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Short Communication

The ultrasonic and image analysis method for non-destructive quantification of the thermal shock damage in refractory specimens

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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 50:50.

Samples were exposed to the water quench test from 950 °C, applying various number of thermal cycles (shocks). Level of surface deterioration before and during quenching was monitored by image analysis. Ultrasonic measurements were used as non-destructive 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 using Image Pro Plus program for image analysis. The capability of non-destructive test methods such are: ultrasonic velocity technique and image analysis for simple, and reliable non-destructive methods of characterization were presented in this paper.

1. Introduction

Furnaces require the use of high temperature ceramics as furnace liners. These ceramics liners undergo rapid temperature changes and high temperature and stress gradients that lead ultimately to the nucleation and growth of cracks by a phenomenon that is generally known as thermal shock. Subcritical damage provoke a gradual chipping of the refractory ceramic tile, and with time, a complete deterioration of the brick. Shutting down the furnace for the brick replacement results in significant economic losses due to production time loss and brick replacement costs. For example, total cost for the maintance of steel production plants is largely due to this problem.

Thermal shock resistance dictates refractory performance in many applications. In many instances, a two-fold approach, i.e. (1) material properties [1–4] and/or (2) heat transfer conditions [5–7] are used to characterize thermal shock behavior of the refractories. As an alternative, information on the thermal shock behavior of refractories can be obtained experimentally. One of tests, which is highly popular because of its simplicity, consists of quenching appropriate specimens from an oven temperature into a medium such as water, liquid metal, oil, or fused salts maintained at a lower temperature. Water quench test is usually applied for thermal stability testing. 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 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 non-destructive testing methods and their advantages for prediction of thermal shock behavior. Destruction of the samples was analyzed using Image Pro Plus Program. This is very convenient method for determining the surface damage level in sample due to the thermal shock. In this paper, relationship between the change in mechanical characteristics (strength and Young modulus of elasticity) and behavior of the samples during thermal shock will be given.

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.

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Cordierite/SiC composite ceramics with weight ratio 50:50 (samples KZ 50) were prepared by milling with Al_2O_3 balls in DI water in polyethylene bottle for 24 h and firing at 1160 °C 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. Each thermal shock cycle consisted of several consequent steps. Slow heating up by a nominal heating speed of 10 °C/min to the quench temperature set at 950 °C, holding at this temperature for 30 min to reach thermal equilibrium in whole specimen volume and finally quenching into water bath at temperature of 23 °C. Samples KZ 50 a were thermally cycled up to 36 cycles. Experimental method is similar to the procedure described in PRE Refractory Materials Recommendations 1978 (PRE/R5 Part 2).

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

3.2. Non-destructive 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 (Fig. 1). Dark areas represents damaged surface. For this investigation damage of the samples was monitored using ImagePro Plus Program, and results for damaged surface level (P/P_0), were given as function of number of quench experiments, N (Fig. 2).

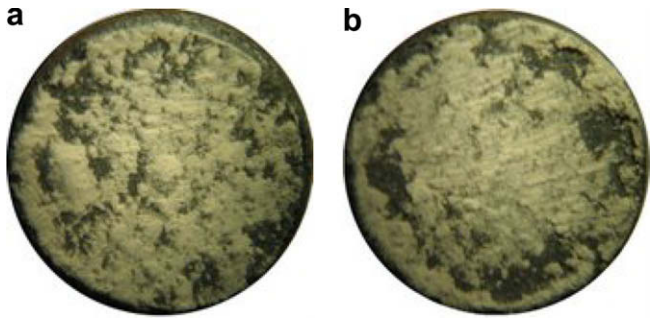

Fig. 1. Photographs of the samples (a) before water quench test ($N = 0$) and (b) after water quench test ($N = 36$).

3.2.2. Ultrasonic determination of dynamic young modulus of elasticity

Ultrasonic pulse velocity testing (UPVT) [8] was first reported being used on refractory materials in the late 1950's. Various publications have dealt with the practical application of UPVT to characterize and monitor the properties of industrial refractory materials non-destructively [8–22]. The UPVT method has been considered in detail in Ref. [8]. 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* path length (m) and T *is* 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 [8–18]:

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

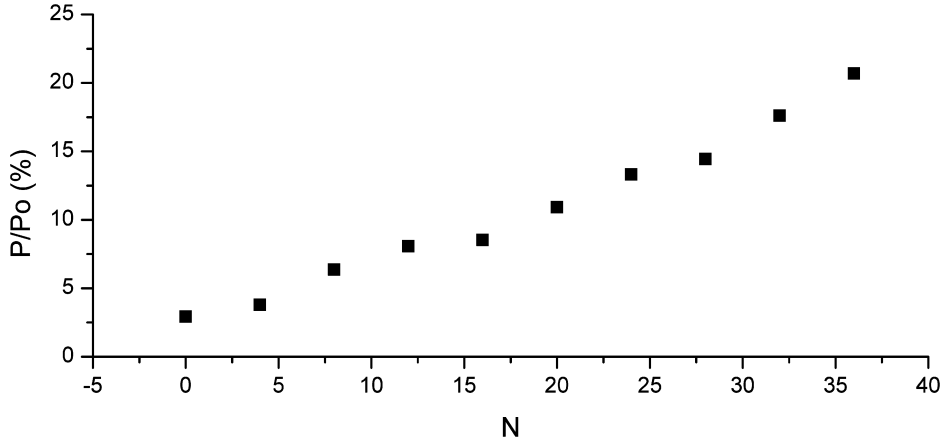

Fig. 2. Damaged surface level (P/P_0) versus number of quench experiments (N).

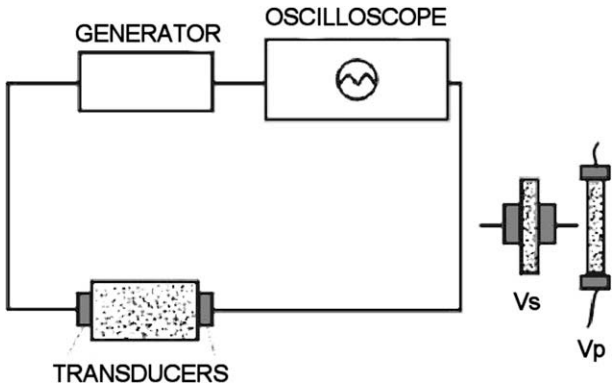

Fig. 3. Schematic drawing of the UPVT method.

where V is the pulse velocity (m/s), ρ is the bulk density (kg/m^3) and μ_{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.) (Fig. 3). 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.

4. Results

4.1. Ultrasonic velocity and strength degradation

Results for material KZ 50 will be presented versus quench experiments and level of degradation during thermal cycling. Degradation of ultrasonic velocity, strength degradation and Young modulus of elasticity changes will be given at the Figs. 4–6.

Obtained results and values of the measured ultrasonic velocity (Vp) by 1000 m/s indicate porosity of the sample. Results for the velocity changes in both materials suggests that materials were very stable during testing, i.e. there is no significant changes in velocity values for the samples before and after water quench test. These results indicates that number of nucleated cracks and crack propagation did not result 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]:

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

where σ_0 is compressive strength before exposure of the material to the thermal shock testing, V_L is longitudinal or transversal ultrasonic velocity after testing, V_{L0} is longitudinal or transversal ultrasonic velocity before testing and n-material constant ($n = 0.488$, Ref. [3]). This equation was used for calculation with longitudinal

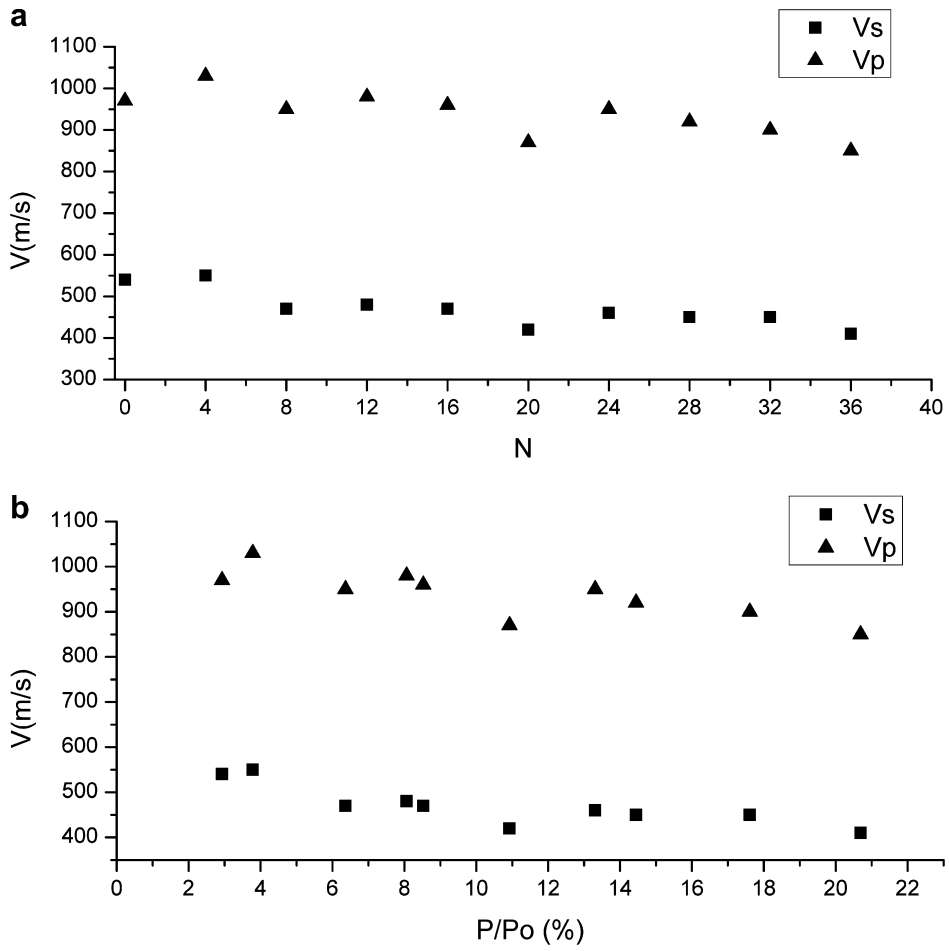

Fig. 4. (a) Values of ultrasonic velocity (V) during testing (longitudinal Vp and transversal Vs) versus number of quench experiments of material KZ 50 and (b) values of ultrasonic velocity (V) during testing (longitudinal Vp and transversal Vs) versus degradation (P/P_0) for material KZ 50.

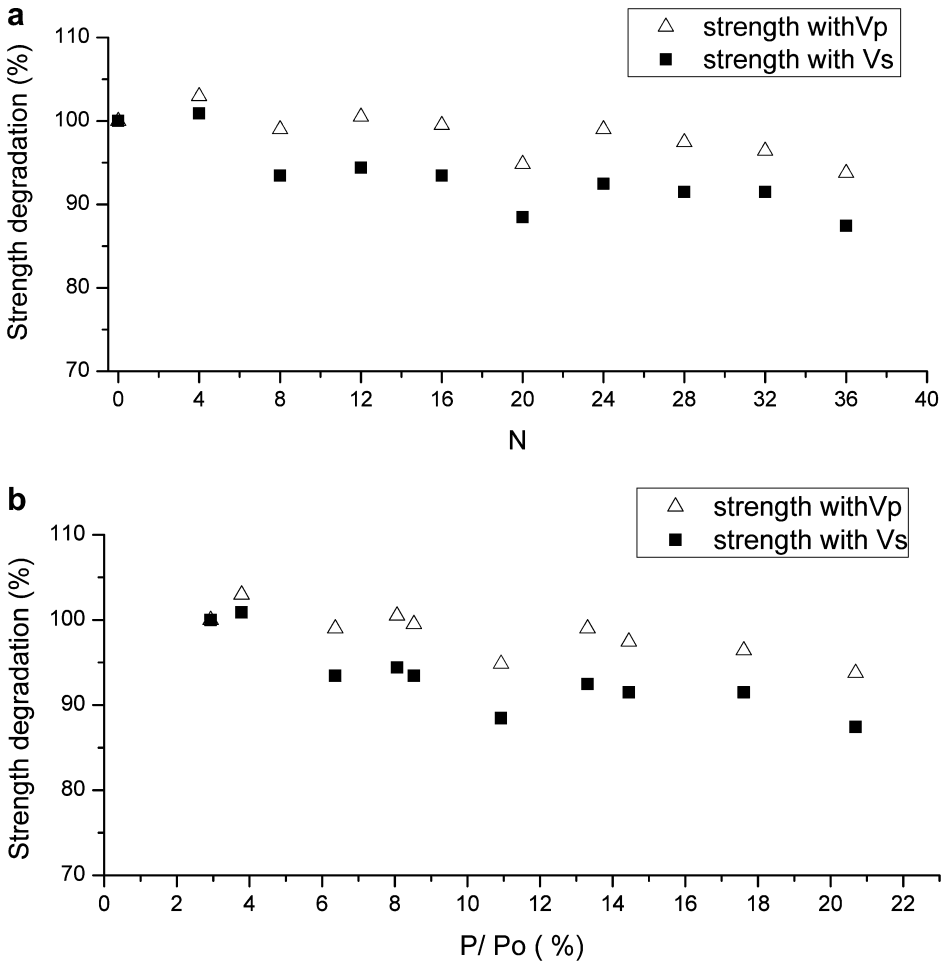

Fig. 5. (a) Strength degradation of material KZ50 versus number of quench experiment and (b) strength degradation of material KZ50 versus degradation (P/P_0).

and transversal ultrasonic velocity. Obtained results for the strength degradation based on results of ultrasonic measurements, and calculated using Eq. (3) were presented at the Fig. 5a and b.

5. Discussion

Thermal shock behavior of the cordierite/SiC composite ceramics 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
- non-destructive determination of dynamic Young modulus of elasticity.

Obtained results showed that material are excellent candidates for the applications 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. 2 showed that during quenching, damage of the surface was not exceed 21% and therefore can be considered as an excellent result.

Results for the strength degradation presented at the Fig. 5a and b showed that degradation at the end of the test was between 90% and 87%. This indicates minimal strength degradation and explains excellent results obtained for samples after 36 cycles of water quench test.

Results presented at the Fig. 6 shows very small changes (degradation) of the Young modulus during quench tests. These results pointing out that the level of destruction in the bulk of the material and fracture nucleation and growth did not exceed the threshold for material destruction. Results obtained for velocity changes and strength degradation also confirms these conclusions.

6. Conclusion

Thermal shock behavior of cordierite/SiC composite ceramics was investigated using non-destructive test methods. Ultrasonic pulse velocity testing was used to determine ultrasonic velocity and Young's modulus of elasticity in cordierite/SiC composite material. Presence of defects in samples during thermal cycling was monitored using Image Pro Plus Program.

Obtained results pointed out the necessity of including other test for thermal stability behavior analysis, beside water quench test, as most popular experimental procedure. Benefits from using image analysis are as follows:

- Fast non-destructive method, samples can be used for further tests.
- Financial aspect: minimizing the number of samples needed for testing.
- Analysis of the surface before quench test is possible; very important information about surface damage can be obtained.

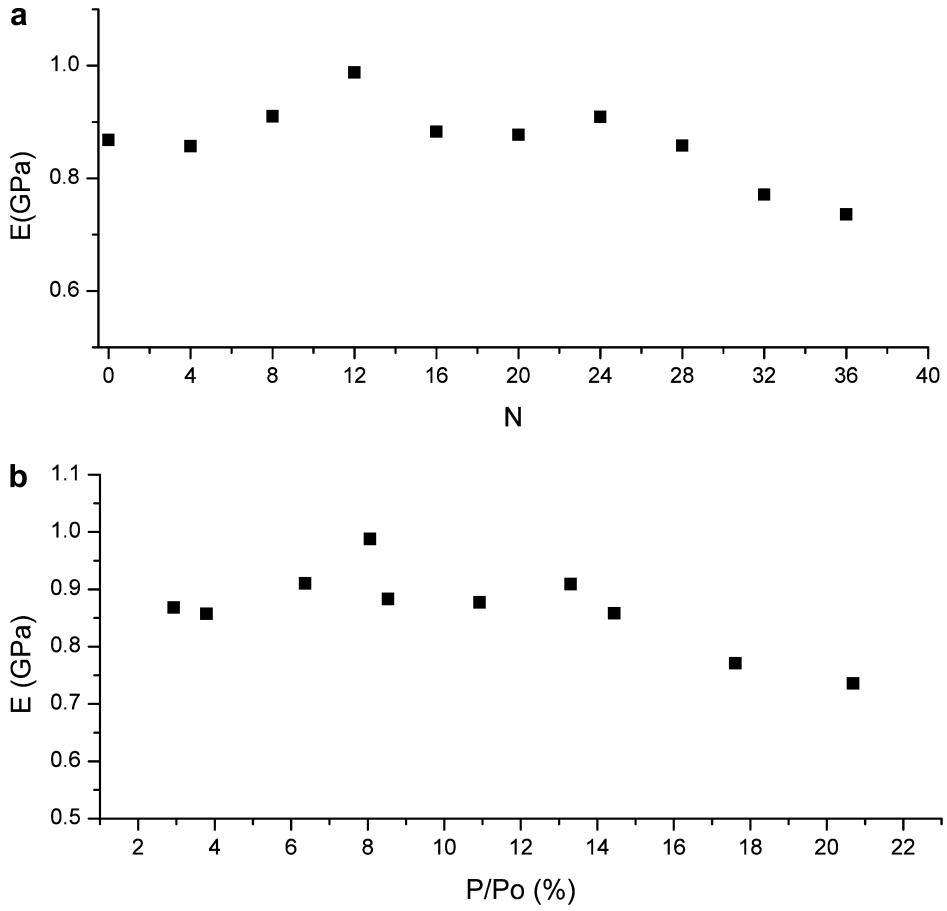

Fig. 6. (a) Dynamic Young modulus of elasticity versus number of quench experiment and (b) dynamic Young modulus of elasticity versus degradation (P/P_0).

– Damage level during quenching can be measured; these results can be used for prediction of sample behavior during testing.

Ultrasonic pulse velocity testing was employed to determine ultrasonic velocity, Young's modulus and the presence of major defects in cordierite–mullite refractory materials. The UPVT apparatus employed is well adapted to the non-destructive, in situ study of elastic changes in refractories submitted to heat treatment. 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, in order to obtain refractory materials with better thermal stability resistance.

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