EVALUATION OF CERAMIC FRACTURE CAUSED BY THERMAL SHOCK

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SYNOPSIS

This work presents the results of determining the resistance of ceramics based on silicon nitride, scandium oxide, yttrium oxide, etc. subjected to thermal shock loads at which the specific features of fracture of the specimens being studied have been investigated following the nondestructive testing techniques. The dependence of the results obtained with those techniques on the specific features of mechanical behaviour inherent to ceramics has been demonstrated. The pros and cons of techniques of acoustic emission, of measuring the velocity of ultrasonic wave propagation, of determining a change in the spectrums of the ultrasonic vibration pulses, etc. employed for evaluating thermal fracture of ceramic specimens have been anlyzed.

KEYWORDS

Thermal shock resistance; fracture; crack; specimen; strength; ultimate strength; brittleness measure; critical temperature difference; acoustic emission; ultrasound velocity.

INTRODUCTION

The ability of ceramics to resist a fracturing effect of thermal shock loads when using it as a structural material in high-temperature machinery is the main factor determining its practical application. However, in the preset operating conditions the ceramics often shows insufficient thermal shock resistance and, consequently, happens to be inoperative. Therefore, investigations of strength of these materials under unsteady temperature conditions and studies of the specific features of their fracture in such conditions draw unremitting attention of researchers (Kingery, 1955; Gogotsi G.A. et al., 1969). These works have become particularly urgent after successful attempts to use ceramics for manufacturing nozzle assemblies, blades, gas turbine disks, diesel engine pistons and other similar products, thus showing this type of material to be pro-

mising for engineering. At the same time, the use of ceramics for such demanding applications calls for more detailed and thorough study of its thermal shock resistance taking into account the specific features of its actual mechanical behaviour instead of idealizing it as it often happens in the case of an elastic solid body model (Gogotsi G.A., 1973). The most important problem in defining the thermal shock resistance, particularly, of structurally-inhomogeneous ceramics is to determine the moment of thermal crack initiation in ceramics (Gogotsi G.A., 1967), i.e. to register the fact of its fracture during tests.

SPECIMENS AND THERMAL SHOCK RESISTANCE EVALUATION TECHNIQUE

Determination of the degree of fracture of specimens for the evaluation of their thermal shock resistance has been carried out using ceramic materials with different mechanical behaviour (Table 1): "brittle", elastically-deformed until fracture, the brittleness measure[1] χ (Gogotsi G.A., 1973) of which equals 1, and "relatively brittle" inelastically-deformed materials the brittleness measure of which is less than I. Most specimens made from those materials were of 6 x 6 x 50mm size (except corundum refractory specimens of 8 x 8 x 110 mm size).

TABLE 1. Characteristics of materials

Material	Density, g/cm³	Brittleness measure	Ultrasonic waves velocity, m/s	Coefficient of linear thermal expansion, °C⁻¹ x10⁻⁶ (20 ÷900°C)
Scandium oxide	3.79	1	7905	8.4
Aluminium nitride	3.15	1	8300	5.5
Reaction-sintered silicon nitride with addition of 40% silicon carbide bide, NKKKM-80	2.58	1	8370	3.6
Reaction-sintered silicon nitride with addition of 40% silicon carbide, NKKKM-81	2.49	1	8690	3.5
Alumoboronitride	1.99	0.58	4385	-
Yttrium oxide with addition of 15% yttrium aluminate	4.82	0.64	4963	8.0
Corundum refractory	3.46	0.27	6783	8.2
Cordierite K-2	2.04	0.56	5411	3.3

[1] Brittleness measure χ is the characteristics of mechanical behaviour equal to the ratio of the specific elastic energy accumulated in the material by the moment of fracture to all specific energy spent for its deformation by this very moment.

Thermal shock resistance has been determined following the most widely acknowledged technique (Hasselman, 1969) which rests with evaluation of the residual bending strength of the specimens tested under thermal shock loads of a given intensity (for example, quenching in water after heating in radiation electric furnace, as in our experiments). In such tests a degree of fracture (damage) of a material has been evaluated according to a change in its residual strength. As a result of the experiments the thermal shock damage resistance diagrams (TSDR-diagrams) have been plotted which show the dependence of the residual strength of the thermal shock damaged specimens on the intensity of the thermal shock loads due to the temperature difference between the furnace and the quenching bath. Such diagrams plotted for brittle and relatively brittle materials differ considerably (Gogotsi G.A., 1978). The TSDR-diagrams for brittle materials featured temperature difference ΔT_c which corresponds to a sharp reduction in the residual strength of material caused, probably, by formation of cracks of critical or close to critical size in the course of quenching.

The TSDR-diagrams for the relatively brittle materials feature a gradual reduction in the residual strength resulting from the growth, as the thermal stresses increase, of the material structure defects (microcracks) which make the basis for the development of macrocracks. It is necessary to mention that the issue of a possibility of subcritical growth of defects under thermal shock loads was investigated by Hasselman (1969). Figure I demonstrates the TSDR-diagrams obtained for materials with different values of the brittleness measure. The Figure suggests that the lesser the brittleness measure of a material, the lesser is the probability of critical-length crack formation in the specimen under thermal shock test and the lesser is its specific reduction in strength (divided by the value of temperature difference). Thus, the value of the brittleness measure makes it possible to determine, although in the first-order approximation, the pattern of the material fracture (the shape of the TSDR-diagram).

It should be noted that in pure bending (four-point bending) of brittle materials specimens subjected to preliminary thermal shock loadings with the intensity corresponding to the descending portion of the TSDR-diagram (higher than T) nonlinear stress-strain relationships were observed. In other words, mechanical behaviour of the thermal shock damaged specimens of the brittle materials may not differ from that of the relatively brittle ones. Similar pattern was observed when using other brittle materials, specifically, scandium oxide and yttrium oxide (Gogotsi G.A. et al., 1977; Gogotsi G.A. et al., 1978). Here, it should be stressed that such specific features of deformation of the thermal shock damaged specimens were particularly evident when a crack pattern was detected on their surface.

ULTRASONIC MEASUREMENTS OF THERMAL SHOCK DAMAGED SPECIMENS

The above-described technique of thermal shock resistance evaluation based on the analysis of the TSDR-diagrams is most

universally applied and easy-to-realize. However, to obtain more detailed information on the character of thermal shock damage in the material and to improve the reliability of experimental data such tests are often accompanied by measurements employing nondestructive testing techniques. The use of these techniques is of special interest when it is necessary to evaluate a degree of fracture of specimens in the process of their thermal shock cyclic tests, i.e. without mechanical fracture following the thermal shock loading. It is well known that the most widespread application for this purpose was gained by the ultrasonic testing based on measuring the time required for the ultrasonic pulses to pass through the specimen material by which the values of the ultrasonic wave velocity and the dynamic moduli of elasticity are calculated. Our experiments have demonstrated that such measurements are effective only when the whole structure of the material is damaged, which is usually inherent to the relatively brittle ceramics. As an example Fig. 3,a shows the TSDR-diagram for cordierite and the corresponding values of its dynamic modulus of elasticity calculated from the results of measuring the ultrasonic wave propagation time carried out at the frequency of 150 kHz.

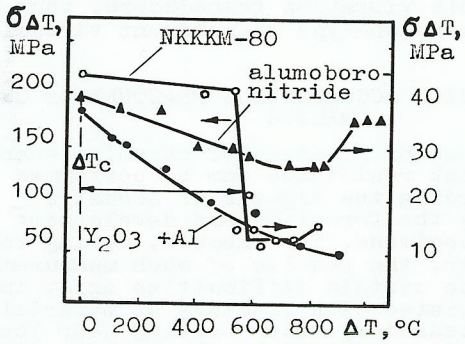

Fig. 1 TSDR - diagram (quenching of specimens in water bath 25°C).

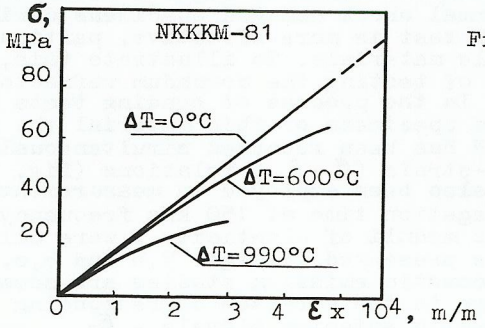

Fig. 2 Strain diagrams of reaction-sintered silicon nitride specimens; T is the temperature difference at which specimens have been thermal shock damaged.

At the same time, this testing technique employed for the specimens made from the materials with brittleness measure $\chi = 1$ happens, as a rule, to be ineffective (Gogotsi G.A., 1980). This can be explained by the fact that the longitudinal ultrasonic waves which were used in the measurements are rather insensitive to defects on the specimen surface so long as they are limited in number, insignificant in size and do not penetrate deep enough into the specimen material. Figure 3,b shows that a noticeable change in the dynamic modulus of elasticity for the scandium oxide specimens is observed only at the temperature differences exceeding by far their critical values. At such temperature differences a fragmentary fracture of the specimen material is observed along with a considerable depth of the cracks (Gogotsi G.A. et al., 1980). In the process of testing other brittle materials based on aluminium nitride even at the temperature differences exceeding by far the critical values only a limited number of small-size cracks has developed on the specimen surface (Gogotsi G.A., 1980) which actually failed to be registered in the above-described ultrasonic testing (Fig. 3,c).

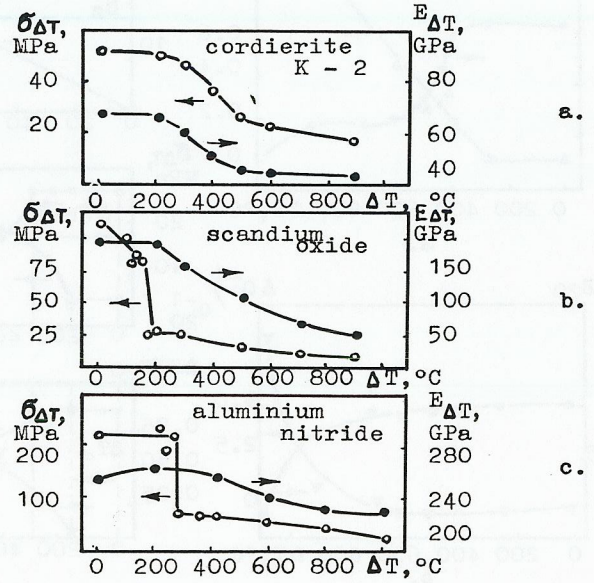

Fig. 3 TSDR - diagrams and dependences of values of dynamic modulus of elasticity on the intensity of specimen thermal shock loading.

Sometimes, in assessing the thermal shock resistance of ceramics a technique is used based on measuring the change in the specific damping capacity Q^{-1} of the specimens resulting from the damage of their structure in the process of thermal shock loading. This technique yielded fairly satisfactory results in evaluating the thermal shock resistance of silicon carbide (Coppola et al., 1973). But in a number of cases, particularly, when monitoring the early stages of fracture of thermally stressed brittle materials, this technique proved to be inoperative (Fig. 4,b). This stems from the fact that formation in the specimens of a limited number of small cracks has no significant effect on their specific damping capacity Q. Here, it is necessary to emphasize that the use of this technique for the relatively brittle materials is unsuitable, since they have, as a rule, a low value of Q^{-1} and therefore it is extremely difficult to determine the width of resonance curve for them. To compare the above-described data the results of

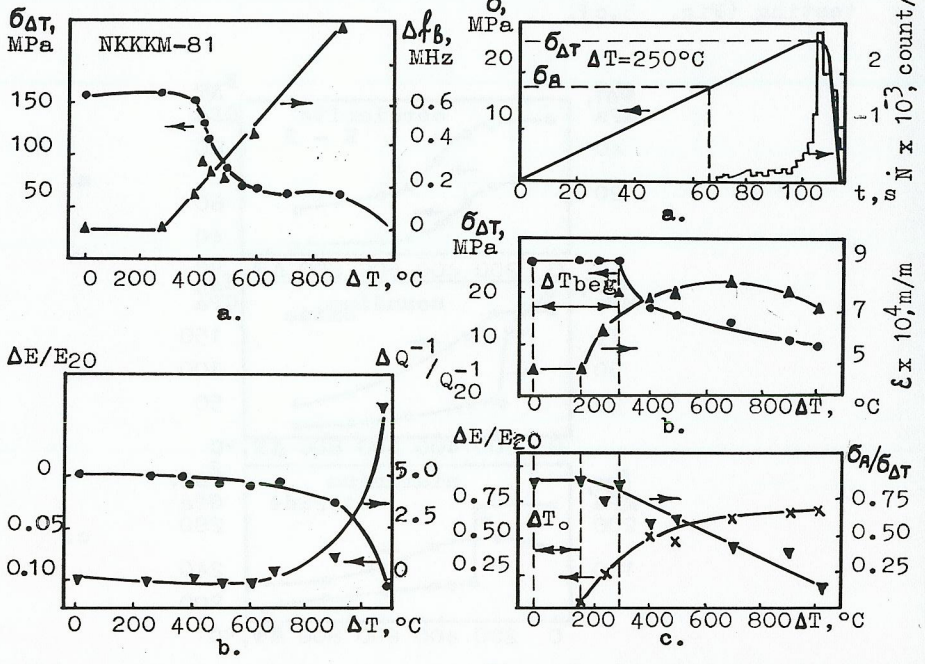

Fig. 4 Change in the reaction-sintered silicon nitride characteristics depending on the intensity of thermal shock loading.

Fig. 5 Results of testing of corundum refractory.

the assessments based on measuring the velocities of ultrasonic wave (Figure 4,b) also presents the normalized (divided by the value obtained using the specimens not tested) change in the modulus of elasticity of the material.

A positive result in evaluating the degree of fracture of the thermal shock loaded specimens has been obtained when employing a technique which rests with recording the change in parameters of spectrums of the ultrasonic pulses passed through the specimens before and after their thermal shock damage, at an immersion input and output of the vibrations. This technique has proved to be viable because the defects in the specimens tested, their number and size considerably change the spectrum of the ultrasonic pulses passed through them. Figure 4,a presents the results of using this technique in testing the thermal shock damage resistance of NKKM-80 specimens. Here, a shift in boundary frequency f_b of the spectrum of the first half-period of the ultrasonic vibrations passed through the specimen depending on the thermal shock load intensity is shown together with the TSDR-diagram. It is necessary to stress that the use of this technique for assessing the thermal shock damage resistance of the relatively brittle material seems to be less expedient, since for the specimens made from these materials it is technically difficult to provide a reliable contact with the ultrasonic vibration transducers, thus making realization of the immersion-type measurement virtually impossible.

ACOUSTIC EMISSION ACCOMPANYING FRACTURE OF CERAMICS

Evaluation of the degree of fracture of ceramics when investigating its thermal shock resistance can be performed by the data obtained in recording the signals of acoustic emission (AE) accompanying the formation and development of thermal shock damage in specimens, for example, during their quenching in water. However, the results of such measurements are often unreliable, since certain difficulties exist in separating the signals associated with fracture of materials in cooling from the signals caused by water boiling near the specimen surface. The experiments conducted by the authors demonstrated that monitoring of acoustic emission signals accompanying deformation of the thermal shock damaged specimens during their subsequent mechanical test is more effective, particularly, for relatively brittle materials. To illustrate this, Fig.5 presents the results of testing the corundum refractory under thermal shock loads. In the process of bending tests of the thermal shock damaged specimens of this material the acoustic emission count rate N has been recorded simultaneously with recording the stress-strain ($\sigma - \varepsilon$) relations (Fig. 5,a). The same specimens have also been employed in measurements of the ultrasonic wave propagation time at 150 kHz frequency by which the values of dynamic moduli of elasticity E were calculated. The data obtained are presented in Figs. 5,b and 5,c. Here, the results of the acoustic emission studies are shown as the dependence of a change in the stresses corresponding to the beginning of the acoustic emission signals - σ_A, normalized with respect to the specimen ultimate strength measured in pure bending $\sigma_{\Delta T}$ while the change in the modulus of elas-

ticity of the material resulting from the specimen thermal shock damage was normalized with respect to the magnitude of this value measured on the specimens not tested. When considering the relations presented in this Figure, it is easy to notice disagreement between the results obtained in investigating the residual strength of the material and its other characteristics: if $\Delta T_{beg.} = 300°C$ corresponds to the beginning of a change in the residual strength, then for the rest of the characteristics such difference ΔT_o equals 180°C. A reduction in the dynamic modulus of elasticity of the material and in the stress level $\sigma_A / \sigma_{\Delta T}$, as well as an increase in the specimen ultimate strain observed at the temperature differences less than $\Delta T_{beg.}$ may point to the fact that at the thermal shock load intensity ranging from ΔT_o to $\Delta T_{beg.}$ the material experiences fracture through development of microcracks saturating its structure and do not affecting its strength. At the temperature differences exceeding $\Delta T_{beg.}$ the dangerous cracks capable of subcritical growth and greater in size than a variety of defects inherent to the material are likely to develop on the specimen surface. Detection of the material fracture without recorded change in its strength in mechanical tests is essential for evaluating its performance under cyclic loading conditions at which these stresses will accumulate to result, in the long run, in softening. At the same time one must mention that fracture in structure and increased deformability of material without reduction in its strength may play a positive role in increasing the load-carrying capacity of products made from this material, since they can provide certain relaxation of acting stresses. The measurements of the acoustic emission signals when testing the brittle materials for thermal shock resistance have proved to be fairly informative too.

SUMMARY

The results presented in this paper indicate a significant effect the specific features of mechanical behaviour of ceramics have on the reliability of information on the degree of fracture of the thermally-stressed specimens obtained using nondestructive testing techniques. The most reliable evaluation of the degree of fracture following these testing techniques is achieved when the measurements are carried out with the relatively brittle materials which in the process of thermal shock loading feature gradual fracture of structure elements (growth of microcracks, numerous structure defects, etc.). At the same time, in the case of testing brittle materials in the specimens of which only single surface defects like cracks may be formed in the course of thermal shock loading, nondestructive testing techniques don't always allow to determine the fact of those cracks nucleation, i.e. to register the thermal shock fracture dangerous for the material and, consequently, to evaluate its actual thermal shock resistance.

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