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AN IMPROVED MEHOD FOR THERMAL STABILITY BEHAVIOR CHARACTERIZATION OF SILICON CARBIDE / CORDIERITE COMPOSITE MATERIAL

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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 ^{o}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 before and during testing 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 investigation.

Key words: Ultrasonic velocity; Image analysis; Refractories; Cordierite/SiC composite ceramics; Thermal shock resistance

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. In most cases the subcritical damage prosesses promote a gradual chipping of the refractory ceramic tile until the furnace have to be shut down for the replacement of brick. The result in significat economic losses in lost production time and the brick replacement costs. It also represent the most significant cost in the maintance of steel production plants.

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 behaviour of the refractories. As an alternative, information on the thermal shock behaviour of refractories can be obtained experimentally. One test for this purpose, 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 behaviour. Destruction of the samples was analyzed using Image Pro Plus Program. This is very convenient method for determining the damage surface level in sample due to the thermal shock. In this paper relationship between change in mechanical characteristics (strength and Young modulus of elasticity) and behaviour 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 hours.

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 hours and firing at 1160º C and 1100º 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 ^oC/min to the quench temperature set at 950 ^oC, holding at this temperature for 30 minutes to reach thermal equilibrium in whole specimen volume and finally quenching into water bath at temperature of 23 ^oC. Samples KZ 50 were thermally cycled up to 36 cycles. Samples 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

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 colours, 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.).

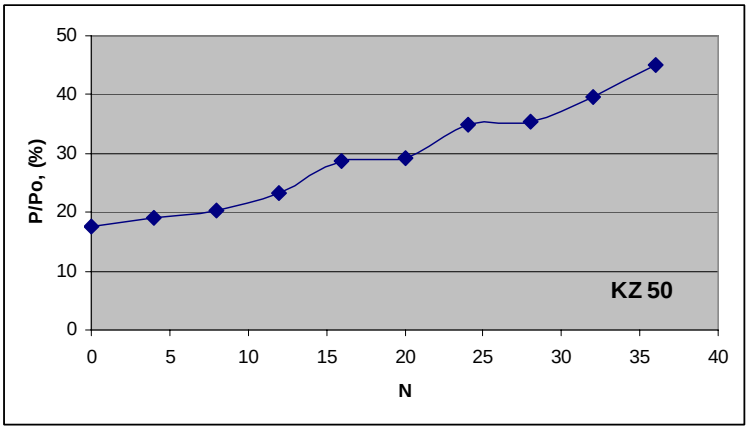

Figure 1. Damaged surface level (P/P_o) versus number of quench experiments (N)

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 [8-21]. 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 travelling 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 = path length (m) and T = 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 [8-22]:

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

Where V is the pulse velocity (m/s), ρ is the bulk density (kg/m^3) and 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.). 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 figures 2, 3 and 4.

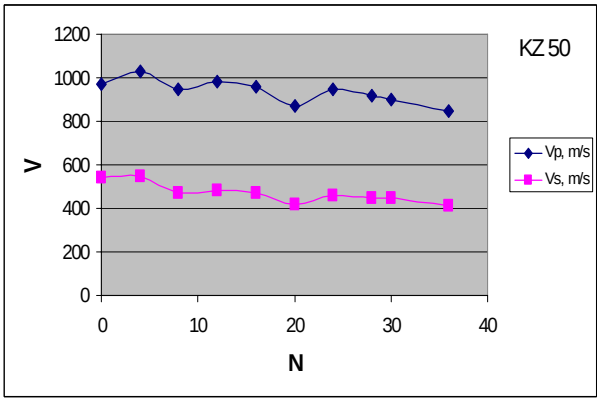

Figure 2a. Values of ultrasonic velocity (V) during testing (longitudinal Vp and transversal Vs) versus number of quench experiments of material KZ 50

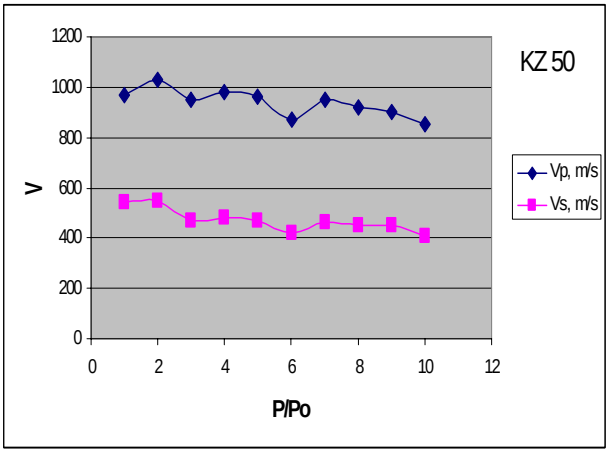

Figure 2b. Values of ultrasonic velocity (V) during testing (longitudinal Vp and transversal Vs) versus degradation (P/Po) for material KZ 50

Obtained results and values of the measured ultrasonic velocity (Vp) 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 too below from the velocity of the sample before water quench test. These results indicate that number of nucleated cracks and crack propagation did not resolute in rapid degradation of strength and Young modulus of elasticity, and samples exhibited excellent thermal shock behaviour.

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_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 - material constant (n = 0.488, ref. [3]). This equation was used for calculation with longitudinal and transversal ustrasonic velocity. Obtained results for the strength degradation base on results of ultrsonic measurments, and calculated using equation 3. were presented at the figures 3 a and 3 b.

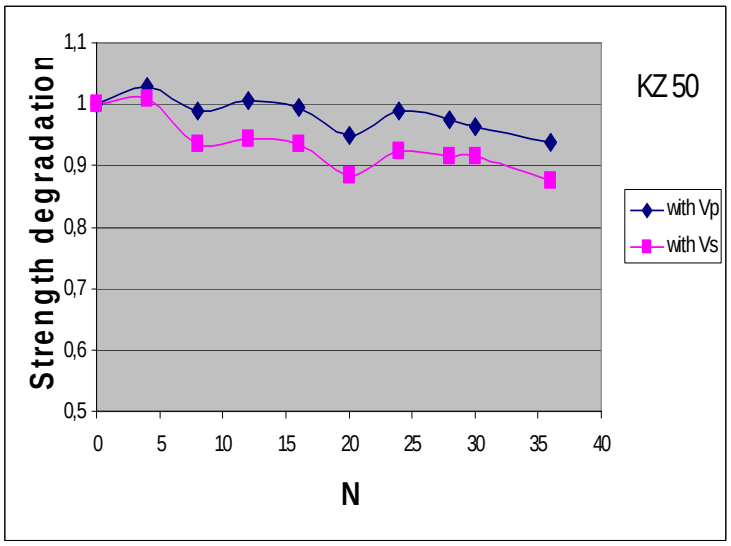

Figure 3a. Strength degradation (MPa) of material KZ50 versus number of quench experiment

Results for the strength degradation presented at the figures 3.a. and 3.b. showed that degradation at the end of the test was between 0, 90 and 0, 87 % for material KZ50.This results indicates minimal strength degradation and explains excellent results for water quench test, as result of 36 rapid temperature changes.

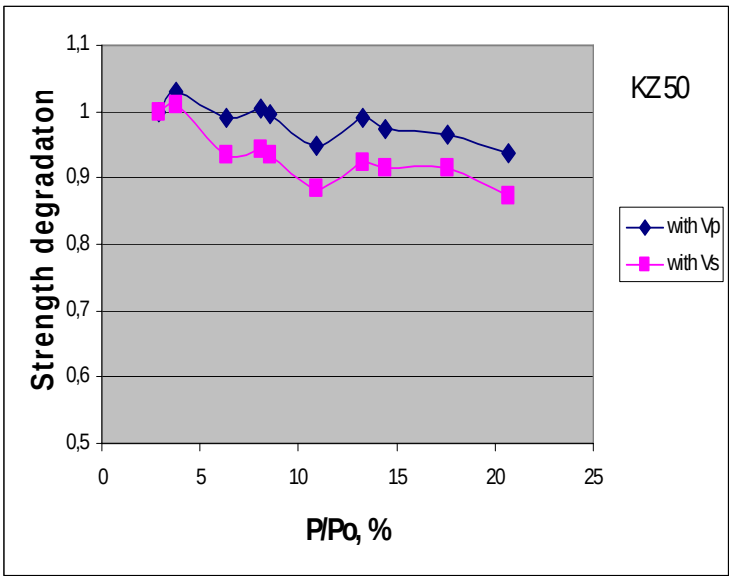

Figure 3b. Strength degradation (MPa) of material KZ50 versus degradation (P/Po)

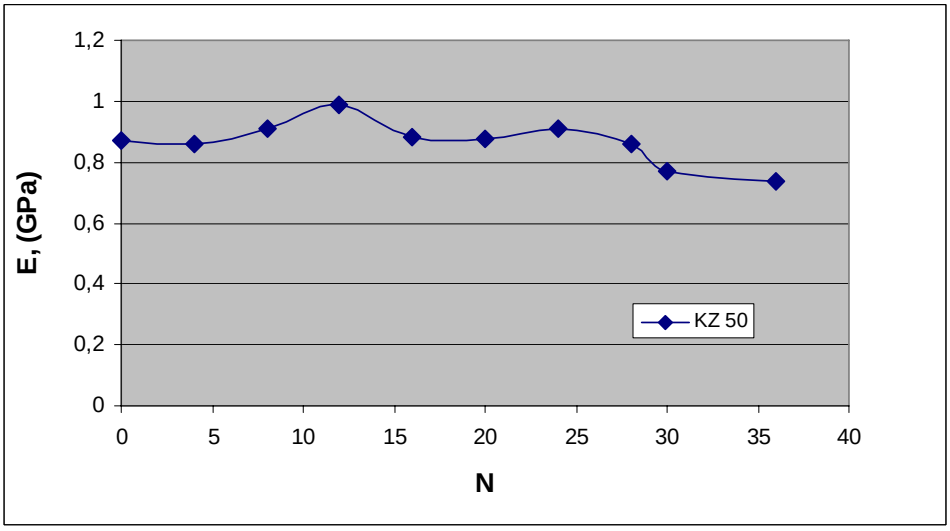

Figure 4.a. Dynamic Young modulus of elasticity versus number of quench experiments (N)

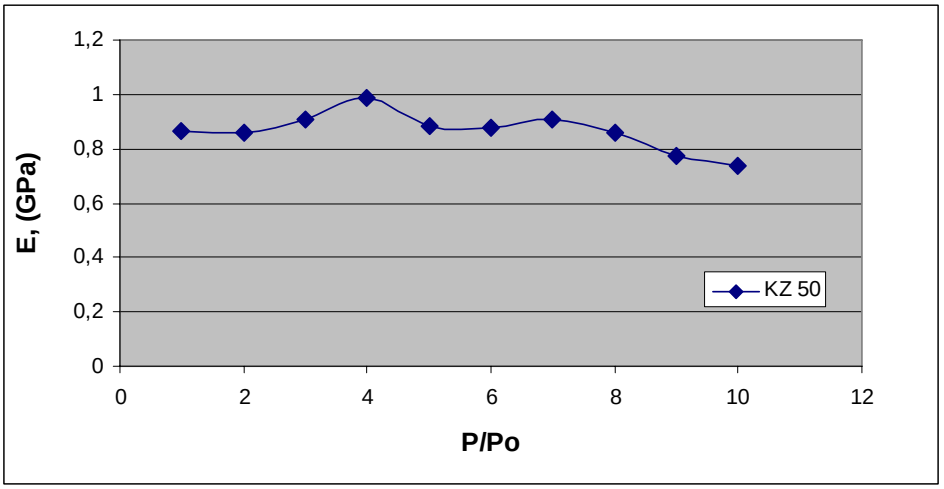

Figure 4.b. Dynamic Young modulus of elasticity versus degradation (P/Po)

5. DISCUSSION

Thermal shock behaviour of the material based on 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 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. Behaviour 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. 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.

Behaviour of the bulk of the sample was monitored using ultrasonic measurements of the Young modulus of elasticity. Results presented at the Fig.4. Showed very small changes and degradation of the Young modulus. These results are pointing 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.

CONCLUSION

Samples under investigation were exposed to certain number of cycles (from 0 to 36). Thermal shock behaviour 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 defect in samples during thermal cycling was monitored using Image Pro Plus Program.

Results presented in this paper pointed out the necessity of including other test for thermal stability behaviour analysis, beside water quench test, as most popular experimental procedure

Ultrasonic measurements could be useful for monitoring Young modulus of elasticity degradation, as well as strength degradation due to the degradation of ultrasonic velocity during testing.

As the experimental procedure added to the water quench test for thermal stability behaviour 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.

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