The Significance of Non-Elastic Deformation in the Fracture of Heterogeneous Ceramic Materials

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The classification of refractory ceramic materials is considered according to the magnitude of their brittleness i.e. the ratio of the specific elastic energy stored in the specimen at the moment of fracture, to the total amount of deformation energy of the specimen prior to its fracture. The materials are classified as brittle ($\varkappa = 1$) and relatively brittle ($\varkappa < 1$). Deformation peculiarities of these materials are analysed and distinctive features of their mechanical behavior are pointed out. It is shown that macrononelasticity of relatively brittle materials is caused by microfractures. It is proved that these features must be taken into consideration when developing design criteria for these materials. The known thermal shock resistance criteria are considered and a new criterion is proposed in which brittleness is introduced as a parameter. The validity of the approach is confirmed by results of tests of single-phase and composite ceramic materials based on aluminium oxide, yttrium oxide, zirconium dioxide, and titanium carbide.

1 - INTRODUCTION

Refractory ceramic materials possessing a number of such important properties as high melting temperature, low heat conductivity, relatively weak chemical activity, etc. are nowadays more and more extensively used in engineering structures, which brings about a considerably growing interest as to how to make more accurate an evaluation of their strength under mechanical and thermal loading. That is why the traditional methods for determining mechanical characteristics of the materials under consideration, and calculations of strength of engineering structures where these materials are used, are not always acceptable. This is conditioned by the fact that the application of such idealized models can often be considered only as a first approximation [1-3] since the non-elastic strain under stresses less than the limiting ones is characteristic of many of them even at room temperature [4-6].

2 - CLASSIFICATION OF MATERIALS

In connection with the essential difference among the mechanical behaviour of such materials, it is good practice to group them by their characteristic features. The interest in the question of dividing materials as to the features of their mechanical properties had been suggested many times (Ref. 7-9 and others) and, for example, any thermal stress resistance criterion may be considered in terms of an order of merit for the materials. But up till now none of the classifications has been widely used, probably due to the fact that they had been developed for particular conditions of loading or based on highly approximated parameters. In paper [10] it is suggested to classify the refractory ceramic materials in terms of their mechanical behaviour. To characterize the latter the energetic parameter-brittleness ($\varkappa$) was introduced; this parameter is defined by the ratio of the specific elastic energy u_e which had been accumulated in the material at fracture, to the whole specific energy u expended to attain the limiting state. If we consider the dependence of stress σ on strain ε shown in Fig. 1, and assuming that the elastic moduli are equal under loading and unloading, the expression for brittleness can be presented in the form:

$$\varkappa = \frac{\sigma^2_{lim}}{2E \int^{\varepsilon_{lim}} \sigma \cdot dE} \qquad [1]$$

where: σ^{lim} is the strength, E stands for the elastic modulus, ε^{lim} representes the strain of the material. As it follows from the definition, brittleness rests within the limits $0 \leq \varkappa \leq 1$.

According to the classification reported in Ref. 11, refractory ceramic materials fall into two major groups: brittle and relatively brittle materials. Brittle materials which are characterized by $\varkappa = 1$, include elastic materials (being subjected to the Hooke's law) right up to a fracture. Such are, mainly, homogeneous, single-phase fine grained oxidic and other compounds like glasses, glass-ceramics, porcelains. Brittle materials are deformed without any structural changes and the moment when their structure starts breaking up, coincides with the beginning of movement of the structure crack, which may arise on the basis of the macro- and mi-

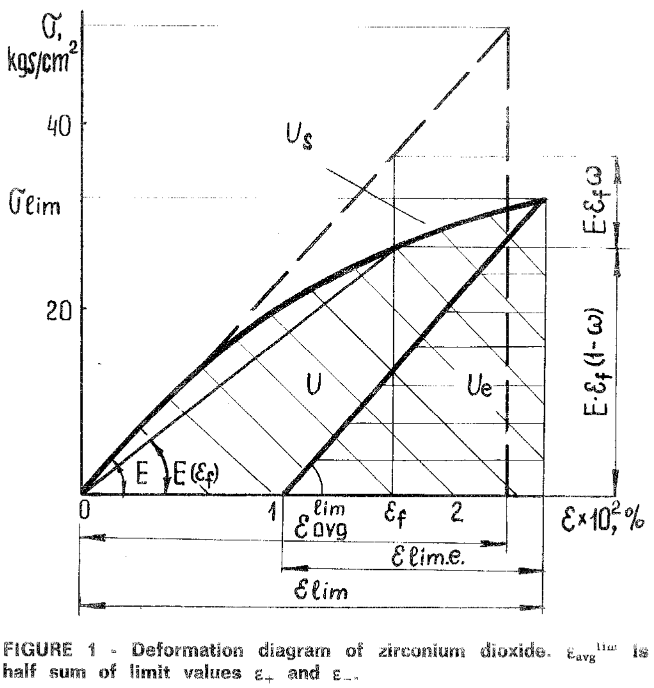

FIGURE 1 - Deformation diagram of zirconium dioxide. ε_{avg}^{lim} is half sum of limit values ε_+ and ε_-.

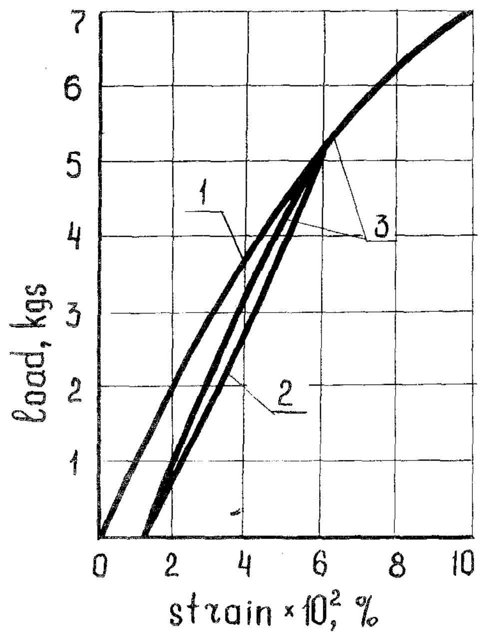

FIGURE 2 - Load-strain dependence for a nitride ceramic specimen 1 - loading; 2 - unloading; 3 - repeated loading.

crodefects of the structure. Regularities in the development of cracks for these materials are well enough described by the Griffith model [12]. More pronounced rating of the effective surface energy, crack initiation determined than in its propagation is more characteristic of these materials [13]. A much larger rating of nominal stresses to initiate fracture than to propagate it makes it possible to consider initiation barrier of prime importance [14]; the load-carrying capacity of an engineering structure is determined to a great extent by the level of the initiation barrier.

Relatively brittle materials, which are characterized by values of $\varkappa < 1$, include those which under loading conditions have some amount of energy dissipated because of non-elastic effects. These materials exhibit non-linear stress-strain behaviour, which reflect the occurrence of non-elastic strains at certain stress levels.

If the limit of proportionality is exceeded while loading specimens of relatively brittle materials, than residual deformations are found during unloading. The above specified deformation features are cheracteristic of many industrial refractory ceramic materials [5], certain composites [15, 16], heterogeneous structure ceramics (Fig. 2). An analogous mechanical behaviour is observed in such materials as graphites [17] and concretes [18]. Although the deformation mechanism has been only worked out in general, it is by now clear that the most characteristic structural feature of relatively brittle materials is associated with the great number of microcracks developed during fabrication or deformation, caused by ruptures of contacts between grains, different phases, filler and matrix, fracture of pores etc. Moreover there is observed a correlation between crack density and brittleness of the ceramic material; the crack density is determined according to the procedure described in Ref. 19. Development of macrocracks in such materials is accompanied by further damage of the structure, which is related with the development of secondary cracks, whose energy consumption might be by one order higher than that for the major crack [20]. The capability of relatively brittle materials to inhibit or block damaging cracks is related to this peculiarity. As brittleness of these materials is reduced, the magnitude of the initiation barrier is lowered and the difference in material resistivity to form and develop cracks is reduced. This phenomenon, and to some extent the capability to relax stresses on account of local fractures causes the reduction of sensitivity of such materials to surface damages and to other stress concentrations.

The variation of the mechanical behaviour of refractory ceramic materials with temperature may be characterized by the reduced brittleness i.e. the ratio of the brittleness at a preset temperature $\varkappa_T$ to the brittleness at room temperature $\varkappa_{20}$ i.e.

$$\varkappa = \frac{\varkappa_T}{\varkappa_{20}} \qquad [2]$$

The above mentioned reduced brittleness, being equal to 1 at the room temperature, assumes other values only when the mechanical properties of the material change with temperature (e.g. in case of the brittle-ductile transition).

It should be noted that it is impossible to classify refractory ceramic materials into brittle and relatively brittle ones from the porosity value or the chemical composition, since not a single one of the described characteristics bears information on the behaviour of the material under loading. Indeed, the results presented in Fig. 3, show that in spite of the changes in porosity from 0.5 to 30 per cent, brittleness and, consequently, the character of material deformation practically remains the same (curves 1 and 2 in Fig. 3). At the same time the introduction of a second phase (in an amount of < 3%) considerably reduces brittleness (curve 3 in Fig. 3), changing essentially the mechanical behaviour of the material.

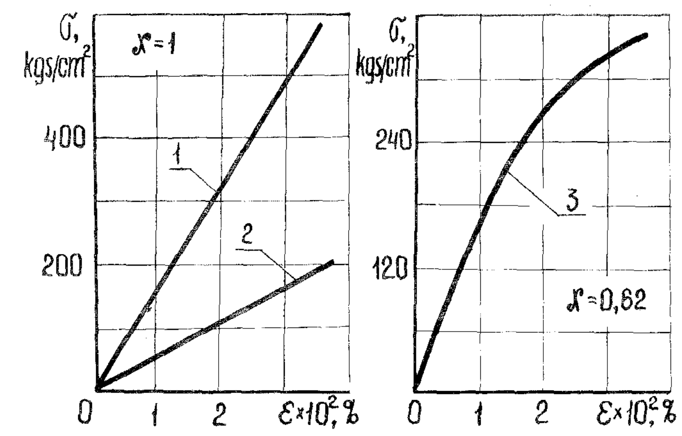

FIGURE 3 - Deformation diagram of yttrium oxide based materials 1-3 - indexes of materials.

The suggested division of the materials and the introduction of the parameter $\varkappa$ do not contradict the known groupings, but in this case it is possible to resort to the exact quantitative characteristic, having a clear-cut physical sense, which sometimes has not been so with the other classification parameters [21].

The brittleness value may also be used for the analysis of the material deformation diagram. Diagrams are usually studied with the help of the dependence $E/E(\varepsilon_i)$ where $E(\varepsilon_i)$ is a secant modulus (see Fig. 1), which is defined by the relation of the stress of the corresponding fluid strain E_i, i.e. $E(\varepsilon) = \sigma(\varepsilon_i)/\varepsilon_i$. The limiting value of this parameter, called the modulus of fracture (i.e. relation $E/E(\varepsilon_{lim})$), was used in Ref. 4 to characterize the material brittleness, although it would probably be more convenient to introduce [10] $\varkappa' = E(\varepsilon_{lim})/E$ since this magnitude is limited $(0 \leq \varkappa' \leq 1)$. The stress-strain relation can also be described by Jljushin's function [22] $\omega = \omega(\varepsilon)$ such one, that $\sigma(\varepsilon) = E \cdot \varepsilon\,(1 - \omega)$ i.e. evaluating the deviation of the deformation diagrams from a straight line.

The form of the deformation diagram is also characteri-

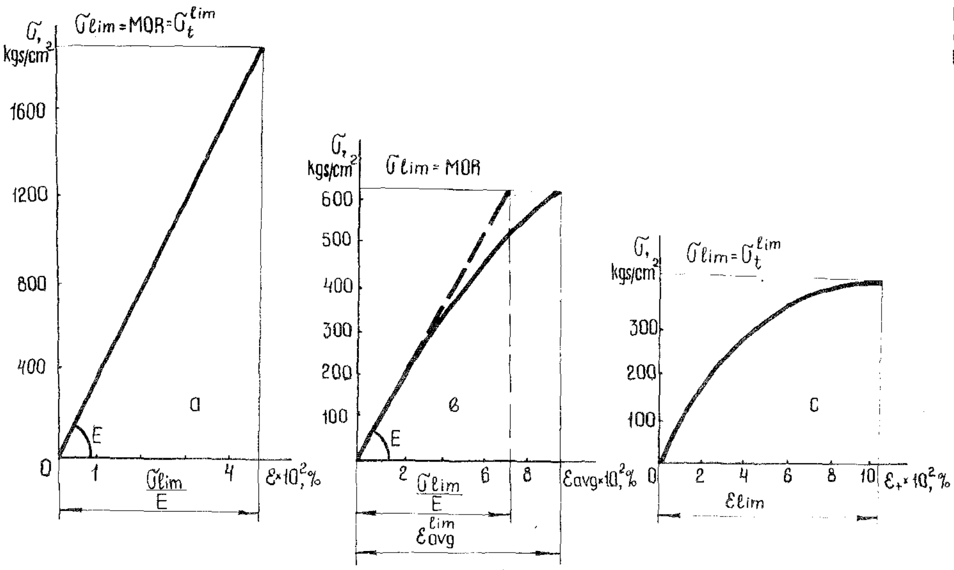

FIGURE 4 - Deformation diagrams of corundum materials: a - material 4; b, c - material 12.

zed by the brittleness measure. It should be noted that ϰ can be expressed through the functions or parameters mentioned above which bear a geometric rather than physical sense.

The classification considered not only provides for the systematization of refractory ceramic materials, but also suggests additional information on peculiarities of their mechanical behaviour, thus facilitating a better choice of those materials to be used in engineering. Thus a brittle material may be used in repeated loading suffering no fracture, in spite of the easy crack propagation, the catastrophic mode of fracture and the high sensitivity to stress concentration.

If the material is relatively brittle then in repeated loading one should take into consideration the changes in its mechanical properties after each loading, as well as an appearance and growth of residual deformations, when the stresses exceed the elastic limit. With relatively brittle materials, a considerable creep and an ability to relax stresses is observed. Sensitivity of such materials to concentration of stresses is lower than for brittle materials, and it decreases as brittleness is reduced.

3 - PECULIARITIES OF DETERMINATION OF MECHANICAL CHARACTERISTICS

Differences in the mechanical behaviour of refractory ceramic materials can be so large that different procedures are probably required to determine their strength. Thus in investigating the mechanical characteristics of brittle materials, which in practice are carried out most commonly under bending conditions, it suffices to register the limiting stress, to calculate the strength by formulas for the strength of materials, and to calculate the limit deformation according to the Hooke's law. In this case the deformation diagram is a straight line, which si defined by the strength and the elastic modulus of the material (Fig. 4a).

On the contrary, relatively brittle materials deform non-linearly under loading, their mode of deformation being often quite dissimilar in tension and compression. By determining the strength of these materials from the maximum load sustained by the specimen in bending, it is possible to calculate only the modulus of rupture (MOR). The deformation diagram as culculated by the modulus of rupture and elastic modulus, rather vaguely reflects the properties of the material (dotted line in Fig. 4b). If we register not only the load P, which is applied to the specimen, but also the strain in the specimen then in making use of the relation:

$$\sigma_n = \frac{3 \cdot l}{bh^2} P \qquad [3]$$

where b is the width, h is the thickness of the specimen of rectangular cross-section, and l is the loading span, it is possible to plot a nominal deformation diagram. It should be noted that MOR is the maximum rating of σ_n. Such diagram (solid line in Fig. 4b) reflects qualitatively the character of material deformation, though strength magnitudes would reflect the more superfluously exceeded ratings, the less is the rating of the brittleness measure ϰ. By applying the following formula 2:

$$\sigma_{avg.} = \frac{2l}{bh^2}\left(P + \frac{\varepsilon_{avg.}}{2} \cdot \frac{dP}{d\varepsilon_{avg.}}\right), \qquad [4]$$

(where $\varepsilon_{avg.}$ is an average deformation of the specimen) we are in a position to determine precisely the strength and to plot a deformation diagram only for those materials which deformed similarly in tension and compression. If this condition is not observed then this relation also results in exceeding in the strength of material [6]. In this case expression 5 may be used to plot a deformation diagram, which represents a dependence between tension stresses (Fig. 4c), acting in the outer layers of the specimen and their deformations [6]:

$$\sigma_t = \frac{l}{bh^2}\left(P + \frac{1}{2}\,\frac{\varepsilon_+ + \varepsilon_-}{\varepsilon'_+ + \varepsilon'_-}\right)\left(1 + \frac{\varepsilon'_-}{\varepsilon'_+}\right) \qquad [5]$$

This relation is similar to that described in Ref. 17. Here ε_+ is a deformation of tensiled outer layers, ε_- — is a deformation of compressed outer layers of the specimen. To determine derivates $\varepsilon'_+ = d\varepsilon_+/dP$ and $\varepsilon'_- = d\varepsilon_-/dP$ the relations $\varepsilon_+ = \varepsilon_+(P)$ and $\varepsilon_- = \varepsilon_-(P)$ are used; they are registered in the experiment. Magnitudes of strength of refractory ceramic materials, which are determined by relations [3-5], are presented in Table I, and are designated as σ^{lim}_{avg} and σ^{lim}_t respectively. The magnitude of the tensile deformation corresponds to the limit if strength is the limiting deformation of material (Fig. 1).

Elastic modulus E which is determined by an incline of the tangent to the curve of deformation at stresses close to zero, is found almost equal (or a little bit less) to the values of the dynamic elastic modulus E_{dyn} (Table I).

4 - EVALUATION OF THERMAL SHOCK RESISTANCE

There is a considerable difference in behaviour of brit-

TABLE I

Material	Index of material	Density g/cm³	Porosity %	Strength kgs/cm²			Strain	Elastic modulus $\times 10^{-6}$, kgs/cm²		Brittleness measure	Coefficient of thermal expansion
				MOR	δ_{avg}^{lim}	δ_t^{lim}	$\varepsilon_{lim} \times 10^2$ %	E	E_{dyn}	χ	$\alpha \times 10^6 deg^{-1}$
Dense yttrium oxide	1	4.75	0.5	596	596	596	3.8	1.48	1.60	1.00	4.75
Porous yttrium oxide	2	3.45	30.0	209	209	209	3.9	0.54	0.43	1.00	5.35
Two-phase yttrium oxide	3	4.71	2.0	326	291	291	3.6	1.10	1.40	0.61	6.30
Lucalox type ceramics	4	3.97	0	1916	1916	1916	4.8	4.05	4.00	1.00	11.0
Aluminium oxide	5	3.72	1.2	1180	1180	1180	3.5	3.33	3.80	1.00	6.00
Reinforced aluminium oxide with 7% aluminium oxide monocrystals	6	3.66	3.1	649	631	615	2.6	2.64	—	0.85	6.70
Reinforced aluminium oxide with 15% aluminium oxide monocrystals	7	3.51	5.1	676	588	524	3.1	2.36	—	0.61	7.20
Titanium carbide	8	4.45	8.1	2395	2395	2395	6.1	3.90	3.70	1.00	—
Carbon reinforced titanium carbide	9	3.41	4.5	685	575	440	12.0	1.00	1.20	0.37	—
Nitride ceramics	10	1.90	—	1888	167	167	10.0	0.24	0.28	0.59	3.50
Zirconium dioxide	11	4.40	17.0	46	40	31	3.2	0.19	—	0.38	10.0
Two-phase aluminium oxide	12	3.45	23.0	671	576	470	9.6	0.94	—	0.40	7.60

tle and relatively brittle materials during thermal shock tests carried out according to a procedure similar to the one presented in Ref. 23. Thus for brittle materials in sympathy with the theory represented in Ref. 24, a different reduction in strength at a quenching temperature difference exceeding the critical one is observed on the thermal damage diagram. Relatively brittle materials on deformation do not follow Hooke's law, and their behaviour under thermal shock is not strictly described by the results reported in Ref. 24. For relatively brittle materials the thermal damage diagram represents a continuous curve, that is why it is senseless to search for a critical quenching temperature difference. Thus a diagram (with an accuracy sufficient for critical purposes) might be plotted approximately by values of the initial strength of the material and strength should be measured after a thermal shock of preset intensity. We would like to note in our view one interesting point. The brittle material (Fig. 5a) after being subjected to a thermal shock whose intensity exceeds the critical value, is deformed non-elastically (Fig. 5b) i.e. it behaves as a relatively brittle material.

For investigations of thermal shock resistance under controlled conditions of thermal load onto hollow cylinders [25] which are further developments of investigation [26], it is also essential to consider which type of material is being tested - brittle or relatively brittle. Thus rupture of brittle materials is detected simply by a damage of an electric conductive layer put onto the surface, or by an abrupt increase of a size of the specimen. For registration of rupture of relatively brittle materials, the above methods proved to be ineffective, and one should search for some others procedures as, for example, that of acoustic emission, based on the analysis of the spectrum of noises accompanying ruptures.

To make a criterial evaluation of the thermal shock resistance of refractory ceramic materials, peculiarities of their mechanical behaviour are also to be taken into account. Thus estimating resistance of brittle materials to thermal stresses, criteria R, R' and R'' are effective. The development of the thermal cracks in such materials is described by the theory of Griffith, which was probably used for the first time in Ref. 27, and it has found its reflection in criteria R''' and R^{IV} (Ref. 24). These or some other criteria, are based, for example, on the static theories of strength. The criteria proved

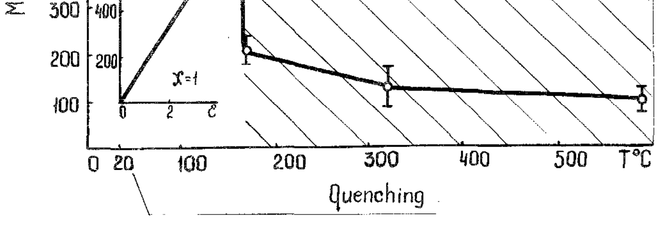

FIGURE 5 - Thermal damage diagram for dense yttrium oxide (quenching in water at 20°C).
T - heating temperature of the specimen, °C;
a - deformation diagram at T = 20°C;
b - deformation diagram after quenching from 170°C;
ε - strain, 10^{-2} %;
σ - nominal stresses, kgs/cm²;
Φ - mean ± deviation.

to be valid for brittle materials but they have to be specified when applying the latter to relatively brittle materials. Indeed, all of them were obtained assuming that the behaviour of refractory ceramic materials is described by the model of a linear-elastic body [28]. This is evidently one of the main reasons as to the observation of coincidence of the analytical and experimental results in determination of thermal resistance in one group of cases, and discrepancy for the other group of cases.

As for the criterial of R-type, the relation σ_{lim}/E presents the magnitude of limit strain ε_{lim} for brittle materials and expresses a logic assumption that the more material is subjected to deformation, the more thermal resistive it is, all other conditions being equal. Since for relatively brittle materials is only an elastic component of limit strain then in development of investigation [29] one can suggest, as a criterion of thermal stress resistance, the relation of limit strain registered at tensile under bending conditions, to the coefficient of thermal expansion α. Let us designate this criterion as R_a (where a means actual).

Thus $$R_a = \frac{\varepsilon_{lim}}{\alpha}$$

For brittle materials, criteria R_a and R coincide.

To obtain the criterion, which could reflect an actual connection between stresses and strains and also considering as it is a relation of thermal resistivity against the elasticity strength, brittleness and linear expansion coefficient, let us consider the stressed state of a component of the structure of refractory ceramic materials. During heating tensile stresses appear in its relatively cold layers which are loaded due to expansion of the hotter layers. The stressed state in dangerous zone of many elements (plates, cylinders, tubes) with their one-side heating may with considerable precision be considered as plain strain. That is why it is possible to compare it with the stressed state in a flat specimen being cooled, rigidly fixed after heating by ΔT degrees. The total strain of such a specimen, being equal to its relative thermal expansion, is described by the equation:

$$\varepsilon_o = \alpha_1 \cdot \Delta T \qquad [6]$$

Here the magnitude α_1 is related to the coefficient of thermal expansion α by relation [30] $\alpha_1 = \alpha(1+\nu)$ where ν is Poisson's ratio.

After cooling the total deformation of the fixed specimen, which in case it is a free one would decrease, does not alter, resulting in appearing in it tensile stresses and, after all, in damaging. Assuming the criterion of the material resistance to thermal shocks to be a difference of heating temperature in the moment of the rupture and let us designate it as C (the first letter of the Russian term meaning « resistance ») we shall find

$$\varepsilon_o = \alpha_1 (\Delta T - C) + F(\sigma_{lim}) \qquad [7]$$

where $F(\sigma)$ is a function describing its deformation diagram.

Let us assume dependence $F(\sigma)$ to be parabolic and assume that it should correctly reflect such main properties of material as strength, elastic modulus and specific energy expanded on deformation before rupture. Considering that the elastic modulus of material is equal to the slope of the tangent to the deformation curve at $\sigma \approx 0$, and using the definition of the brittleness measure, we shall find that:

$$F(\sigma) = \frac{1}{E_1} \cdot \sigma + \frac{3}{4} \cdot \frac{1 - \varkappa}{E_1 \sigma_{lim} \cdot \varkappa} \sigma^2 \qquad [8]$$

where the magnitude E_1 is connected with the elastic modulus at uniaxial stress by relation [30]: $E_1 = \frac{E}{1 - \nu^2}$.

TABLE II

Index of material	R^{III} cm²/kG	$R^{III}a$ cm²/kG	Number of heat changes before rupture * 1300⇄20°	Peculiarities of rupture of cylindrical specimens **
4	1.1	1.1	1	
5	2.4	2.4	1	
6	6.3	7.0	8	
7	5.1	8.6	12	
12	2.1	4.3	5	
11	89.8	197.7	—	

* Specimens — small cubes with the volume of 3.0 x 3.0 x 3.0 cm.
** Specimens with outside diameter of 5.0 cm, inside diameter of 2.5 cm, height of 1 cm were monotonously heated inside at the rate of 200°/min.

We shall see then from [6]-[8] that criterion of thermal shock resistance may be expressed in the form of:

$$C = \frac{\sigma_{lim}(1 - \nu)}{\alpha E} \cdot \frac{\varkappa + 3}{4\varkappa}. \qquad [9]$$

It follows from [9] that with strength, rigidity and linear expansion coefficient being equal, that a material is more thermally resistant the less brittle it is. At $\varkappa - 1$, i.e. for brittle materials criterion C changes into the known criterion [27]

$$R = \frac{\sigma_{lim}(1 - \nu)}{\alpha E} \qquad [10]$$

For the refractory ceramic materials operating at non-uniform temperatures the criteria may be suggested like in Ref. 27

$$C^I = C\lambda \text{ and } C^{II} = CQ$$

where λ is thermal conductivity and Q is thermal diffusivity of material. By developing the idea that the criterion of material resistance to the development of the thermal cracks may be a relation of effective surface energy γ_{eff} to the energy accumulated in the unity of the volume of the material before its rupture, we can generalize criteria R^{III} and R^{IV} also for the relatively brittle materials. Thus, for example, when using the results of bending tests, one should consider not an apparent energy U_s (Fig. 1) calculated by the value of the modulus of rupture and the elastic modulus, but only the elastic component U_e of the energy accumulated in the material since only this component can be expended for the development of a crack. The remaining amount of the energy related to non-elastic effects, dissipates unreversibly in the material. Thus criteria R^{III} and R^{IV} for relatively brittle materials may be written in the form:

$$R_a^{III} = \frac{1}{\varepsilon_{lim.e.} \cdot \sigma_{lim}},$$

$$R_a^{IV} = R_a^{III} \cdot \gamma_{eff}$$

where $\varepsilon_{lim.e.} = \sigma_{lim}/E$ is the elastic component of the limit strain. As an example of the criteria which reflect an actual relation between stresses and strains, let us consider the data presented in Table II. It gives the magnitudes of criteria R_a^{III} and $R_a^{III} = E/(MOR)^2$ as well as the results of the experimental estimate of resistance of material to rupture. As it is seen from the data presented, the suggested criterion R_a^{III} describes the results of the experiments more accurately in comparison with R^{III}.

5 - SUMMARY

While investigating strength under conditions of mechanical and thermal loads one should take into consideration the actual behaviour of the materials. In this connection it is good practice to classify refractory ceramic materials by peculiarities of their de formation making use of the magnitude of the brittleness measure.

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