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Determination of thermal shock resistance of silicon carbide/cordierite composite material using nondestructive test methods

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Abstract

In the present work Mg-exchanged zeolit and silicon carbide were used as starting materials 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.D8.319.). Level of surface deterioration before and during quenching was monitored by image analysis. Ultrasonic measurements were used as nondestructive quantification of thermal shock damage in refractory specimens. When refractory samples are subjected to the rapid temperature changes crack nucleation and propagation occurs resulting in loss of strength and materials degradation. The formation of cracks decreases the density and elastic properties of material. Therefore measuring these properties can directly monitor the development of thermal shock damage level. Dynamic Young modulus of elasticity and strength degradation were calculated using measured values of ultrasonic velocities obtained by ultrasonic measurements. Level of degradation of the samples was monitored before and during testing using Image Pro Plus program for image analysis. The capability of ultrasonic velocity technique and image analysis for simple, and reliable nondestructive methods of characterization are presented in this investigation. It was found that both composite materials exhibit good thermal shock resistance.

Keywords: Ultrasonic velocity; Image analysis; Refractories; Cordierite/SiC composite ceramics; Thermal shock resistance; Testing

1. 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, and 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.[1,2]

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 nondestructive 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

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degradation) and behavior of the samples during thermal shock will be discussed.

2. 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 h.

Cordierite/SiC composite ceramics with weight ratio 30:70 (sample KZ 30) and 50:50 (sample KZ 50), respectively, were prepared by milling with Al_2O_3 balls in DI water in polyethylene bottle for 24 h and firing at 1160 and 1100 °C, respectively.

3. Experimental

3.1. Thermal shock

Thermal stability of the refractories was determined experimentally by water quench test (JUS.B.D8.319.). Samples were cylinders with 1 cm diameter and 1 cm high. The samples were dried at 110 °C and then transferred into an electric furnace at 950 °C and held for 15 min. The samples were then quenched into water and left for 3 min and dried before returning to the furnace at 950 °C. This procedure was repeated until failure, the number of quenches to failure was taken as a measure of a thermal shock resistance. 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).

Both materials exhibited excellent resistance to the rapid temperature changes. Samples were not exhibit total destruction during test procedure till 36 cycles.

3.2. Nondestructive measurements

3.2.1. Monitoring the damaged surface area in refractory specimen during thermal shock

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 ImagePro Plus Program, and results for material destruction, were given as function of number of quench experiments, N (Fig. 1).

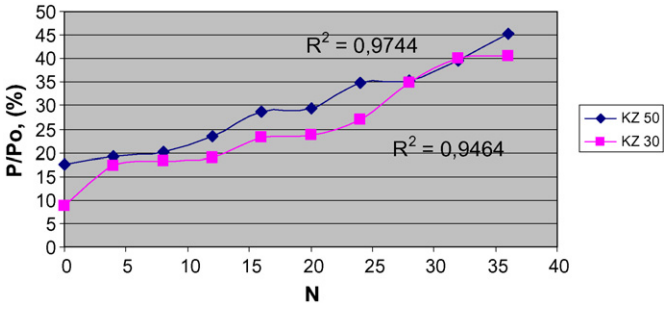

Fig. 1. Damaged surface level (P/P_0) vs. number of quench experiments (N).

3.2.2. Ultrasonic determination of dynamic Young modulus of elasticity

Ultrasonic pulse velocity testing (UPVT)[2] was first reported being used on refractory materials in the late 1950s. Various publications have dealt with the practical application of UPVT to characterize and monitor the properties of industrial refractory materials nondestructively.[3–14] The UPVT method has been considered in detail in Ref.[2] 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:

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

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[3,8–14]:

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

where V_{p} is the pulse velocity (m/s), ρ the bulk density (kg/m^3) and μ_{dyn} is 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.

First obtained results for ultrasonic velocity during testing are presented in the Fig. 2a and b for materials with different content of Mg-exchanged zeolit.

Obtained results and values of the measured ultrasonic velocity (V_{p}) but 1000 m/s indicate porosity of the sample. Results for the velocity changes in both materials suggests that materials were very stable during testing, as degradation of the velocity was not ovecome level of 20% at the end of experiment. These results indicates that number of nucleated cracks and crack prop-

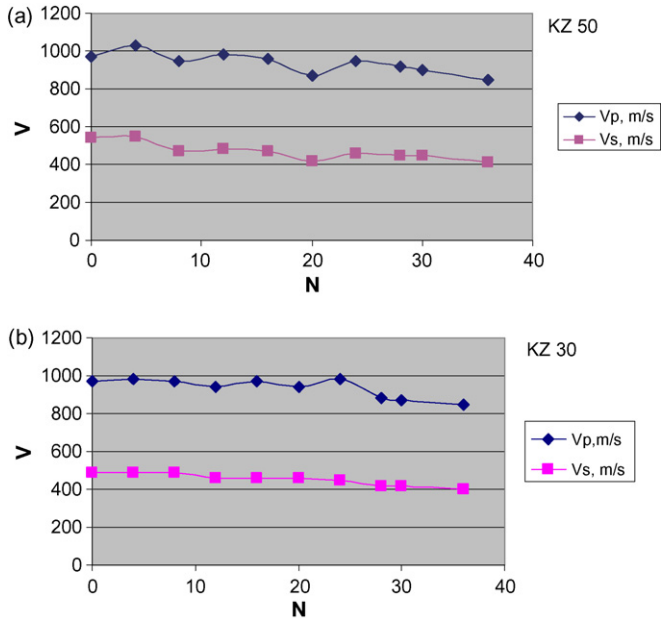

Fig. 2. (a) Values of ultrasonic velocity (V) during testing (longitudinal V_p and transversal V_s) vs. number of quench experiments of material KZ 50. (b) Values of ultrasonic velocity (V) during testing (longitudinal V_p and transversal V_s) vs. number of quench experiments of material KZ 30.

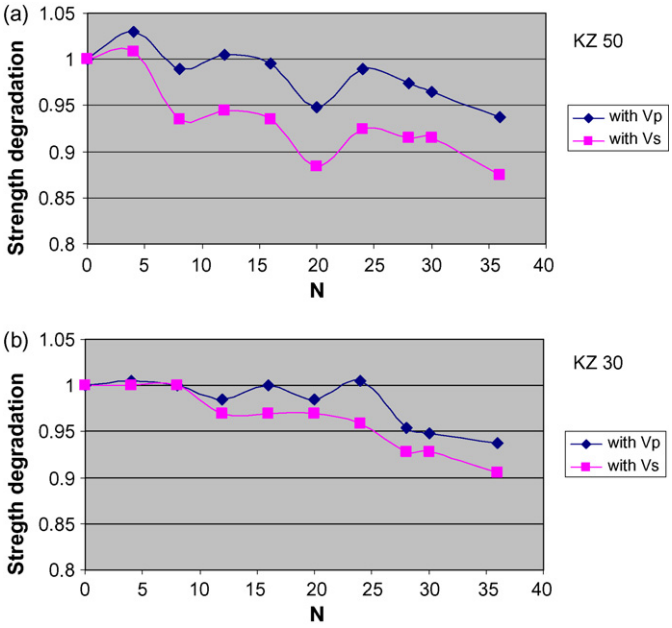

Fig. 3. (a) Strength degradation of material KZ50 vs. number of quench experiment. (b) Strength degradation of material KZ30 vs. number of quench experiment.

agation did not resolute in rapid degradation of strength and Young modulus of elasticity, and samples exhibited an excellent thermal shock behavior.

The expression for the strength degradation, based on decrease in ultrasonic velocity was used[3,6,9]:

$$\sigma = \sigma_0 \left(\frac{V_L}{V_{L_0}} \right)^n \tag{3}$$

where σ_0 is the compressive strength before exposure of the material to the thermal shock testing, V_L the longitudinal or ultrasonic velocity after testing, V_{L_0} the longitudinal or ultrasonic velocity before testing and n is the material constant ($n = 0.488$, Ref.).[3] This equation was used for calculation with longitudinal and transversal ultrasonic velocity.

Obtained results for the strength degradation base on results of ultrasonic measurments, and calculated using Eq. (3). were presented at the Fig. 3a and b.

Results for the strength degradation presented at the Fig. 3a and b showed that degradation at the end of the test was between 0.90 and 0.87% for material KZ50 and 0.90 and 0.93% for the material KZ 30. This results indicates minimal strength degradation and explains excellent results for water quench test, as result of 36 rapid temperature change.

Results for the monitoring changes of the Young modulus of elasticity during quenching are shown at the Fig. 4. Results for the dynamic Young modulus of elasticity showed that values before testing indicates that material is porous, but degradation during testing was less than 5% from level before water quench test, which explained 36 cycles of water quench test.

4. Discussion

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

- water quench test, as most usually used experimental method,
- detection of damaged surface area in refractory specimen during thermal shock,
- 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 Fig. 1 showed that during quenching damage of the original surface was not exceed 50%. Original surface showed damage about 17.6% for the KZ

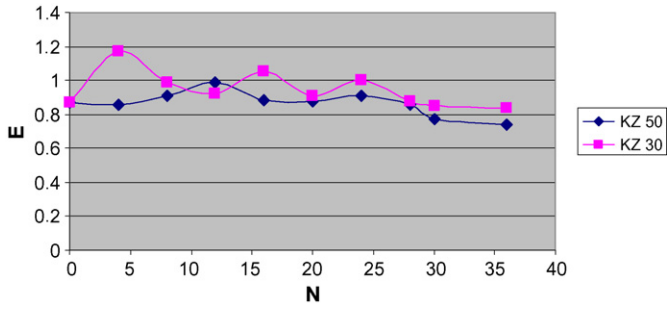

Fig. 4. Dynamic Young modulus of elasticity vs. number of quench experiments (N).

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, which 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 Fig. 2 showed very small changes and degradation of the Young modulus. These results are point out that the level of destruction in the bulk of the material and fracture nucleation and growth did not exceed level for material destruction. Results for velocity and strength degradation pointed out these conclusions.

5. Conclusion

Thermal shock behavior of cordierite/SiC composite ceramics was investigated. Ultrasonic pulse velocity testing was used to determine ultrasonic velocity, Young's modulus of elasticity in cordierite/SiC composite material. Presence of defects in samples was monitored using Image Pro Plus Program.

Results presented in this paper point out the necessity of including other test for thermal stability behavior analysis, beside water quench test, as most usually used experimental procedure. Benefits from using image analysis could be as followed:

1. It is fast, nondestructive method, so samples could be used for further tests, and financial aspect in minimizing number of samples for testing is also very important.
2. Analysis of the surface before quench test is possible, and very important information about damage of surface could be obtained.
3. Damage level during quenching could be measured. These results could be useful for prediction of sample behavior during testing.

Ultrasonic measurements could provide benefits such as:

1. It is also nondestructive method, very fast and reliable.
2. Results for degradation of parameters such are ultrasonic velocity, strength and Young's modulus could be connected with the results for number of quench experiments (N) as well with damaged surface area (P/P_0).
3. These parameters showed very strong correlation with number of quench experiments (N) as well with damaged surface area (P/P_0) and that could be used for prediction of sample behavior.

As the experimental procedure added to the water quench test for thermal stability behavior determination was described and discussed in detail, it appears that implementation of these methods and their advantages will improve materials characterization and help in preventing and improvement of material properties and synthesis conditions for achieving the best results in thermal stability resistance characteristics of material.

Acknowledgements

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