

# MONITORING THE DAMAGE LEVEL DURING THERMAL STABILITY TESTING OF REFRACTORY SAMPLE USING SONIC MEASUREMENT

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## ABSTRACT

Thermal stability of the refractory material with the content of 28 %  $Al_2O_3$  was investigated. Water quench test (JUS.B.D8.319.) was applied as experimental method for thermal stability testing. Dynamic Young modulus of elasticity was calculated using measured values of longitudinal ( $V_p$ ) and transversal ( $V_s$ ) velocities. Values of dynamic Young modulus and changes of in compressive strength during testing were presented as function of the measured number of quench experiments. Changes in resistance parameters caused by thermal shock was presented, also. Analysis of the thermal shock behavior of the samples based on the obtained results was given.

**Key words:** refractories, sonic measurement, dynamic Young modulus, resistance parameters, thermal stability

## 1. INTRODUCTION

Thermal shock resistance dictates refractory performance in many applications. In many instances, a two-fold approach, i.e. 1) material properties [1-4] and 2) heat transfer conditions [5-7] is taken to characterize thermal shock behavior of the refractories. As an alternative, information on the thermal shock behavior of refractories can be obtained by experimental means. 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 [8-10], liquid metal [11], oil [12], or fused salts [13] maintained at a lower temperature.

Thermal quenching of the refractories leads to the crack nucleation and/or propagation resulting in loss of strength. Since the formation of the cracks has profound influence on the ultrasonic velocity and the Young modulus of the material, measuring either of these properties can directly monitor the development of the thermal shock damage level.

## 2. MATERIALS

The refractories used in the present study were obtained directly from commercial producer. These refractories were used in several previous studies in which a number of physical properties related to their thermal shock behavior were measured [17-25].

## 3. EXPERIMENTAL

Thermal stability of the refractories was measured by water quench test (JUS. B. D8. 319.) as experimental method. Cylindrical specimens 5 cm diameter and 5 cm high were used in experiment. The samples were dried at 110 °C and then transferred into electric furnace at 950°C, the temperature being adrieved in to 15 minutes, and held for 15 minutes. The pieces were then quenched in into water and left for 3 minutes before returning to the furnace at 950°C. This was repeated until failure. The number of quenches to failure was taken as a measure of a thermal shock resistance.

*Table1 - Thermal and Mechanical Properties*

Parameter	value
Al <sub>2</sub> O <sub>3</sub> , (%)	28
Density, ρ (g/cm <sup>3</sup> )	2.62
Thermal conductivity, k (W/mK)	1.7
Specific heat, c (kJ/kgK)	1.05
Thermal expansion coefficient, α (%)	0.7
Compressive strength, σ <sub>p</sub> (MPa)	51
Young modulus, E ( GPa)	25.5
Thermal stability (number of quench experiments, N)	6

The measurement of ultrasonic velocity was done using the equipment OYO model 5210 according to the standard testing ( JUS. D. B8. 121.). The transducers were rigidly placed on the two paralel faces of the cylindric specimen 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. Dynamic Young modulus was calculated using the expression [14, 26]:

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

$$\rho = \gamma / a \quad (2)$$

$$\alpha = Vp / Vs \quad (3)$$

$$\mu_{dyn} = (2\alpha^2 - 1) / (2\alpha^2 - 2) \quad (4)$$

where:

$V_p$  – ultrasonic velocity of longitudinal waves (m/s),  $V_s$  – ultrasonic velocity of transversal waves (m/s),  $\mu_{dyn}$  - dynamic Poisson ratio,  $\gamma$  - density ( $kN/m^3$ )

**4. RESULTS AND DISCUSSION**

The effect of the thermal shock fatigue on the Young modulus is shown on the figure 2. The good agreement between decreasing the Young modulus and number of cycles suggests that thermal shock damage could also be quantitatively evaluated nondestructively by measuring the dynamic Young modulus. Unfortunately, coefficient of correlation ( is in this case lower than it was in comparison the ultrasonic velocity with the number of cycles [24].

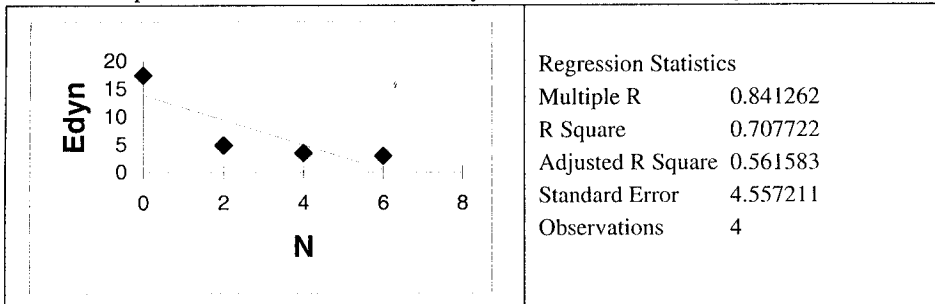


Figure 1 - Dynamic Young modulus as function of number of cycles of the quench experiment (N)

Since very good results were obtained by monitoring changes in ultrasonic velocities, these were used for the correlation of strength degradation caused by thermal shock. Expression for the strength degradation was used [14], described as:

$$\sigma = \sigma_o ( V_L/V_{Lo} )^n \tag{6}$$

where

$\sigma_o$  - is compressive strength of the material before exposure of material to the thermal shock testing, n - material constant (n = 0.488, ref. 14.)

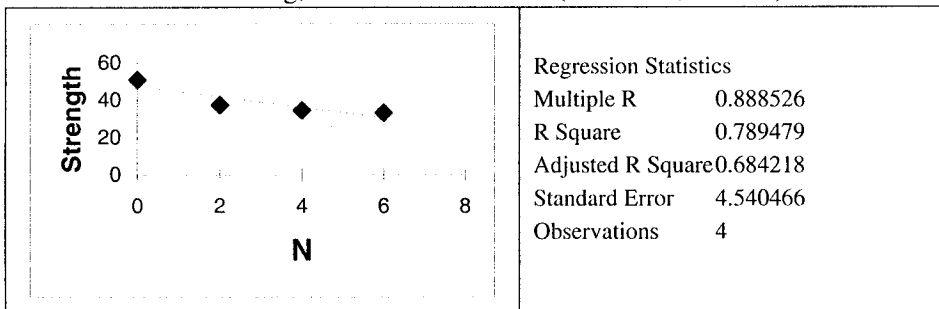


Figure 2 - Compressive strength ( $\sigma$ , MPa) degradation as function of number of cycles of the quench experiment (N)

High values of coefficient of correlation suggests that degradation in strength during thermal stability testing is in strong correlation with the water quench test experiment results.

Thermal shock stability of the refractories could be presented using resistance parameters [1-4]. Fracture and damage resistance parameters were calculated from thermal and mechanic properties listed in Table I., and usual equations are presented :

$$R = \sigma (1 - \nu) / E \alpha \quad (7)$$

$$R' = k R \quad (8)$$

The first fracture resistance parameter (R) accounts for the cases of steady temperature distribution in a specimen and/or the cases of thermal shock with an infinite heat transfer coefficient. The second fracture resistance parameter (R') is for the cases of thermal shock under the condition where the Biot number is comparatively low. Both fracture resistance parameters describe the relative resistance against the nucleation of fracture. Degradation of resistance parameters due to the degradation of strength, were also presented in Figures 3. and 4.

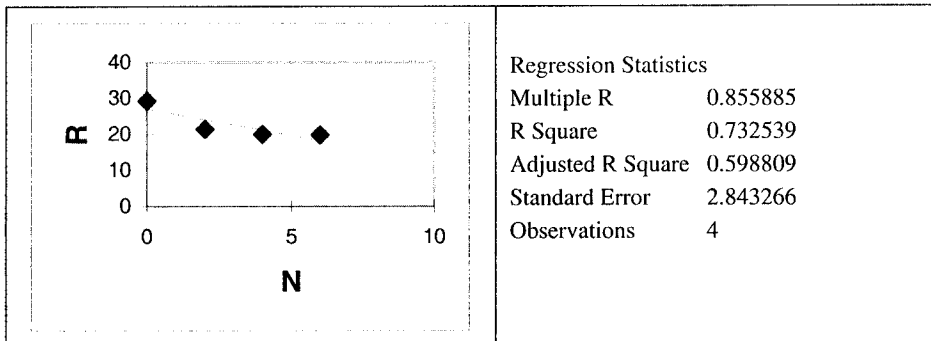


Figure 3 - First fracture resistance parameter degradation as function of number of cycles of the quench experiment (N)

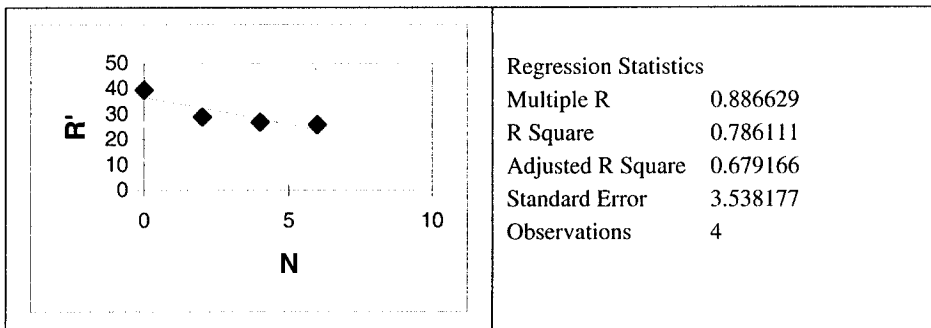


Figure 4 - Second fracture resistance parameter degradation as function of number of cycles of the quench experiment (N)

From the Figures 3. and 4. strong correlation between fracture resistance parameters degradation and the number of cycles of the quench experiment was observed.

$$R'' = E/\sigma^2(1-\nu) \tag{9}$$

$$R''' = \gamma R'' \tag{10}$$

The second damage resistance parameter  $R'''$ , describes the relative resistance against the propagation of cracks once nucleated [1-4]. This parameter is representing the resistance of the refractory to thermal shock damage by kinetic crack growth. The first damage resistance parameter  $R''$ , is a simplified formula derived from  $R'''$  by eliminating the term of fracture energy.

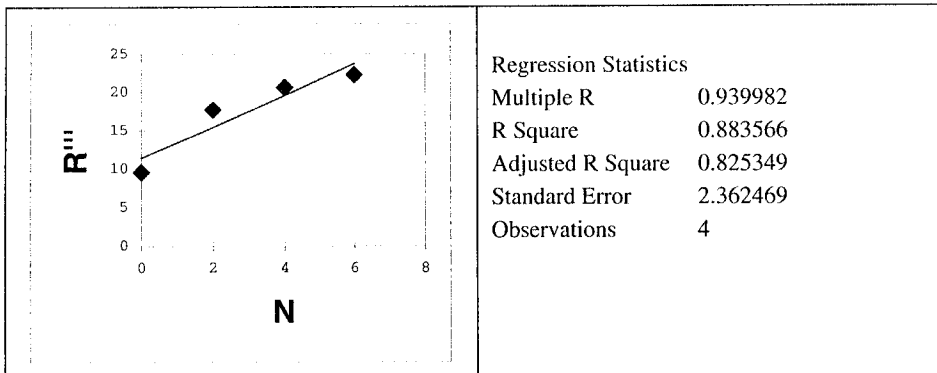


Figure 5 - First damage resistance parameter change as function of number of cycles of the quench experiment (N)

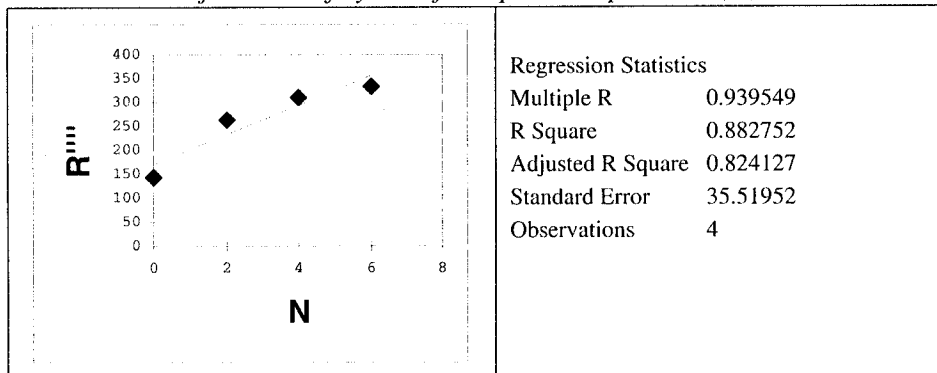


Figure 6 - Second damage resistance parameter change as function of number of cycles of the quench experiment (N)

From the Figures 5. and 6. excellent correlation between damage resistance parameters degradation and the number of cycles of the quench experiment was observed. In case of monitoring damage resistance parameters changes due to the thermal shock, higher values of coefficient of correlation were observed, comparing to the fracture resistance parameters.

## 5. CONCLUSION

Monitoring of thermal shock damage development in refractories by nondestructive method is of high industrial significance in relation to productivity as well as safety. Sonic measurement is very useful tool to solve this problem since the advantages are:

- instrument is small in size, portable, and very easy to use on site, if necessary,
- results of the method could be obtained very fast, and as they are related to damage, the damage level in material could be predicted with very good accuracy.

Results presented in this study suggest that of the sonic measurements could be used for prediction of damage level in specimen due to the thermal shock. Monitoring the damage level could be obtained using the decreasing of the ultrasonic velocity. Also, as the results in the Chapter 4. suggest the decreasing of the Young modulus could be also used for the same purpose. The lower values of the coefficient of correlation with the water quench results in that case suggests more accurate analysis, possibly combined with other correlation to get more accurate prediction. Strength degradation in material due to thermal hocking could be also evaluated, with very strong correlation with the number of cycles of the quench experiment (N). Prediction of strength degradation could be a very useful parameter for life time prediction of material.

Results presented in this paper showed that higher values of fracture resistance parameters are expected for the sample before thermal shocking. After shocking both fracture resistance parameters are decreasing, with strong correlation with water quench test.

For the damage resistance parameters higher values were observed after thermal cycling. Calculated values of both damage resistance parameters for the sample were lower before than after quenching. Better results for the correlation with the results of quench experiment were observed for the damage resistance parameters.

### *Acknowledgements*

This work has been supported by the Ministry of Science, Technology and Development of Serbia.

### LITERATURE

- [1] D. P. H. Hasselman, "Unified Theory of Thermal Shock Fracture Initiation and Crack Propagation in Brittle Ceramics ", *J. Am. Ceram. Soc.* , Vol. 52 , No.1, 600 -604 (1969)
- [2] D. P. H. Hasselman, "Elastic Energy at Fracture and Surface Energy as Design Criteria for Thermal Shock", *J. Am. Ceram. Soc.* Vol 46, No 11, 535-540 (1963)

- [3] J. Nakayama *J. Am. Ceram. Soc.*, "Direct Measurements of Fracture Energies of Brittle Heterogeneous Materials", *J. Am. Ceram. Soc.*, Vol 48. No 11., 583 - 587 ( 1965)
- [4] J. Nakayama, M. Ishizuka, "Experimental Evidence for Thermal Shock Damage Resistance", *Amer. Cer.Soc.Bull.*, Vol. 45 , No.7 666-669 (1965)
- [5] W. D. Kingery, "Factors Affecting Thermal Stress Resistance of Ceramic Materials", *J. Am. Ceram. Soc.*, Vol 38, No 1, (1955) 3-15
- [6] S. S. Manson, R. W. Smith, "Theory of Thermal Shock Resistance of Brittle Materials Based on Weibull's Statistical Theory of Strength", *J. Am. Ceram. Soc.*, Vol 38, No 1, (1955) 18-27
- [7] H. Hencke, J. R. Thomas, JR. and D. P. H. Hasselman, "Role of the Material Properties in the Thermal-Stress Fracture of Brittle Ceramics Subjected to the Conductive Heat Transfer", *J. Am. Ceram. Soc.* Vol 67, No 6, (1984) 393-398
- [8] R. W. Davidge, G. Tappin, "Thermal Shock and Fracture in Ceramics" , *Trans. Br. Ceram. Soc.* Vol 66, No 8, (1967) 405-22
- [9] D.P.H.Hasselman, "Strength Behavior of Polycrystalline Alumina to Thermal Shock", *J. Am. Ceram. Soc.* Vol 53, No 9, (1970) 490-95
- [10] P. F. Becher. D. Lewis III, K. R. Carman, A. C. Gonzales, "Thermal Shock Resistance of Ceramics : Size and Geometry Effects in Quench Test", *Ceram. Bull.* Vol 59, No. 5, (1980)
- [11] M.Oguma, C.J.Fairbanks, D.P.H.Hasselman, "Thermal Stress Fracture of Brittle Ceramics by Conductive Heat Transfer in a Liquid Metal Quenching Medium", *J.Am.Ceram.Soc.* Vol 69, No 4, C-87-C88 (1986)
- [12] J. P. Singh, Y. Tree, D. P. H.Hasselman, "Effect of Bath and Specimen Temperature on the Thermal Stress Resistance of Brittle Ceramics Subjected to the Thermal Quenching", *J. Mater. Sci.* Vol 16, (1981) 2109-18
- [13] W. B. Crandal, J. Ging, "Thermal Shock Analysis of Spherical Shapes" *J. Am. Ceram. Soc.* Vol 38, No 1, (1955) 44-54
- [14] S. K. Nyiogi, A. C. Das, "Prediction of the Thermal Shock Behaviour of Castable Refractories by Sonic Measurements" *Interceram*, Vol 43, No 6, (1994), 453-457
- [15] J. H. Ainworth and R. H. Herron, "Thermal Shock Damage Resistance of Refractories", *Ceramic Bulletin*, Vol.53, No.7, (1974) 533-538
- [16] J. P. Singh, J. R. Thomas, D. P. Hasselman, *J. Am. Ceram. Soc.* Vol. 63, No. 3-4, (1980) 140-144
- [17] T.Volkov-Husović, Ph.D. Thesis, Belgrade, 1999.

- [18] T.Volkov-Husović, R.Jančić, M.Cvetković, D.Mitraković, Z.Popović, "Thermal Shock Behavior of Alumina Based Refractories : Fracture Resistance Parameters and Water Quench Test", *Materials Letters*, 38 (1999) 372-378
- [19] T.D.Volkov-Husović, Z.V.Popović, "Resistance parameters and water quench test as criteria of thermal shock behaviour of alumina reafactories", *Material Science and Technology*, Vol 15, No 10 (1999) 1216-1219
- [20] T.Volkov-Husovic, R.M.Jancic, "Thermal Shock Behavior of Alumina Based Refractories: Comparison between Damage Resistance Parameters and Water Quench test", *Industrial Ceramics*, Vol 20, No 3, (2000) s 94-97
- [21] T.D.Volkov-Husovic, R.Jancic, "Influence of natural convection on thermal stability of refractory specimen", *Journal of Metallurgy*, No 1, Vol 7, (2001) 59-67 (in Serbian)
- [22] T. D. Volkov-Husović, R. M. Jančić, V. Radojević. Z. Popović, "Prediction of the thermal shock behavior of alumina based refractories: temperature difference, damage resistance parameters and water quench test" *EUROMAT 2001, Rimini, Italy, 10-14 June 2001, Abstracts and Papers CD* p 150
- [23] T.D.Volkov-Husović, R.M.Jančić, "Thermal Shock Behavior of Alumina Based Refractories", *Industrial Ceramics*, Vol. 21, No 1. (2002) 159-164
- [24] D. Prtenjak, M. Matijašević, M. Cvetković, J. Majstorović, T. Volkov-Husovic, "Odredjivanje dinamičkog Jungovog modula elastičnosti tokom ispitivanja termostabilnosti vatrostalnog materijala", "Investigation of the dynamic Young modulus during thermal stability testing of the refractory material", (in Serbian), *Journal of Metallurgy*, to be published