



## Thermal shock resistance of ceramic fibre composites characterized by non-destructive methods

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### Abstract

Alumina based ceramic fibres and alumina based ceramic were used to produce composite material. Behaviour of composite ceramics after thermal shock treatments was investigated. Thermal shock of the samples was evaluated using water quench test. Surface deterioration level of samples was monitored by image analysis before and after a number of quenching cycles. Ultrasonic measurements were done on samples after quench tests. Dynamic Young modulus of elasticity and strength degradation were calculated using measured values of ultrasonic velocities. Strengths deterioration was calculated using the non-destructive measurements and correlated to degradation of surface area and number of quenches. The addition of small amount of ceramic fibres improves the strengths and diminishes the loss of mechanical properties of samples during thermal shock experiments.

*Keywords:* ultrasonic velocity, image analysis, thermal shock resistance, alumina fibres, ceramic matrix composites

### I. Introduction

Ceramic materials have high thermal resistance and are very hard and resistant to compressive load. 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. Thermal shock introduces cracks into the structure and therefore there is need to improve cracking resistance of material.

In fibre reinforced composites, strong and stiff fibres are usually embedded into a ductile matrix with the aim of 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 geome-

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

Ultrasonic pulse velocity testing on refractory materials was first used in late 1950s. Young's modulus of representative samples is often calculated using measured values of ultrasonic velocities obtained by ultrasonic pulse velocity technique [1–11].

Thermal stability of the alumina based samples can be measured using standard water quench test [JUS B. D8. 319].

The goal of this work is to use non-destructive testing methods and to show their advantages for prediction of the thermal shock behavior. Destruction of samples was analyzed using Image Pro Plus program. In this paper correlation between deterioration, ultrasonic velocity and strength and the thermal stability of the samples were investigated for samples without 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.

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## II. Experimental

### Materials

In this experiment Thermal Ceramics bulk fibres were used as reinforcement. They provide good chemical stability and resistance to chemical attack. To compare with other refractory materials, bulk fibres and products made of them are light, resistant to thermal shock and they provide low thermal conductivity.

Two series of samples were prepared. Bauxite, chamotte and clay were used as raw materials and they were mixed in mortar. One series consists of raw materials without the addition of alumina fibres. Second series of samples was prepared using the addition of 1 wt.% (139.2  $\mu\text{m}$  mean lengths) of alumina fibres (it had aspect ratio  $l/d \approx 20$ ). The samples were sintered at 1200°C for 2h. Micrograph of fibres used for preparation of samples is shown in Fig. 1.

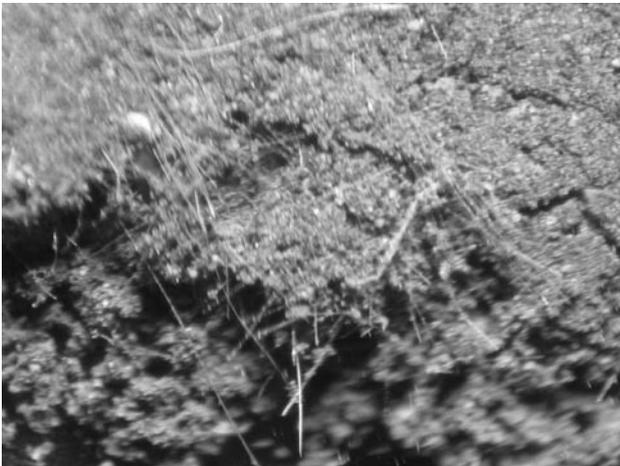


Figure 1. Image of fibres distribution in the sample after mixing of fibres and matrix materials

### 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. In experimental procedure 12 samples were used and one sample was selected randomly from the set after 4, 8, 12, 16, 20 up to 40 cycles to be examined using ultrasonic measurements and image analysis. The samples were dried at 110°C and then transferred into an electric furnace at 950°C 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°C. 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.

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.

### Image analysis

Image analysis was performed on samples after sintering and on samples exposed to 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 images were treated in the Image Pro Plus program. 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

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 diameter and 8.0 mm height 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]:

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

where  $V_L$  is the velocity of longitudinal waves (m/s),  $\mu_{dyn}$  the dynamic Poisson ratio and  $\rho$  is density ( $\text{kg/m}^3$ ).

## III. Results and Discussion

### Image analysis

Simple visual inspection shows that samples do not exhibit total destruction during test procedure until 40 cycles. Samples without fibres after 4, 8 and 12 quench cycles are presented in Fig. 2 together with the sample that was not at all exposed to quenching.

Images of samples with 1 wt. % (139.2  $\mu\text{m}$  mean lengths) of fibres ( $l/d = 20$ ) after 4, 8 and 12 quench cycles are presented in Fig. 3 together with the sample that was not at all exposed to quenching.

In this study image analysis was used for the determination of surface damage level before and after a defined numbers of quenches. Ratio of increase of surface damage to the original surface was chosen as the parameter for surface damage characterisation. The ratio changes after every quenching experiment and this is shown in Fig. 4.

### Ultrasonic determination of dynamic Young modulus of elasticity

Results for ultrasonic velocity and dynamic Young's modulus of samples are presented in Fig. 5 and Fig. 6 for samples without fibres and samples with 1 wt.% of fibres.

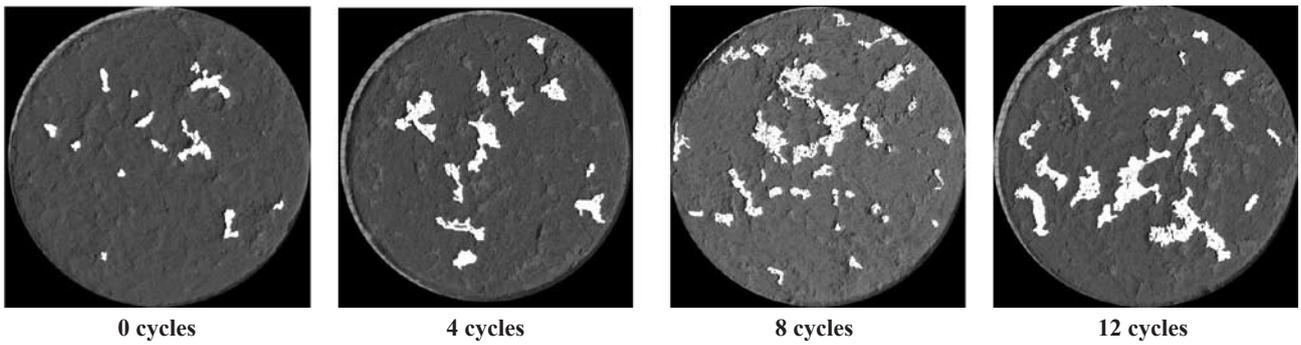


Figure 2. Samples without fibres before and during testing

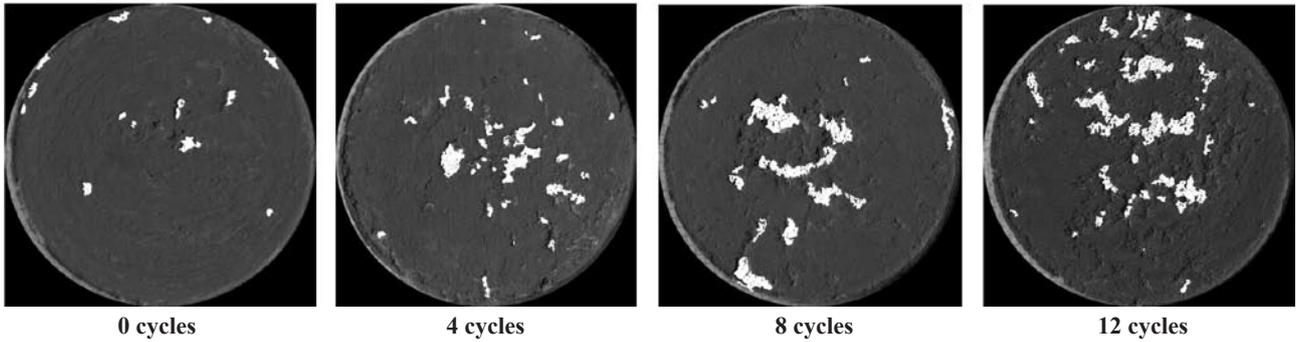


Figure 3. Samples with 1 wt.% of (139.2 μm mean lengths) fibres before and during testing

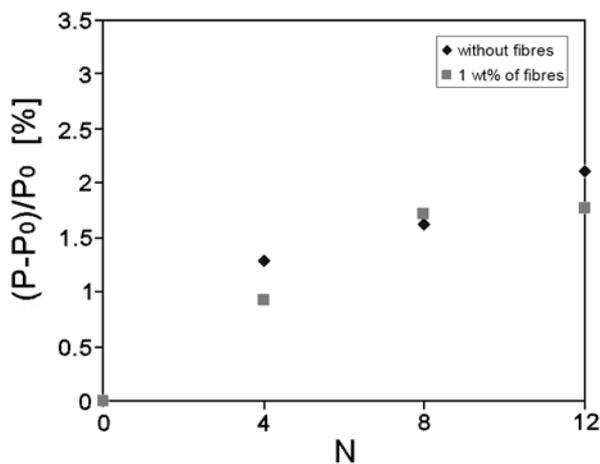
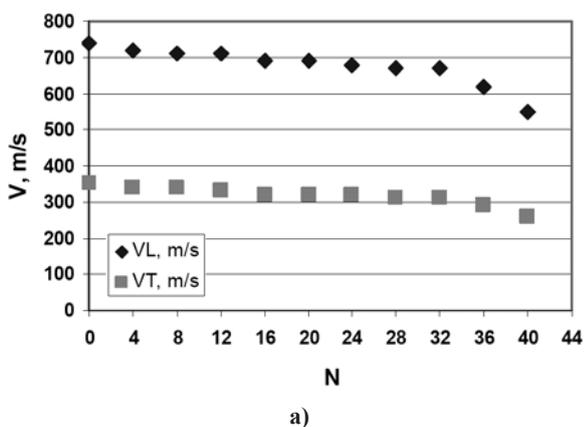
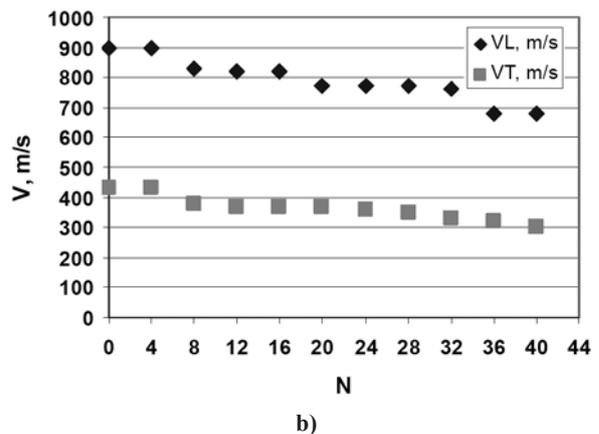


Figure 4. Damage surface level (P) vs. number of quenching experiments (N)



a)



b)

Figure 5. Values of ultrasonic velocity ( $v$ ) during testing (longitudinal  $V_L$  and transversal  $V_T$ ) vs. number of quenching experiments  $N$  of: a) samples without fibres and b) samples with 1 wt. % of fibres

The samples with 1 wt.% of fibres have higher Young's modulus of elasticity therefore these samples have higher strength.

For calculation of compressive strength degradation, equation based on decrease in ultrasonic velocity was used [12–15]:

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

where  $\sigma_0$  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 wave's velocities. Decrease in strengths is about 20 % of the original value

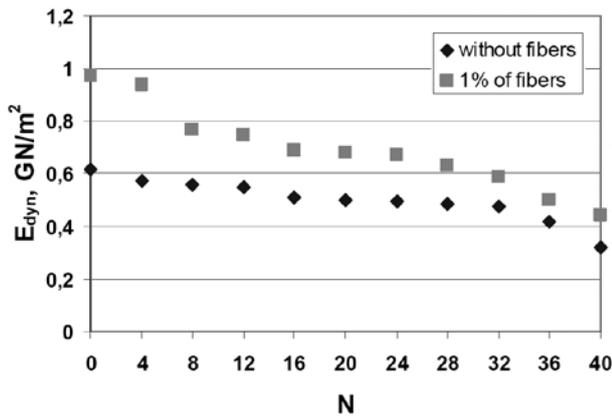


Figure 6. Values of Dynamic Young's modulus of elasticity vs. number of quenching experiments ( $N$ ) of samples without fibres and samples with 1 wt. % of fibres

for both specimens. However, the initial strengths value for specimens with 1 wt.% fibres were higher than for specimens without fibres and the samples at the end of the procedure have better characteristics than those without fibres. Results for strength of samples without fibres and samples with 1 wt.% of fibres are presented in Fig. 7.

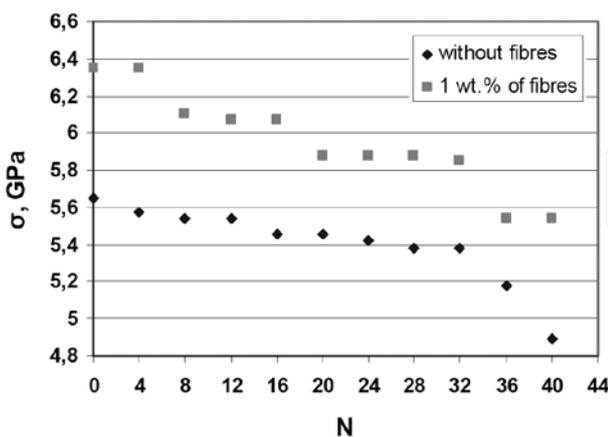


Figure 7. Values of strength vs. number of quenching experiments ( $N$ ) of samples without fibres and samples with 1 wt. % of fibres

#### IV. Conclusions

Different tests were performed to investigate thermal stability behaviour of samples. Samples were stable till 40 cycles of quench tests and these materials are very promising for the applications where thermal shock resistance is required. The goal of this paper was to determine thermal stability resistance and strength of alumina composites, as well as influence of fibres to the material behaviour. Samples with 1 wt.% of fibres have lower surface damage level and higher Young modulus of elasticity than samples without fibres.

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