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# Influence of residual stresses on thermal stress resistance of refractory ceramic

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#### Abstract

The conditions of formation of residual stresses (RS) in a thermo-loaded ZrC disk at the cost of relaxation of thermal stresses are considered. The creation of RS was carried out during the heating of samples by an electron beam and induction methods at different rates in a range 1600–2300 K. The kinetics of formation and distribution of RS depending on temperature, nonuniformity of the temperature field and temperature exposure period is investigated. The influence of the sign and level of residual stresses on the thermal stress resistance and fracture character are established. Acceptable modes of thermal loading, eliminating sample fracture under RS or the formation of a favourable stress field increasing the thermal stress resistance are defined.  $\bigcirc$  2000 Elsevier Science Ltd. All rights reserved.

Keywords: Thermal stress resistance; Residual stresses; Fracture; Carbides; ZrC

# 1. Introduction

The formation of residual stresses (RS) is possible in products of refractory ceramic materials in non-uniform temperature fields at high temperatures. The RS appear in ceramic heating elements, nuclear ceramic rods, ceramic lining of spacecrafts, metallurgical furnaces etc. The RS can cause an essential influence on the bearing capacity and duration of operation life.

It is known that the generation of residual compressive stresses on article surfaces of alumina by quenching in the silicon oil from 1700 to 2000 K increases the strength<sup>1</sup> and the resistance to the initiation of thermal stress fracture.<sup>2</sup> As a result of poor ductility of ceramic materials, in contrast to metals, they are strengthened at rather small values of the Biot number  $\sim 10^{-1}$ , e.g. by blowing a cold gas stream over a heated ZrC sample or by radiation cooling.<sup>3</sup> At higher values of Biot the rate of thermal-elastic stress relaxation turns out to be less than the rate of their increase, this leads to cracking. The temperature range of strengthening is limited in the lower domain by the temperature of the brittle–ductile transition and above the upper domain by undesirable structural changes causing the strength decrease or the appearance of cracks.

Nevertheless, the condition of the formation of RS in ceramic materials and especially the influence of RS on fracture under thermal loading has not been studied adequately. For this purpose the research of originative conditions of RS is being conducted using other means, permitting a wide variance of the RS level and methods for measuring it. An essential problem of the work is to determine the RS influence on thermal stress resistance and fracture behaviour of ceramics.

# 2. Experimental procedure

The experiments were conducted on disk samples of zirconium carbide with porosity ~5% and grain size 7  $\mu$ m. The module of elasticity E = 400 GPa, coefficient of thermal expansion  $\alpha = 9.0 \ 10^{-6} \ \text{K}^{-1}$ , thermal conductivity  $\lambda = 20 \ \text{W/mK}$ , bending strength  $\sigma = 250 \ \text{Mpa}$  and Poisson's ratio  $\mu = 0.25$ . All the properties are for the temperature range 20–1300 K.

The residual stresses arise during heating of the sample in the range 1600–2300 K by electron or induction heating methods. The schema of RS origination is the following:

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- heating of the disk with the establishment of radial temperature gradient;
- exposure of the disk with preservation of the constant value of temperature gradient at a particular temperature;
- cooling of the disk after reduction or elimination of temperature gradient.

In the case of the electron heating method,<sup>4</sup> the electron beam was focused at the centre of the disk with the electron spot diameter d=0.3 D, where D is the disk diameter within the limits of 20-24 mm and a height of 1-2 mm. The value of thermal flux Q in the sample is determined on the basis of electrical parameters of the electron device:  $Q = \beta UI$ , where U = voltage of anode, I = current through the sample,  $\beta$  = coefficient proportionality equal to 0.95 in this case. The Q is also measured by the water calorimeter attached to the sample with error not more than 10%. The temperature field in the disk under constant heat flux is calculated numerically from the data of the measured Q and can also be measured by thermocouples at 4-5 points or by a pyrometer. The elastic stresses in the disk can be calculated easily on the measured temperature field using quasi-stationary theory of thermal elasticity,<sup>5</sup> since the axial stress  $\sigma_z$  may be neglected for the plate samples with thickness  $\delta \ll D$ . The tangential stress  $\sigma_{\varphi}$  and radial stress  $\sigma_r$  at radius r=0 is compressive. The  $\sigma_{\varphi}$ becomes tensile with an increase of radius. Changing the thermal flux Q we may produce various levels of thermal stresses up to the fracture stress. The analogous experimental setup with electron beam which is guided over the graphite disk sample of a larger size is presented in Ref. 6.

The side surface of the ZrC disk of the same dimensions was heated up by the induction method.<sup>7</sup> The radial temperature gradient forming in the course of heating can easily be controlled by the electrical power of the device. The temperature field on the radius of the disk is measured by a fast-response pyrometer. The measurement time of the temperature field in 20 points comprises 0.2 s. The error of measurement does not exceed, in this case, 12%. The distribution of thermal stresses in the disk is calculated on the measured temperature field in the same way as in the case of electron beam heating. The values of calculated stresses in elastic approximation for ZrC are valid for a wide temperature range. The stress-strain diagrams under compression and tension are linear for ZrC up to 1500 and 2300 K accordingly at the moderate stress rate  $10^{-3}$  c<sup>-1.8</sup> The ZrC is brittle even at 3000 K at more higher stress rate up to  $10^{-1}$  c<sup>-1</sup>. We emphasise that thermal stress resistance (TSR) of ZrC is constant in temperature range 300-1600 K at the rate of thermal loading 1-40 MPa/s. The TSR increases at temperatures above 1700 K, as more as the lower rate of thermal loading<sup>9</sup> owing to the relaxation of the local stresses. It should be noted also that the long term loading of ZrC can cause a measurable creep at 1600 K.<sup>10</sup>

The exposure of samples in the nonuniform temperature field induces relaxation of thermal stresses in most heated parts of the disk and the redistribution of the stress components throughout the whole volume. Residual stresses with diagrams opposite in signs to thermal stresses occur after some exposure of the samples under thermal load and subsequent cooling by radiation heat transfer from the face side of the disk. The schema of distribution of the residual stresses under induction heating and electron-beam heating are given in Fig. 1. The tensile stresses appear in the opposite zones of the disk. The values of relaxed stresses relate nonlinearly to the plastic deformation level. Theoretical estimation of relaxed stresses under thermal loading even with a number of simplifications is rather bulky.<sup>11</sup>

The residual tangential stresses were evaluated experimentally by an X-ray  $\sin^2 \psi$ -method<sup>12</sup> with an error of 15 MPa for such high-module material as ZrC. In some cases the stresses were determined by the measurement of the slit entered into the stressed sample. General principles of RS determination are given in Ref. 13. There is practically a linear relationship between the disclosure and RS measured by the X-ray  $\sin^2 \psi$ -method and so the residual stresses can be estimated by a less tedious method. For example, the disclosure of the slit of 25 µm corresponds to 240 Mpa.

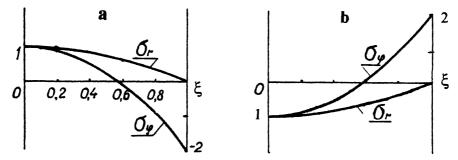


Fig. 1. The schema of distribution of residual tangential  $\sigma_{\psi}$  and radial stress  $\sigma_r$  versus  $\xi$  coordinates after electron-beam heating of central zone of the disk (a) or induction heating of lateral side (b).  $\xi = r/R$  is the scaled disk radius.

TSR is determined by two methods:

- by water cooling of the lateral surface of the previously heated disk with thermal-insulated end faces;
- or by heating the same disk assembly in melted tin.

The temperatures of the sample or the tin bath increased stepwise through 25 K up to the temperature of quenched samples  $T_c$  or the temperature of melted tin  $T_b$  causing their fracture. The maximum difference  $\Delta T_m = T_m - T$  is calculated numerically on the experimentally found temperature dependence of heat transfer in water<sup>14</sup> or in melted tin<sup>9</sup> and temperatures  $T_c$  or  $T_b$  at the moment of fracture. The  $T_m$  is the mean integrated temperature over the cross-section of the disk and T is the temperature of any point of the disk. Thermal stresses are evaluated on the  $\Delta T_m$  value:

$$\sigma_f = \alpha E \Delta T_m \tag{1}$$

## 3. Results

The residual tangential stresses induced in the disk after various exposures at prearranged temperatures are measured by the X-ray  $\sin^2 \psi$ -method and by the slit disclosure (Fig. 2). The residual stresses after various exposures are measured in most cases on the same disk. The maximum value of the temperature on the periphery of the disk is used in plotting the RS curve. The first signs of the thermal stress relaxation in the zirconium carbide are observed at a temperature of 1600 K. Appearance of plastic deformation at the mechanical loading by bending is at a higher temperature (2300 K).<sup>8</sup>

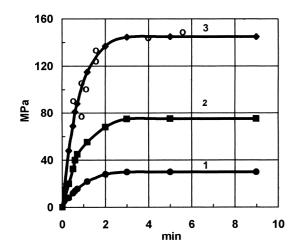


Fig. 2. Dependence of residual stresses on time of exposure and heating temperature under induction thermal loading of ZrC disks. The RS are measured by X-ray  $\sin^2 \psi$ -method after temperature exposure. 1. 1600 K; 2. 1800 K; 3. 2300 K; o — calculated values of RS with use of the relation (2).

The rate of relaxation with temperature rise increases and the process of relaxation and formation of residual stresses at 2300 K finishes in some minutes. The value of RS can vary, not only by the temperature level, but also by exposure period under thermal load. The formation of tension stress components on the periphery of the disk reduces the TSR measured by the quenching method the higher the level of residual stresses (Fig. 3). At first the tangential residual stresses were measured in the disks after induction or electron heating at various parameters. The quenching tests of 3-5 samples on each definite level of RS then were performed. The change of the TSR is governed by summation of the residual stresses with the tension thermal stresses during the quenching test. The compressive RS increase the TSR and the tension RS decrease it. The disks can crack spontaneously without thermal loading if the tension RS achieve the strength limit.

As the TSR changes nearly proportionally to the residual stresses value we propose to estimate the RS by the calculating method using the difference of thermal stress resistance values  $\Delta T_m^i$  and  $\Delta T_m$  respectively for samples with and without residual stresses.

$$\sigma_f = \alpha E \left( \Delta T^i_m - \Delta T_m \right) \tag{2}$$

The comparison between calculated and measured  $\sigma_r$  testifies their good consistence (Fig. 2).

The formation of residual tension stresses exceeding the strength limit results in the appearance of radial cracks in the peripheral zone of the sample without total fragmentation [Fig. 4(a)]. In the case of formation of compressive residual stresses in the peripheral disk zone the TSR is increased up to 2–2.5 times (Fig. 3). Any further increase of the thermal stress resistance due to compressive stresses appears to be impossible because of the increase of the tension stress components in the central part of the sample up to the strength limit. In

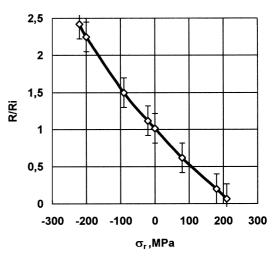


Fig. 3. Influence of the surface residual stresses measured by quenching on the TSR of ZrC disk.

this case the sample is fragmented completely [Fig. 4(b)]. Various kinds of fracture on the residual and thermal stresses are determined by a non-uniform stress field with a different ratio of tension and compressive zones and distribution of stresses in these zones.

Such heterogeneity of the stress field can be expressed on the basis of force fracture mechanics by a parameter N,<sup>15</sup> representing the magnitude of the average ratio value of tension tangential stresses  $\sigma_{\psi}$  (the averaging is made on the considered area of the crack) to a maximum value of temperature tension stresses  $\sigma_t$  at the fracture moment. For radial cracks in the disk this parameter is:

$$N = \int \sigma_{\psi} \mathrm{d}\xi / \sigma_t \tag{3}$$

where  $\xi = r/R$ , R – radius of the disk. The value of N for various distributions of residual stresses is calculated numerically.

The achievement of critical stress intensity coefficient  $K_{1C}$  in the central zone of the disk causes full fragmentation of the disk [Fig. 4(b)] since  $N \ge 0.1$ . The same happens when tests are conducted by heating the disk in melted tin even without RS. Testing of samples with and without the residual stresses by the quenching method at achievement of  $K_{1C}$  in the peripheral zone of the disk is finished only by partial failure since  $N \leq 0.1$ [Fig. 4(a)]. This is due to the fact that the crack started in the region of tension stresses is capable of propagating in the compressive stress zone equally under continuous increase of thermal load. The total fragmentation comes only through interaction of spreading cracks at higher load.<sup>16</sup> The fracture of the brittle body by the mechanical compressive load stems just the same way from the interaction of cracks at stresses 8-12 times higher than the stress of crack start.<sup>17,18</sup> By contrast to the energetic principle of fracture, 19,6,20 neglecting a kind of stress condition, our criterion  $N_{\rm C}$  takes proper account of the physical difference in the propagation mode of cracks in the field of tension and compression. The general store of fracture energy for the compressed

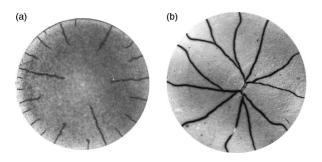


Fig. 4. Type of fracture under tension residual stresses in a peripheral (a) or in a central zone (b) of ZrC disks.

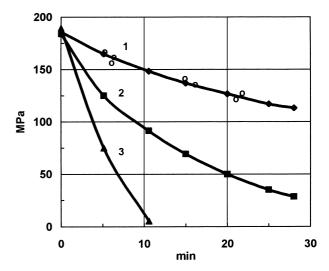


Fig. 5. Residual stresses in ZrC disks measured by X-ray  $\sin^2 \psi$ method after annealing. 1. 1600 K; 2. 1800 K; 3. 2000 K; o — the values of (RS) determined by the disclosure of the slit.

body exceeds many times the level of the stored energy necessary for fracture of an elastic body in a uniform field of tension stress.

The formed residual tension stresses in the peripheral zone of the ZrC disk after induction heating are stable in samples at temperatures of annealing less than 1600 K. A higher temperature and longer exposure of the annealing decrease residual stresses readily (Fig. 5). It is very interesting to compare the measured relaxation rate with data of other authors but the investigations on the thermal relaxation are very scarce. Fortunately, our colleague Professor Zubarev performed the investigation of relaxation of ZrC under bending in the temperature range 1900-2500 K.<sup>21</sup> The curves of relaxation have a similar form to ours but the rate of the relaxation at related temperature is lower by a factor of 10. Such a difference is moderate for creep and relaxation and may be associated first of all with the distinction of stress state and with a variance of composition and structure.

To decrease dangerous residual stress levels after full cooling down of the thermo-stressed products, it is necessary to anneal them at an intermediate temperature level. The time of annealing is selected to be sufficient for the partial relaxation of stresses at the chosen temperature.

### 4. Conclusion

1. The conducted research established the influence of residual stresses on the thermal fracture character. The start of a crack in the central zone of a disk with the thermal or residual tension stresses is completed by full fragmentation. The thermal stresses or stresses combined with residual in the peripheral zone of the disk causes only partial failure at the achievement of critical stress intensity coefficient  $K_{1C}$ . Total fracture is made possible after increased thermal loading by a factor of 8–12. The parameter of heterogeneity of stress field N and criteria  $N_{\rm C}$  are introduced for the predication of fracture type on the base of linear fracture mechanics.

- 2. Different distribution of RS can originate in a ZrC disk with use of induction or electron-beam methods at variance with heating parameters.
- 3. The kinetics of formation and distribution of RS were estimated: by X-ray  $\sin^2 \psi$ -method, by way of disclosure of a slit introduced into the stressed sample and by calculation of the difference between values of the TSR of the sample with residual stresses and without them. The values of RS determined by the three methods are in close agreement.
- 4. Acceptable modes of thermal loading, eliminating sample fracture under RS or the formation of favourable stress field increasing the thermal stress resistance are defined.

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