Nondestructive Methods for Thermal Shock Characterization of Silicon Carbide / Cordierite Composite Material

M.Posarac[1, a], M.Dimitrijevic[2, b], J.Majstorovic[3, c], T.Volkov–Husovic[2, d], A.Devecerski[1, e] and B.Matovic[1, f]

[1]Institute of Nuclear Sciences " Vinca", P.O. Box 522, 11001 Belgrade

[2]Faculty of Technology and Metallurgy, Karnegijeva 4, POB 3503

[3]Faculty of Mining and Geology, Djusina 7, 1100 Belgrade

[a]milicap@vin.bg.ac.yu, b , [c] tatjana@tmf.bg.ac.yu , d, [e] drak@vin.bg.ac.yu , [f] mato@vin.bg.ac.yu

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Abstract. In the present work Mg-exchanged zeolit was used as starting material for obtaining cordierite/SiC composite ceramics with weight ratio 30:70 and 50:50. Behavior of composite ceramics after thermal shock treatments was investigated. Thermal shock of the samples was measured using standard laboratory procedure, water quench test (JUS.B.D8.306.). Level of surface deterioration before and during quenching was monitored by image analysis. Dynamic Young modulus of elasticity and strength degradation were determinate by ultrasonic measurements. It was found that SiC /cordierite composite ceramics has an excellent resistance to thermal shock.

Introduction

The knowledge of the thermal shock resistance of refractory materials is one of the most important characteristics since it determines their performance in many applications, from ceramic manufacturing to oil refinery lining, thermal insulation, nuclear power, chemical and petrochemical industries. 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 .

Thermal quenching of the refractories leads to the crack nucleation and/or crack propagation resulting in loss of strength. Since the formation of the cracks has a profound influence on the ultrasonic velocity and the Young's modulus of the material, measuring either of these properties may be applied to monitor the development of the thermal shock damage level.

The goal of this work is to use nondestructive testing methods and their advantages for prediction of thermal shock behavior. Destruction of the samples was analyzed using the Image Pro Plus Program. In this paper the relationship between change in mechanical characteristics (Young modulus of elasticity) and behavior of the samples during thermal shock will be given.

Materials

A mixture of Mg-exchange zeolite, alumina (Al_2O_3) and quartz (SiO_2) corresponding to a cordierite stoichiometry was attrition milled in ethyl alcohol media for 4 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 1160º C and 1100º C, respectively.

Experimental

Thermal Shock. Thermal stability of the refractories was determined experimentally by water quench test (JUS.B.D8.306.). Experimental method is similar to the procedure described in PRE Refractory Materials Recommendations 1978 (PRE/R5 Part 2). Geometry of the samples was changed, smaller samples were used for all experiments related to thermal shock behavior. Samples were cylinders with 1 cm diameter and 1 cm high. The samples were dried at 110 ºC and then transferred into an electric furnace at 950 ^oC and held for 40 minutes. The samples were then quenched into water and left for 3 minutes, dried before returning to the furnace at 950 ^oC. This procedure was repeated until failure, the number of quenches to failure was taken as a measure of a thermal shock resistance. Experimental method is similar to the procedure described in PRE Refractory Materials Recommendations 1978 (PRE/R5 Part 2).

Both of the materials exhibited excellent resistance to the rapid temperature changes. Samples were not damaged during test procedure till 36 cycles. According to the standard applied to the samples, procedure could be stopped if material is not damaged over 50% of original surface till 30 cycles. For the further investigations test procedure was performed till 36 cycles for both materials.

Detection of Damaged Surface Area in Refractory Specimen During Thermal Shock. Photographs of the samples were taken, before and after water quench test. When surface damage of the samples was investigated, samples were covered with the thin film made by chalk powder 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 ImagePro Plus Programe, and results for material destruction, were given as function of number of quench experiments, N (Fig. 1).

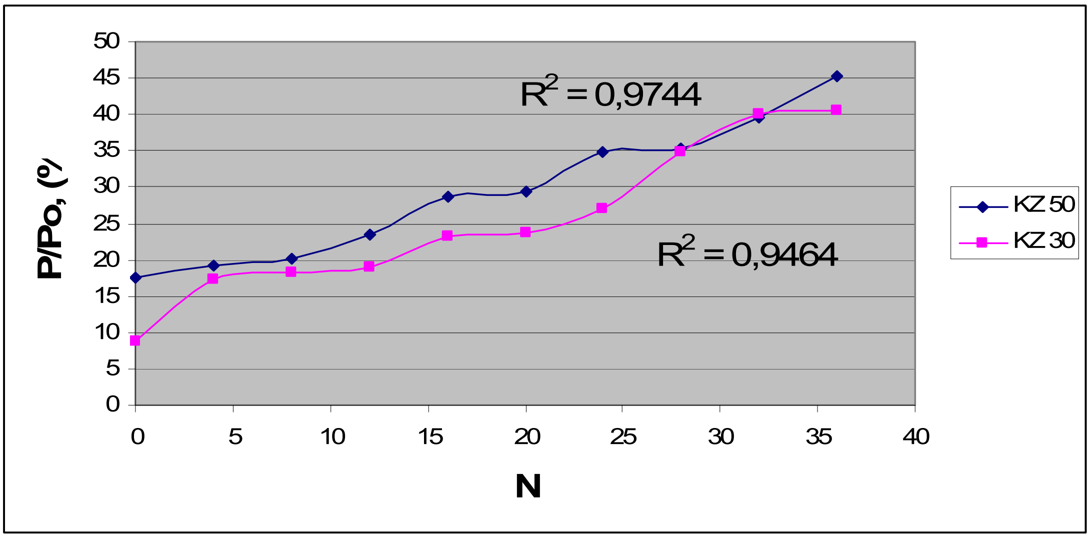

Fig. 1. Damaged surface level (P/P_o) versus number of quench experiments (N)

Nondestructive Determination of Dynamic Young Modulus of Elasticity. 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 5 cm diameter and 5 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. Dynamic Young modulus was calculated using the expression:

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

where: V_p – ultrasonic velocity of longitudinal waves (m/s), μ_{dyn} - dynamic Poisson ratio, γ - density (kg/m^3).

Results for the monitoring changes of the Young modulus od elasticity during quenching are shown at the Fig. 2.

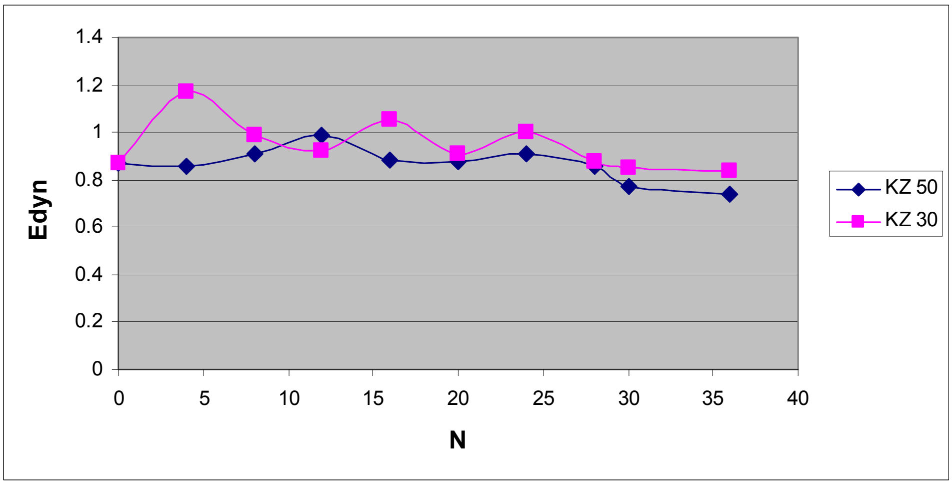

Fig. 2. Dynamic Young modulus of elasticity versus number of quench experiments (N)

Results and Discussion

Thermal shock behavior of the two materials was investigated. Three different techniques were applied:

- water quench test, as most popular experimental method,
- detection of damaged surface area in refractory specimen during thermal shock and
- nondestructive determination of dynamic Young modulus of elasticity

Obtained results showed that 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. Results given at the figure 1 showed that during quenching damage of the original surface was not exceed 50 %. Original surface showed damage about 17,6% for the KZ 50 and 8,8 % for the KZ 30. This damage before the test explains higher values for the damage at the end of the procedure, that had not overcome level 45 % at the end of the test, which is excellent result.

Behavior of the bulk of the sample was monitored using ultrasonic measurements of the Young modulus of elasticity. Results presented at the figure 2 showed very small changes and decrising of the Young modulus. This results are pointing out an that the level of destruction in the bulk of the material and fracture initation and growth did not exceed level for material destruction.

Conclusions

Two types of materials based cordierite/SiC composite ceramics were investigated. First was with weight ratio 30:70 (KZ30) and 50:50 (KZ50). Behavior of composite ceramics after thermal shock treatments was investigated. Water quench test as experimental method was applied, and additional experiments were performed in order to monitor behavior of the samples during water quench test. Obtained results given at the Fig. 1 showed that during quenching damage of the original surface was not exceed 50%. The damage before the test explains higher values for the damage at the end of the procedure, that had not overcome level 45 % at the end of the test, which is excellent result. Results for the Young modulus of elasticity confirmed that destruction of the samples during testing for the bulk of the material was minimal, as values for the Young modulus of elasticity exibit very small decreasing during testing.

All these results presented in this paper pointed out that both materials are an excellent candidates for applications where requirements for thermal stability behavior are high. Better results were obtained for material KZ 30, where level of destruction of the samples was between 8,8,% and 40,4 % before and at the end of testing. Material KZ 50 showed higher destruction before testing (17,8 %) as well as at the end of experiment (45,15 %).

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