordierite/ silicon carbide composites after cyclic thermal shock

M. POSARAC*, M. DIMITRIJEVIC[a], T.VOLKOV -HUSOVIC[a], A.EGELJA, A. DEVECERSKI, B. MATOVIC
*Institute of Nuclear Sciences " Vinca", P.O. Box 522, 11001 Belgrade*
*[a]Faculty of Technology and Metallurgy, Karnegijeva 4, POB 3503*

In this paper composite material made from silicium carbide and cordierite was used in experiments. Behavior of the material in conditions of rapid temperature changes was investigated. Modified water quench test was applied to determine thermall stability behavior of the samples. Image analysis was used to monitored level of surface deterioration before and during quenching. Ultrasonic measurements were used for determination of dynamic Young modulus of elasticity and strength degradation during quenching. Obtained results are used for analysis of the behavior of the samples in conditions of rapid temperature changes and method for the improvement of the thermal stability behavior.

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*Keywords*: Ultrasonic velocity, Image analysis, Refractories, cordierite/SiC composite ceramics, Thermal shock resistance

## 1. Introduction

Thermal stability and acceptable thermal shock resistance together with satisfactory mechanical properties are the key requirements on the refractory materials. Cordierite has a superior thermal stability, thermal shock resistance and low thermal expansion coefficient. Silicon carbide has superior hardness, good chemical resistance, high values of conductivity, low values of thermal expansion coefficient, and excellent thermal stability and thermal shock resistance. The ceramic multi-component composite material could exhibit advantages of its constituents when the components have optimized properties and they are mixed in the proper ratio.

The thermal shock resistance is measured in terms of the number of cycles that a refractory material can withstand when subjected to sudden temperature changes [1].

When refractory materials are subjected to the industrial thermal cycles crack nucleation and propagation occurs resulting in loss of strength and material degradation. The formation of cracks decreases the velocity of ultrasonic pulses traveling in the refractory because it depends on the density and elastic properties of the material. Therefore measuring either of these properties can directly monitor the development of thermal shock damage level. Young's modulus of representative samples was calculated using measured values of ultrasonic velocities obtained by ultrasonic pulse velocity technique. Results were compared with water quench test data of thermal shock behavior of the investigated materials. The capability of the ultrasonic velocity technique for simple, sensitive, and reliable non-destructive characterization of thermal shock damage was demonstrated in this work. Thermal shock damage level was monitored before and during thermal quenching. Photographs of the samples were taken and level of destruction was monitored using Image Pro Plus Program.

The goal of this work is to use nondestructive testing methods and their advantages for prediction of the thermal shock behavior. Destruction of the samples was analyzed using the results of image analysis of the samples before and during thermal stability testing. In this paper the relationship between change in mechanical characteristics (Young modulus of elasticity and strength degradation) and behavior of the samples during thermal shock will be given.

## 2. Experimental

A mixture of commercially available spinel, quartz ($SiO_2$) and alumina ($Al_2O_3$) corresponding to a cordierite stoichiometry was attrition milled using $Al_2O_3$ balls and ethyl alcohol as media for four hours. Cordierite / SiC composite ceramics with weight ratio 30:70 and 50:50, respectively, were prepared by milling with $Al_2O_3$ balls in DI water in polyethylene bottle for 24 hours and firing at 1300ºC and 1250ºC, respectively.

Thermal stability of the refractories was determined experimentally by water quench test ( JUS. B. D8. 306.). Samples were cylinders with 1 cm diameter and 1 cm high. The samples were dried at 110 $^{O}$C and then transferred into an electric furnace at 950 $^{O}$C and held for 40 minutes. The samples were then quenched into water and left for 3 minutes and dried before returning to the furnace at 950 $^{O}$C. Failure is defined according to the standard test as total destruction of sample, or destruction of 50 and more percent of surface area before quenching. Experimental method is similar to the procedure described in PRE Refractory Materials Recommendations 1978 (PRE/R5 Part 2).

Ultrasonic pulse velocity testing (UPVT) was first reported being used on refractory materials in the late 1950's [4]. Various publications have dealt with the practical application of UPVT to characterize and monitor the properties of industrial refractory materials

non-destructively [4-10]. The UPVT method has been considered in detail in ref. [4]. Briefly, pulses of longitudinal elastic stress waves are generated by an electro-acoustical transducer that is held in direct contact with the surface of the refractory under test. After traveling through the material, the pulses are received and converted into electrical energy by a second transducer.

Most standards describe three possible arrangements for the transducers:

1) the transducers are located directly opposite each other (direct transmission),

2) the transducers are located diagonally to each other; that is, the transducers are across corners (diagonal transmission),

3) the transducers are attached to the same surface and separated by a known distance (indirect transmission).

The velocity, v, is calculated from the distance between the two transducers and the electronically measured transit time of the pulse as:

$$v(m/s) = \frac{L}{T} \tag{1}$$

where L is the path length (m) and T is the transit time (s). By determining the bulk density, the Poisson's ratio and ultrasonic velocity of a refractory material it is possible to calculate the dynamic modulus of elasticity using the equation below [5,10]:

$$E_{dyn} = v^2 \rho \left( \frac{(1+\mu_{dyn})(1-2\mu_{dyn})}{1-\mu_{dyn}} \right) \tag{2}$$

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

The measurement of ultrasonic velocity was performed using the equipment OYO model 5210 according to the standard testing procedure (JUS. D. B8. 121.). The transducers were rigidly placed on two parallel faces of the cylindrical sample having 1 cm diameter and 1 cm height using Vaseline grease as the coupling medium. The ultrasonic velocity was then calculated from the spacing of the transducers and the waveform time delay on the oscilloscope.

## 3. Results

Photographs of the samples were taken before and after water quench test. Samples surfaces were marked by different colors, in order to obtain a better resolution and difference in damaged and non/damaged surfaces in the material. For this investigation damage of the samples was monitored using Image Pro Plus Program, and results for material destruction, were given as function of number of quench experiments, N (fig.1.).

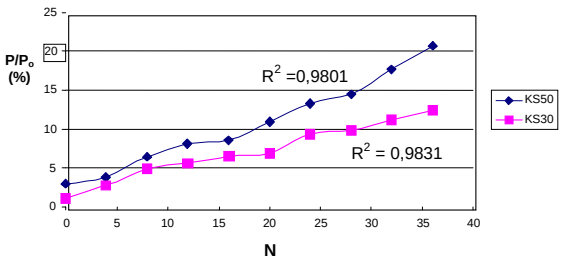


*Fig. 1. Damaged surface level ($P/P_o$) versus number of quench experiments (N).*

Results for changes of ultrasonic velocity during testing were presented at the Fig.2.a and Fig 2.b. for samples with different cordierite content.

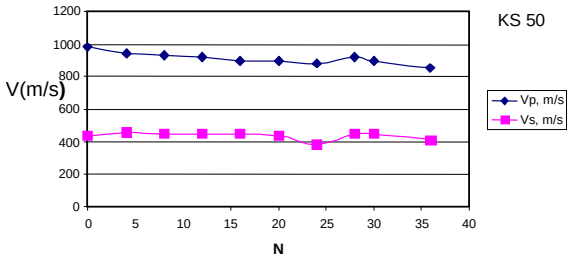


*Fig. 2.a. Ultrasonic velocity (V) during testing (longitudinal Vp and transversal Vs) versus number of quench experiments of KS 50 sample.*

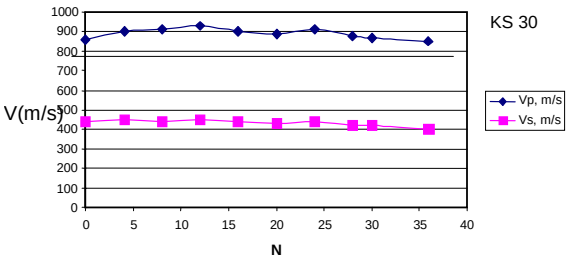


*Fig. 2.b. Ultrasonic velocity (V) during testing (longitudinal Vp and transversal Vs) versus number of quench experiments of KS 30 sample.*

Results for the velocity changes in both materials suggests that the samples were very stable during testing, as degradation of the velocity was not too below from the velocity of the sample before water quench test.

The expression for the strength degradation,σ, based on decrease in ultrasonic velocity was used [5,8,11]:

$$\sigma = \sigma_0 \left( \frac{V_L}{V_{L0}} \right)^n \tag{3}$$

where $\sigma_o$ is compressive strength before exposure of the material to the thermal shock testing, $V_L$ is longitudinal or ultrasonic velocity after testing, $V_{L0}$ is longitudinal or ultrasonic velocity before testing and $n$ is material constant ( $n$ = 0.488, ref. [5] ).

The strength degradation based on results of ultrasonic measurments, and calculated using equation (3) were presented at the Figs. 3.a. and 3.b.

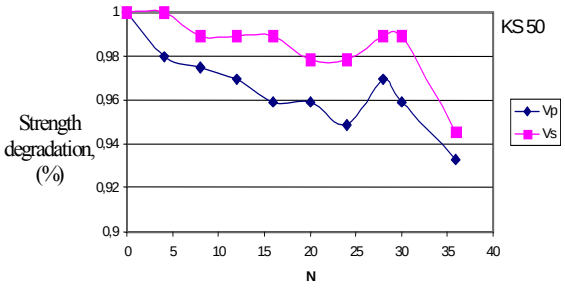


*Fig. 3.a. Strength degradation of material KS 50 versus number of quench experiment.*

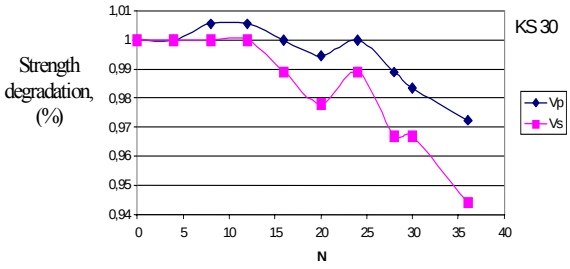


*Fig. 3.b. Strength degradation of material KS 30 versus number of quench experiment.*

The degradation at the end of the test was between 0. 93 and 0. 94 for KS50 samples and 0. 97 and 0. 94 for KS 30 samples after 36 cycles. These results indicate minimal strength degradation and explains excellent results for water quench test as result of 36 rapid temperature changes. Results for the monitoring changes of the Young modulus of elasticity during quenching are shown at the Figure 4. The Young modulus before testing indicates that material is porous, but degradation during testing was very stable, which explained 36 cycles of water quench test.

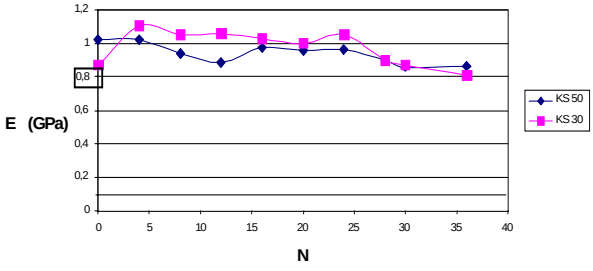


*Fig. 4. Dynamic Young modulus of elasticity versus number of quench experiments (N).*

## 4.Discussion

Two available systems of cordierite/silicon carbide refractory materials have been evaluated in this paper. The sample were subjected to the N cycles thermal shocks by water quench technique and subsequently the level of deterioration was evaluated accompanied with ultrasonic monitoring of Young modulus.

Both materials are excellent candidates for the application where thermal shock resistance is required. Water quench results showed that samples were stable till 36 cycles. Behavior of the samples was monitored during water quench test in order to determine damage of the original surface of the samples. During quenching damage of the original surface was not exceeded 50 %. Original surface showed damage about 20.7% for the KS 50 and 12.4 % for the KS 30 samples at the end of the experiment, after 36 cycles.

There are very small changes and degradation of the Young modulus during cycling. Thus the level of destruction in the bulk of the material and fracture nucleation and growth did not exceed level for material destruction.

## 5.Conclusions

Thermal stability of cordierite/silicon carbide composite ceramics was investigated.

Results pointed out the necessity of including other tests for thermal stability behavior analysis (beside water quench test), like image analysis and ultrasonic measurements in order to improve materials characterization.

## Acknowledgement

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*Corresponding author: milicap@vin.bg.ac.yu