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ScienceDirect

Procedia Engineering 10 (2011) 2153–2157

Engineering Procedia

ICM11

Morphological analysis of surface degradation of advanced alumina based refactories subjected to thermal shock

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Abstract

Thermal stability of the alumina based samples were measured using the water immersion test. Mechanical characteristics such as strength, dynamic modulus of elasticity resulting from resonance frequency measurements were considered. Image analysis was used to measure the fiber lengths distribution, homogeneity of fiber distribution in the matrix and finally the measurement and characterization of surface degradation of specimens during thermal stability testing. The morphology of resulting surface destruction was analyzed using mathematical morphology tools.

Key words: image analysis, thermal shock, refractories, alumina fibers,

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Introduction

The mechanical properties of ceramic matrix composites (CMC) have been intensively investigated during the last twenty years. Fibre-reinforced ceramic composites comprising a matrix of sintered particulate material and fibres of sintered ceramic material distributed through the matrix are a proposing class of structural material for use in applications where high strength, high stiffness, low thermal expansion and high thermal stability are desired and where high toughness is desired and many methods have been proposed for the production of such fibre-reinforced ceramic composite.

In fibre reinforced composites, strong and stiff fibres are usually embedded into a ductile matrix with the aim enhancing mechanical properties, mainly strength, strength-to-weight ratio, etc. Under load, the matrix transmits the force to the fibres which carry the most of applied load. Fibre incorporation can have a benefit even in a brittle matrix, and then the toughness of matrix can be enhanced. The geometry and

 doi:10.1016/j.proeng.2011.04.356

arrangements of fibres are also important in controlling the mechanical properties of a fibre reinforced composite.

Alumina is the most widely used engineering ceramic material due to such favourite properties as high hardness (25 GPA or 9 on the Mohs scale), high melting point (2054 ºC), good electrical and thermal insulation.[4]

The most important properties usually determined for refractories are refractoriness, working temperature and thermal stability. Thermal shock resistance of refractory materials is one of the most important characteristics since it determines their performance in many applications. Young's modulus of representative samples was calculated using measured values of ultrasonic velocities obtained by ultrasonic pulse velocity technique.[2-12]

Thermal stability of the alumina based samples was measured using standard water quench test. Photographs of samples were taken and level of destruction was monitored using Image Pro Plus program. Image Pro Plus program was used to measure the fiber lengths distribution, homogeneity of fiber distribution in the matrix and finally the measurement during thermal stability testing.

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 Image Pro Plus program. In this paper correlation of deterioration, ultrasonic velocity and strength on thermal stability of the samples were investigated for samples having no added fibers and for samples with small amount of added fibers. The results were used for validation of the model for the prediction of thermal stability behavior of refractory samples.

Materials

Two series of samples were prepared. For raw materials: bauxite, chamotte and clay were used. Raw materials are mixed in mortar. First series consists of known composition of row materials, but in second series of samples 1 percent of alumina short fibres (it had ratio l/d ˜ 10) were added. The samples were sintered at 1200 ºC for 2h.

In this experiment Thermal Ceramics bulk fibres were used. Those fibres offer a maximum temperature range of between 1260º - 1549º C (2300º - 3000º F).

Experimental

Thermal shock

Thermal stability of the refractories was determined experimentally by water quench test (JUS.B.D8.319.). Samples were cylinders with 30.0 mm diameter and 8.0 mm high. The samples were dried at 110 ºC and then transferred into an electric furnace at 950 ºC and held for 40 min. The samples were then quenched into water and left for 3 min, dried and returned to the furnace at 950 ºC. This procedure was repeated until total destruction of sample or destruction of 50 and more percent of surface. The number of quenches to failure was taken as a measure of thermal shock resistance. Experimental method is similar to the procedure describe in PRE Refractory Materials Recommendations 978 (PRE/R5 Part2).

Image analysis

Image analysis was performed on samples as produced and after a defined number of quench experiments. The surface of the specimen was coloured with blue chalk in order to enable determination of non-damaged and damaged surfaces. The surface was measured using the options that provides program Image Pro Plus. The program gives possibility to select parts of the image that are coloured in a defined colour and this was used to separate damaged and non-damaged surface.

Ultrasonic determination of dynamic Young modulus of elasticity

Ultrasonic pulse velocity testing on refractory materials was first used in late 1950s. The ultrasonic velocity was measured with the OYO model 5210 according to the standard testing (JUS. D. B8. 121). The transducers were rigidly placed on the two parallel faces of the cylindrical sample having 30.0 mm

width and 8.0 mm using petroleum jelly as the coupling medium. The ultrasonic velocity was then calculated from the spacing of the transducers and the wave from time delay on the oscilloscope. Dynamic Young's modulus was calculated using the expression [12-15]:

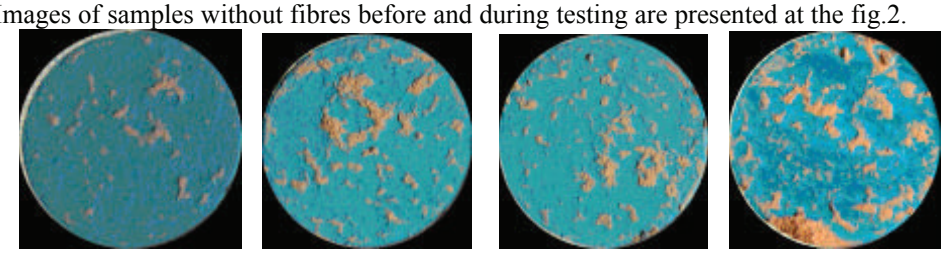
$$E_{dyn} = \rho V_P^2 \frac{(1+\mu_{dyn})(1-2\mu_{dyn})}{(1-\mu_{dyn})} \quad (1)$$

Where V_P is the velocity of longitudinal waves (m/s), μ_{dyn} the dynamic Poisson ratio and ? is density (kg/m^3).

Results and discussion

Image analysis

Simple visual inspection says that samples do not exhibit total destruction during test procedure until 40 cycles. Images of samples without fibres before and during testing are presented at the fig.2.

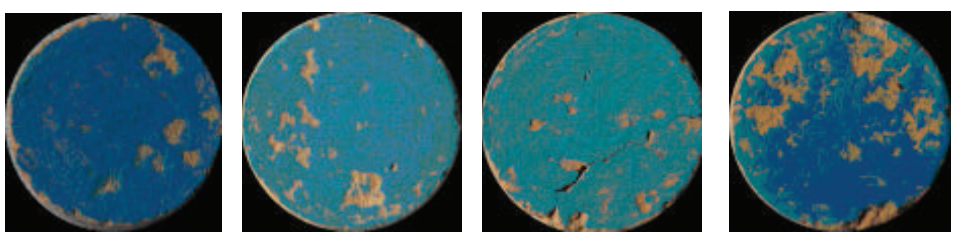


0 cycles 12 cycles 24 cycles 40 cycles

Figure 2: Samples without fibres before and during testing

Images of samples with 1 percent of short fibres before and during testing are presented in fig.3

In this study image analysis was used for the determination of surface damage level before and after a defined numbers of quenches. Sample's surfaces were marked by blue colour in order to obtain better resolution and observe difference in damaged and none-damaged surfaces of the material. Results were given as ratio P (damage surface) and P_0 (surface before quenching) as function of number of quench experiments, N (Fig. 4).



0 cycles 12 cycles 24 cycles 40 cycles

Figure 3: Samples with 1 percent of (73, 81 um mean lengths) fibres before and during testing

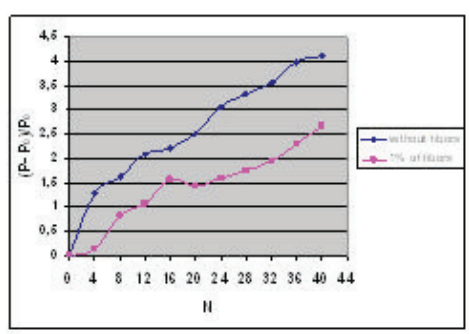
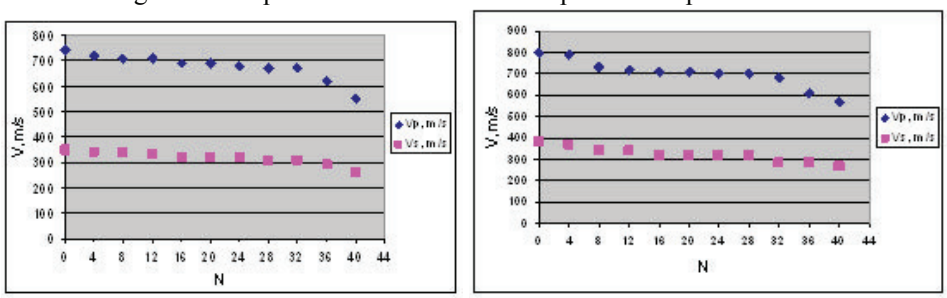


Figure. 4: Damage surface level (P) vs. number of quench experiments (N).

Ultrasonic determination of dynamic Young modulus of elasticity

First obtained results for ultrasonic velocity and dynamic Young's modulus during testing are presented in fig 5a and b and fig. 6 for samples without fibers and samples with 1 percent of fiber.



(a) (b)

Figure 5: (a) Values of ultrasonic velocity (v) during testing (longitudinal V_P and transversal V_S) vs. number of quenching experiments of samples without fibres, (b) Values of ultrasonic velocity (v) during testing (longitudinal V_P and transversal V_S) vs. number of quenching experiments of samples with 1 percent of fibres

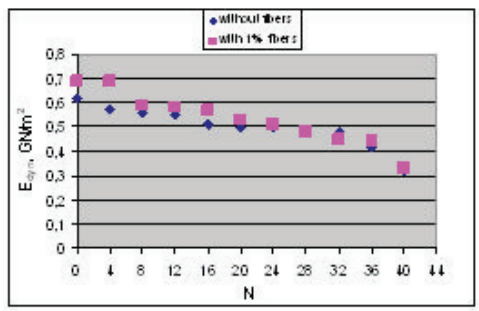


Figure 6: Values of Dynamic Young's modulus of elasticity vs. number of quenching experiments of samples without fibres and values of Dynamic Young's modulus of elasticity vs. number of quenching experiments of samples with 1 percent of fibres.

Obtained results indicate that both samples have similar values of dynamic Young's modulus. The samples with one percent of fibres have bigger Young's modulus of elasticity therefore these samples have higher strength.

The expression for the compressive strength degradation, based on decrease in ultrasonic velocity was used [12-15]:

$$\sigma = \sigma_0 \left(\frac{V_L}{V_{L0}} \right)^n \tag{2}$$

Where s_o is compressive strength before exposure of the material to the thermal shock testing, V_L the longitudinal velocity after testing, V_{L0} the longitudinal velocity before testing and n is the material constant (n = 0.488). This equation was used for calculation with longitudinal and transversal ultrasonic velocity.

Conclusion

Different tests were performed to investigate thermal stability behaviour of samples. Water immersion test was applier as the most convenient method for this purpose. Nondestructive ultrasound method for determination of dynamic Young modulus of elastitcuty was used. Surface damage was determined using image analysis.

Water quench test results showed that samples were stable till 40 cycles and these materials are excellent candidates for the applications where thermal shock resistance is required. The quality of the surface was compared to the degradation of samples using the image analysis methods. The results could be used to compare the specimens having the short ceramic fibers as reinforcement to the specimens having no reinforcement. The specimens having the reinforcement in their composition were more resistant to the thermal shock and the degradation of their mechanical properties was less present if the short fibers were in the composition.

Acknowledgements

This research has been financed by the Ministry of Science of Serbia as part of projects TR-34011 and III 45012

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