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SOME FEATURES OF FORMATION OF THE STRUCTURAL STRENGTH OF CERAMIC MATERIALS

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On the basis of preliminary investigations of the characteristics of the crack-propagation resistance of various types of ceramic and cermet materials, we give a general evaluation of their potentialities in the context of the influence of the structural-metallurgical factor and the micromechanisms of fracture on the serviceability of the materials. We propose to evaluate the mechanical efficiency of ceramic materials by comparing their diagrams of structural strength at room and service temperatures. We found certain advantages of cermets in ensuring crack resistance under short- and long-term loading. We propose a classification of dominant and secondary micromechanisms of stress relaxation that manifest themselves in the process of crack growth in ceramics and cermets.

Owing to the significant advantages of ceramic materials over traditional metallic materials with respect to the surface strength (hardness, wear resistance, erosion resistance, and resistance to corrosion fracture) at high temperatures, the former are more widely used for the production of elements of the piston-link assembly of internal-combustion engines, the nozzle-blade assembly of gas and steam turbines, the combustion chamber of rocket engines, etc. [1]. This favors an increase in serviceability, ensures particular functional properties of the components and units of certain types of equipment, and creates potentialities for the production of machines and engines whose characteristics are significantly better than the corresponding characteristics of their traditionally made analogs.

However, ceramic materials are susceptible to brittle fracture due to low crack resistance under short- and long-term loading. For this reason, the problem of extension of the functional application of ceramic materials is reduced, first of all, to the development of methods for increasing the crack-growth resistance, which is important for ensuring sufficient structural strength [2].

In the present work, on the basis of investigations of crack-growth resistance performed earlier, we compare potentialities of ceramic materials developed in this country in the context of the influence of the structural-metallurgical factor and the micromechanisms of fracture on their serviceability.

Materials and Methods for Evaluation of Their Properties

To perform comparative evaluations, we use three types of domestic ceramic materials that reflect the world trend toward the modification of structural ceramics (Table 1). They include an alumina composition (Al_2O_3), which is a specific model of simple monophase ceramics, an alumina ceramics modified with zirconium dioxide (Al_2O_3–ZrO_2), and a typical silicon-nitride ceramics modified with yttrium oxide (Si_3N_4–Y_2O_3). Both modifications of ceramics were preliminarily tested for a long period of time in order to choose the optimal ratio of the components and the technological mode of sintering that allowed one to guarantee the optimal level of their structural strength [3–8]. In addition, for comparison, we consider Ti–Si cermets of electroslag, plasma-arc, and electron-beam melting with a two-phase ($\alpha + \beta$) structure of titanium intended for parts of adiabatic internal-combustion engines. The content of silicon and zirconium in this cermet varied within the ranges 2–8.5 and 2–13 wt. %, respectively (Table 1). Since a cermet of the type WC–Co is widespread among cutting, in particular, boring, tools, we consider this cermet as well.

To evaluate the serviceability of ceramic materials by plotting adequate structural-strength diagrams for them, we must compare their structural strength (surface and bulk) with the level of fracture toughness (K_{Ic}) and investi-

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gate serviceability in the case of subcritical crack growth. It is most convenient to evaluate these characteristics under laboratory conditions at room temperature, although it makes sense to record simultaneously the stability of the characteristics in the range of high service temperatures. By using previously approved methods [8, 9], we determined the limit of short-term bending strength σ_u and the Vickers or Rockwell hardness on smooth prismatic specimens. Fracture toughness (K_{Ic}) was evaluated on beam specimens with sharp lateral notches. For WC–Co cermet, we also used specimens with chevron notches. By taking into account specific difficulties caused by the creation of fixed cracks, we investigate their subcritical growth on specimens with initial sharp lateral notches under mild and severe loading.

Results of Testing Ceramic Materials

In what follows, we present briefly the results of investigations of important mechanical characteristics of all five ceramic materials and evaluate the influence of the structural-metallurgical factor and the micromechanisms of fracture on their serviceability. They are based on the already published [3–8] and unpublished data.

1. Alumina Ceramics (Al_2O_3)

Monophase ceramics based on sintered Al_2O_3 particles belongs to the simplest ceramics of model type. Just for this reason, intergranular fracture can be realized in this ceramics in a pure form (however, for a high inhomogeneity of the structural particles-grains and, in particular, in the presence of individual large grains, regions with intragranular fracture can also be observed [10]). According to our estimates for smooth beam specimens, their bending strength increases as the mean size of the Al_2O_3 particles decreases (Fig. 1a). At the same time, the fracture toughness nonmonotonically depends on the grain size d and reaches a maximum for a certain value of its mean size [11] (Fig. 1b). This dependence of K_{Ic} on d is caused by the fact that, for a simple ceramics, fan-like intergranular cracking, which induces a specific intergranular "loosening" of the crack tip, is the main mechanism of stress relaxation and energy dissipation in the process of fracture (scheme 1 in Table 2). In a fine-grained ceramics, the crack is quite sharp despite the intergranular growth and slightly deviates from the main direction. But in a coarse-grained ceramics, a ramified "fan-like" shape of the front part of the crack is observed. It is such a mechanism of stress relaxation that causes the unique phenomenon of retardation (and even arrest) of a subcritical crack under the action of mild static loading (Fig. 2). Fett and Munz observed similar behavior in simple ceramic materials in [12–14].

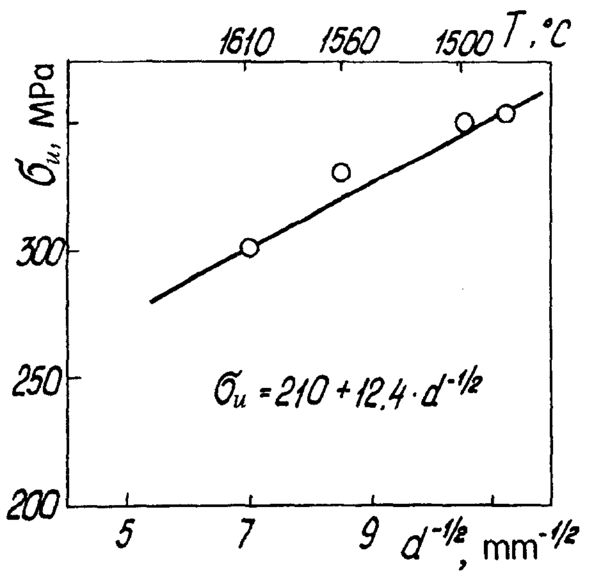
2. Alumina Ceramics Modified with Zirconium Dioxide

Various Al_2O_3–ZrO_2 ceramic compositions are promising materials of new technology due to an enhanced level of toughness, in particular, at high temperatures (600–800°C). As shown in [4, 5], the toughness of this ceramics is increased owing to a large amount of tetragonal zirconium dioxide in the disperse material, which depends both on the mode of sintering of the ceramics and on the absolute content of ZrO_2 (at most 15 wt. %, Fig. 3). For this ratio of the components, tetragonal ZrO_2 transforms into the monoclinic phase, which favors the appearance of compressive stresses near a crack tip, unloading it (scheme 2 in Table 2). This hypothesis was corroborated by the direct analysis of the phenomena occurring in the vicinity of the crack tip in zirconium ceramics by Raman spectroscopy [15]. The above-mentioned fan-like intergranular cracking serves as an additional factor in the formation of toughness in ceramics of the Al_2O_3–ZrO_2 type. However, since the structure becomes more complicated, the effects of an abrupt drop in the rate under long-term static loading are absent, and only slight "teeth" are observed on the $v-K_I$ curves (Fig. 4).

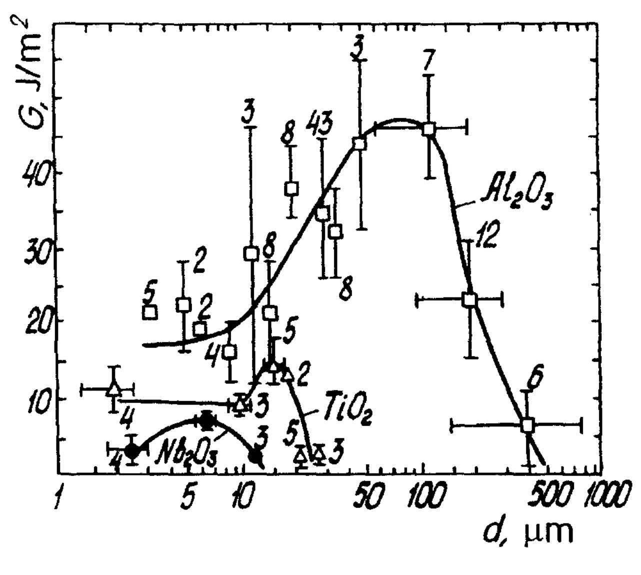
Table 1. Geometric and Physical Characteristics of Materials and Elements of Their Structure

Material		Type of technology	Characteristic of structural elements		
			Stoichiometry	Ratios of phases, wt. %	Size of sintered particles, μm
Al_2O_3		solid-phase sintering	Al_2O_3	> 99.6	5–25
			—	—	—
Al_2O_3–ZrO_2		solid-phase sintering	Al_2O_3	85–88	0.6–0.8
			ZrO_2 (t) ZrO_2 (m)	8.5–10.5 (t) 3.5–4.5 (m)	0.1–0.2 (t) 0.2–0.3 (m)
Si_3N_4–Y_2O_3		liquid-phase sintering	Si_3N_4	70–85	0.3–0.5
			Y_2O_3–SiO_2–Si_3N_4	15–30	—
	M1	electroslag melting		70–85	60–90
				15–30	30–60
	M2	electric-arc melting		70–80	30–50
			$(\alpha + \beta)$ Ti	20–30	60–130
Ti–Si	M3	electron-beam melting	Ti_5Si_3	65–75	30–50
			(Ti, Zr)$_5$Si$_3$	25–35	10–50
	M4	plasma-arc melting with stepwise treatment		70–85	20–40
				15–30	5–10
	M5			80–90	10–30
				10–20	5–50
WC–Co		solid-phase sintering	WC	85–95	2–5
			Co	5–15	—

Note: Characteristics of the main grain are in the numerator and characteristics of the other phases or inclusions are in the denominator. The symbols "t" and "m" refer to tetragonal and monoclinic phases, respectively.



(a)



(b)

Fig. 1. Dependences of the bending strength σ_u (a) and the fracture energy G (b) on the grain size d of alumina ceramics.

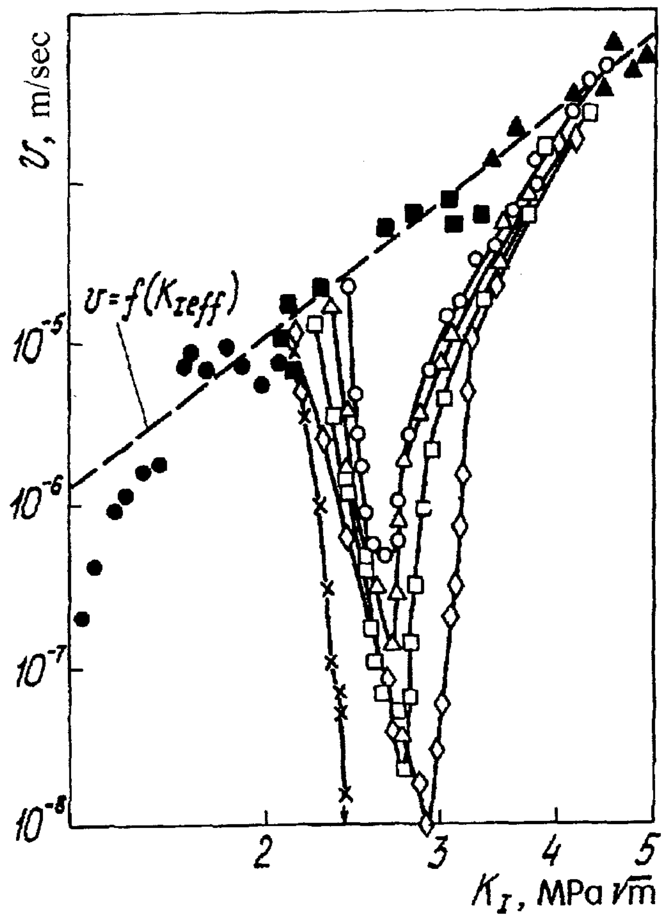
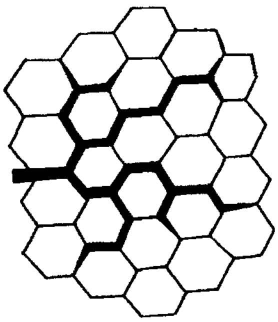
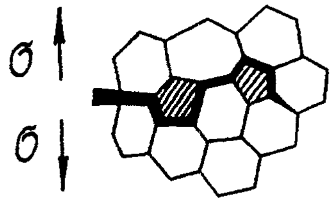
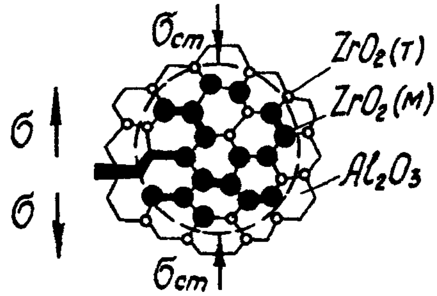
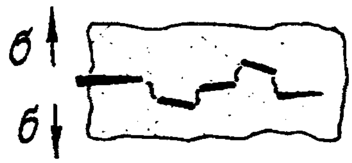
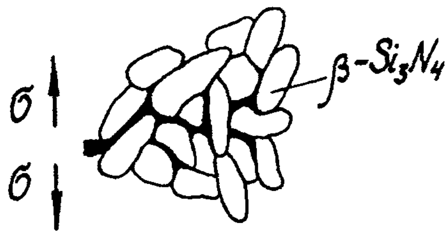
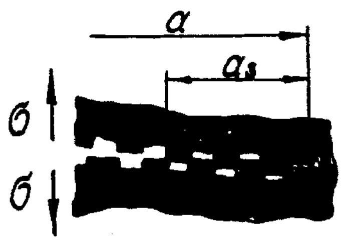
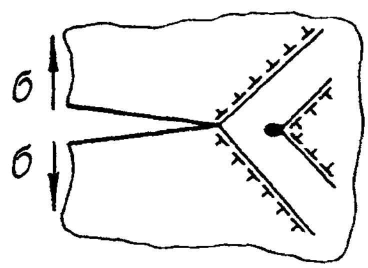


Fig. 2. Kinetic curves of fracture of alumina ceramics under mild (blank symbols) and severe (filled symbols) loading [8].

Table 2. Mechanisms of Stress Relaxation in the Process of Propagation of Cracks in Ceramic Materials

Material	Mechanisms of stress relaxation	
	dominant	secondary
Al_2O_3	1. Fan-like intergranular cracking 	5. By-pass of particles 
$Al_2O_3-ZrO_2$	2. Formation of a field of compressive stresses in the process of a polymorphic transformation 	6. Bridging of cracks 
$Si_3N_4-Y_2O_3$	3. Barrier action of elongated grains 	7. Effects of interaction (closure) of the crack lips 
Ti–Si	4. Dislocation mechanism 	
WC–Co		

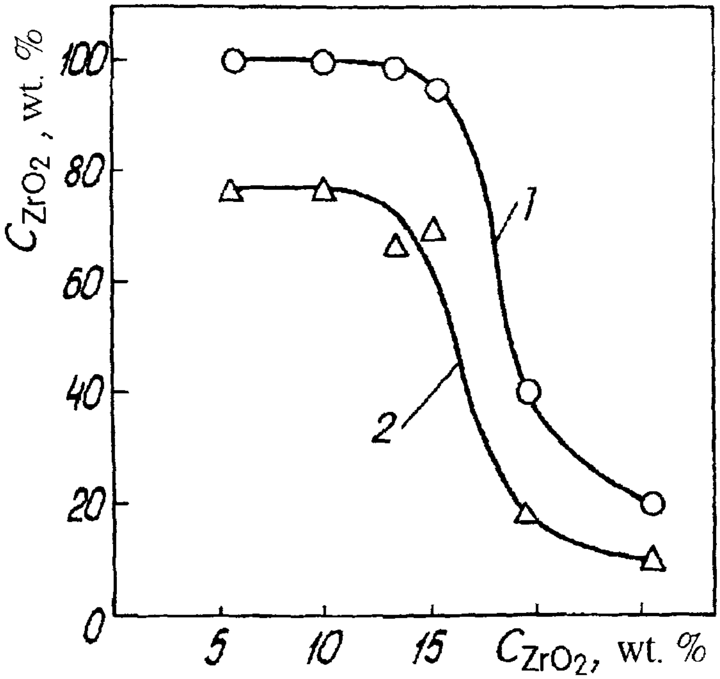


Fig. 3. Dependence of the content of the tetragonal phase of zirconium dioxide in the polished surface of a specimen (1) and in the fracture surface (2) on the total content of zirconium in Al_2O_3–ZrO_2 ceramics (in wt. %).

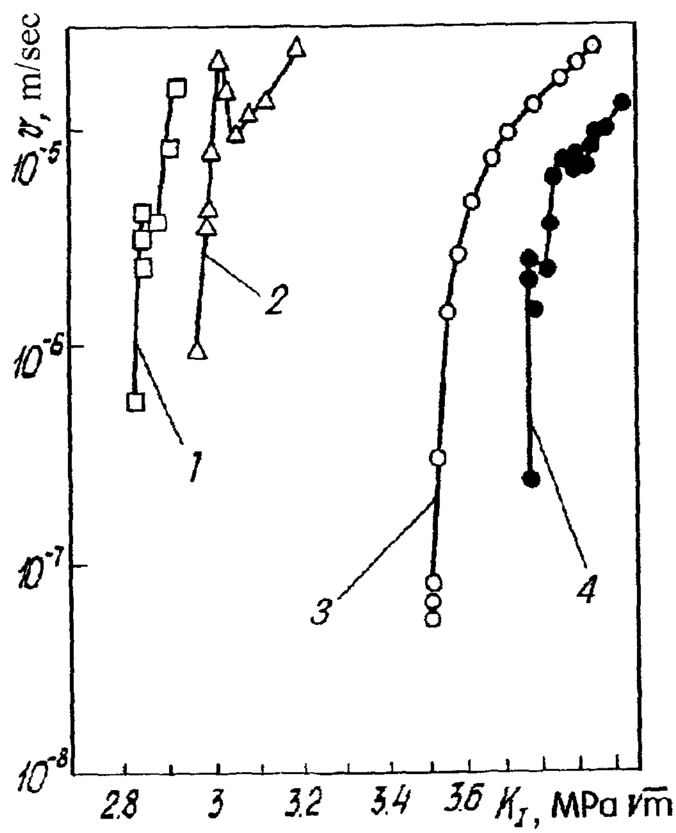


Fig. 4. Kinetic curves of fracture of Al_2O_3–ZrO_2 ceramics: (1) the sintering temperature of the ceramics is 1650° C, the sintering time is 0.5 h, the cooling rate is 600°C/h, (2) 1650/2/600, (3) 1700/2/600, and (4) 1700/2/1200.

3. Modified Nitride Ceramics

Technological experience shows that contemporary methods of sintering ceramics from highly disperse powders of silicon nitride and yttrium oxide ($Si_3N_4-Y_2O_3$) enable one to obtain a material with increased strength and crack resistance [16–18]. Sintering decreases the harmful porosity of yttrium oxide which forms the intergranular glass phase promoting the process of fan-like intergranular cracking. On the other hand, there is reason to believe that Y_2O_3 favors predominant growth of needle-shaped and elongated grains of β-Si_3N_4 near boundaries [6, 7]. These grains are randomly located and mainly play the role of barriers for a main crack, which results in additional expenditure of energy on its growth (scheme 3 in Table 2). This is an additional factor increasing the fracture toughness of modified silicon-nitride ceramics. Without a doubt, crack resistance, just like strength, strongly depends on the technology of preparation of the mixture for sintering [mechanical (MC) or plasma (PC) composition] and on the temperature-temporal mode of sintering (Table 3). According to our investigations, this mode can be optimized with respect to the Larsen–Miller parameter $P = T(20 + \log\tau)\cdot 10^{-4}$, where T is the sintering temperature (in degrees Celsius) and τ is the time of sintering (in hours) [4, 7].

4. Cermet Alloys (of the Ti–Si System)

It is reasonable to compare the mechanical properties of classical (traditional) types of ceramics and cermet alloys, which have higher contact serviceability with considerable gain in toughness, despite somewhat inferior surface characteristics. Work on a promising cermet is in the stage of search. For evaluations, we took alloys of the Ti–Si system modified with zirconium and aluminum developed by a research group in Dnepropetrovsk [19]. For experiments, we used five different types of these compositions (see Table 1). This cermet consists of a eutectic mixture of platelets of α- and β-titanium with extended boundary segregations of ceramic components of titanium and zirconium silicides [Ti_5Si_3 and $(Ti, Zr)_5Si_3$].

Ti–Si cermet, being significantly inferior to the above-mentioned materials in hardness, has significantly higher bending strength σ_u at room and elevated (700°C) temperatures and, in addition, an incomparably greater short-term crack-growth resistance K_{Ic} (Table 3). This is obviously caused by domination of the dislocation mechanism of energy dissipation in the course of fracture. The silicide skeleton additionally changes structure, which increases the bending-strength limit σ_u (scheme 4 in Table 2).

5. Cermet of the VK-Type (WC–Co)

Cermet of the VK-type, which is widely used in particular in cutting (including boring) tools, has quite high potentialities. As is known, intergranular interlayers in Ti–Si material have a solid-ceramic structure, and the morphology of grains is similar to the morphology of conventional alloys. But for a cermet of the VK -type, the situation is opposite. Namely, solid grains of WC tungsten carbide are connected by a relatively plastic cobalt binder. It is no coincidence that this material is superior to Ti–Si cermet in HV hardness and strength σ_u even at a high service temperature (Table 3). Evaluations of crack resistance also give significantly enhanced fracture toughness K_{Ic} at both room and high (700°C) temperatures. In the course of testing for fracture toughness K_{Ic}, fracture proceeds mainly through grains.

Comparative Evaluation of the Structural Strength of Ceramic Materials

Relations between the characteristics of strength and crack resistance of ceramic materials are used to plot diagrams of their structural strength. Taking into account the functional purpose, it makes sense to plot structural-strength diagrams of ceramic materials with regard for their surface serviceability and behavior at elevated temperatures.

Table 3. Mechanical Characteristics of the Materials Studied at Normal and High (700°C) Testing Temperatures

Materials		Sintering mode	K_{Ic}, MPa $\sqrt{m}$	σ_u, MPa	K_{Ic}^{700}, MPa $\sqrt{m}$	σ_u^{700}, MPa	HV, GPa
Al_2O_3		1	3.4	300	3.5	320	16.5
		2	2.9	350	3.0	360	18.0
Al_2O_3–ZrO_2		1	4.8	600	4.9	620	18.0
		2	4.4	520	4.5	530	16.0
		3	3.5	450	3.5	470	11.0
		4	3.0	350	3.1	360	5.0
Si_3N_4–Y_2O_3	MC	1	6.0	340	6.1	340	18.4
		2	5.4	330	5.5	330	19.1
		3	5.1	350	5.1	360	17.3
		4	4.9	260	4.9	250	16.0
		5	3.2	220	3.2	240	5.7
		6	3.1	200	3.0	210	9.4
	PC	1	4.9	310	5.0	315	18.7
		2	4.8	250	4.9	240	18.7
		3	4.6	250	4.7	250	18.1
		4	4.3	330	4.3	340	17.8
		5	3.8	240	3.8	230	15.7
		6	3.6	210	3.7	210	14.1
Ti–Si		M1	18.3	1027	21.9	720	4.25
		M2	16.9	817	20.7	650	5.2
		M3	16.0	815	17.0	650	5.45
		M4	20.6	1410	26.7	678	5.0
		M5	18.0	1250	22.6	690	4.75
WC–Co		1	22.5	1800	19.0	1400	11.0
		2	18.0	1850	15.1	1500	14.0
		3	15.0	1950	14.5	1700	15.5
		4	11.5	1900	10.4	1600	17.0

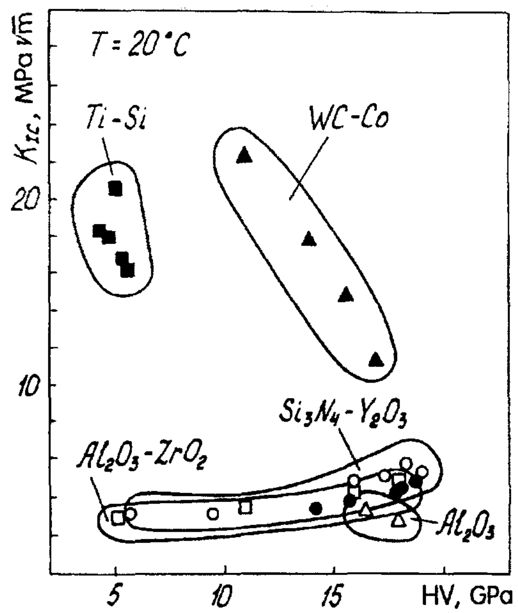


Fig. 5. Structural-strength curves (hardness HV vs fracture toughness K_{Ic}) of ceramic and cermet materials.

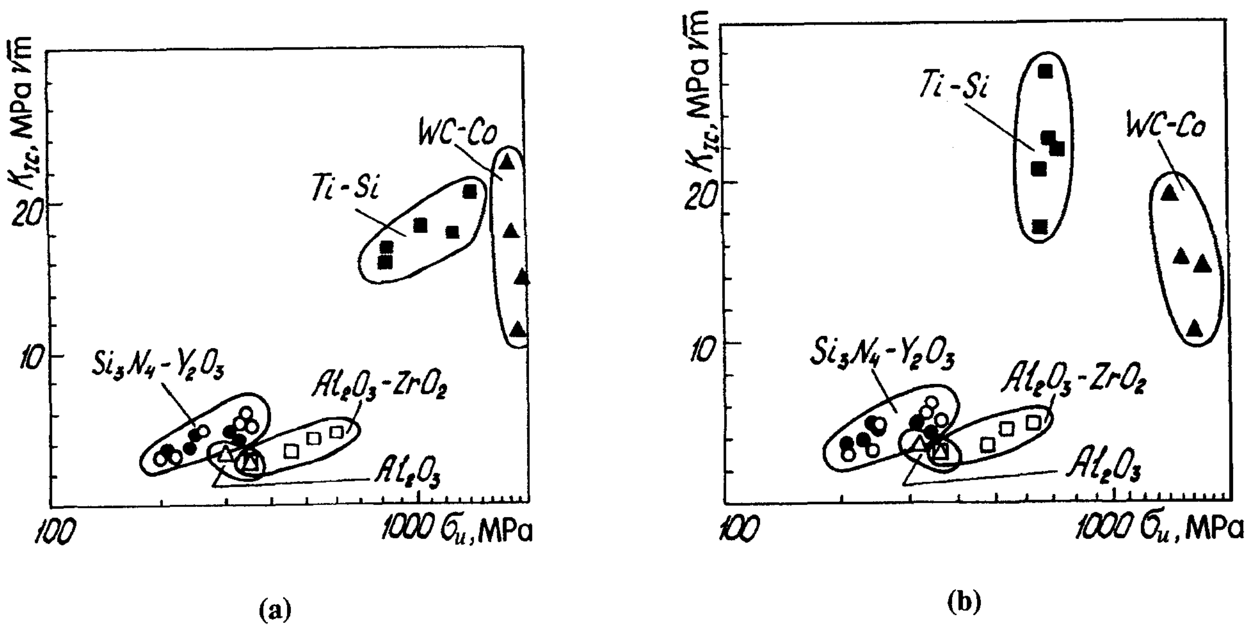
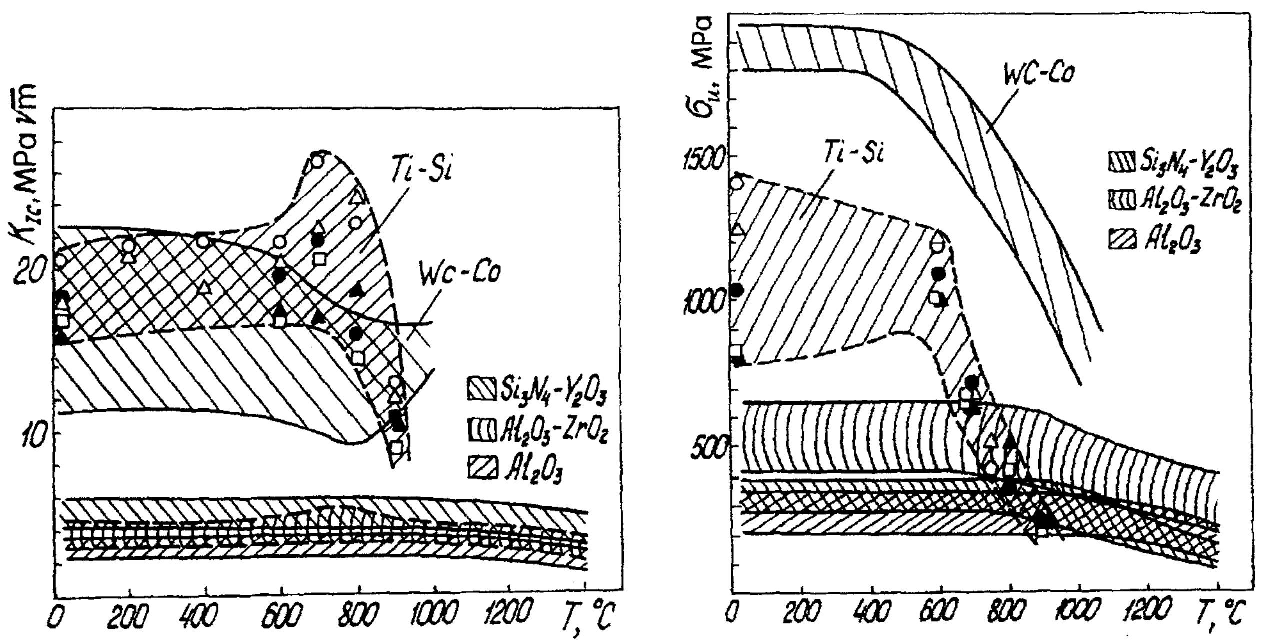


Fig. 6. Structural-strength curves of ceramic and cermet materials at room temperature (a) and 700°C (b).

As follows from the curves in Fig. 5, for conventional Al_2O_3, Al_2O_3–ZrO_2, or Si_3N_4–Y_2O_3 ceramics, at a hardness of up to 20 GPa, the quantity K_{Ic} can reach a value of about 5–6 MPa · $\sqrt{m}$. The toughness of Ti–Si cermet is much higher (up to 20 MPa · $\sqrt{m}$) but its surface strength is low. Tungsten-cobalt compositions of the VK-type have considerable advantages because, being at a slight disadvantage in relation to classical ceramics in hardness, they are significantly superior to all other tested materials in the level of K_{Ic}. A similar conclusion can be made on the basis of structural-strength diagrams of the ordinary type [20], comparing K_{Ic} with σ_u (Fig. 6a). At the same time these characteristics of Ti–Si cermets approach the characteristics of tungsten-cobalt compositions, while the aluminum-zirconium ceramics has better parameters than silicon-nitride ceramics. An increase in the testing temperature to 700°C has no fundamental effect on the number of indices of structural strength, although it significantly decreases the strength of cermet. Nevertheless, its advantages over classical ceramics hold and can be used at high temperatures (Figs. 6b and 7).



(a) (b)

Fig. 7. Temperature dependences of the fracture toughness K_{Ic} (a) and the bending strength σ_u (b) of ceramic and cermet materials.

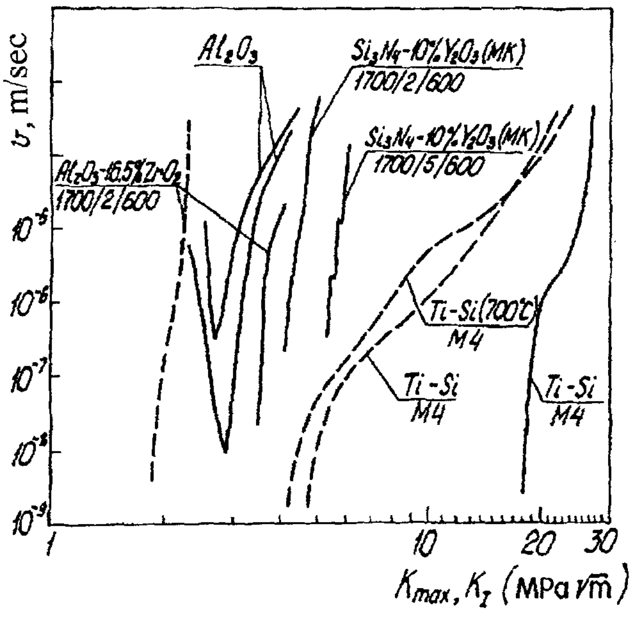


Fig. 8. Kinetic curves of fracture of ceramic and cermet materials under static (solid curves) and cyclic (dashed curves) loading. The numbers below the curves correspond to the sintering temperature (in degrees Celsius), sintering time (in hours), and cooling rate (in °C/h). The curves for the M4 modification of Ti–Si cermet are recorded at room and high (700°C) temperatures.

Subcritical Crack Resistance of Ceramic Materials

A dangerous feature of traditional brittle materials is their inability to ensure a considerable service life under conditions of subcritical crack growth under long-term loading. For this reason, testing and comparison of ceramic materials in the range of subcritical propagation of cracks are important for service reliability.

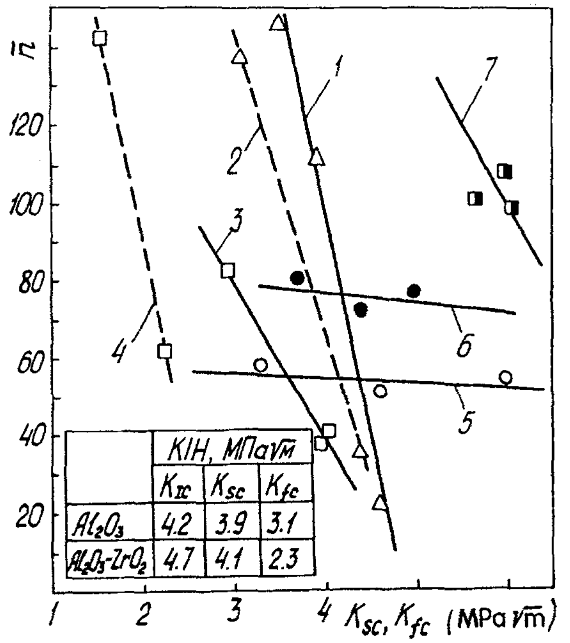


Fig. 9. Dependence of the angular coefficient $\bar{n}$ on the critical stress intensity factors under static long-term K_{sc} (solid curves) and cyclic K_{fc} (dashed curves) loading: (1) and (2) Al_2O_3, (3) and (4) Al_2O_3–ZrO_2, (5) Si_3N_4–Y_2O_3 (mechanical composition), (6) Si_3N_4–Y_2O_3 (plasma composition), and (7) ZrO_2–Y_2O_3.

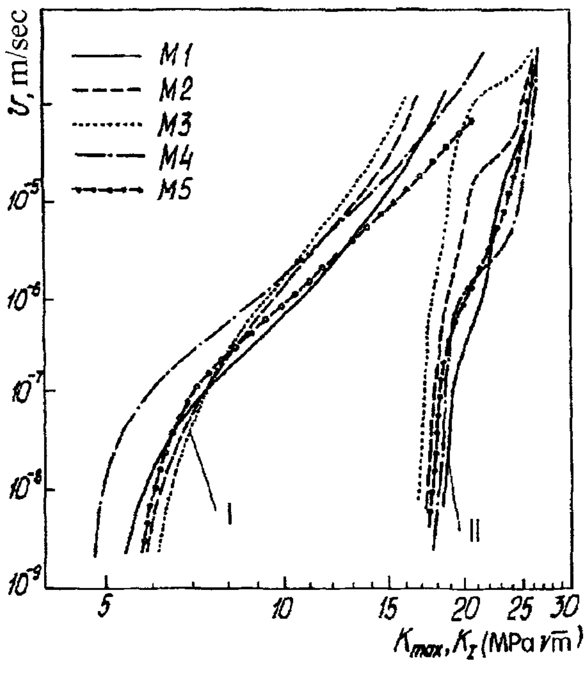


Fig. 10. Kinetic curves of fracture for Ti–Si cermet under cyclic sinusoidal (I) and static (II) loading: M1–M5 are the investigated materials.

Our investigations show that, in classical ceramics of Al_2O_3, Al_2O_3–ZrO_2, and Si_3N_4–Y_2O_3 types, the description of creation of fatigue cracks is an extremely difficult problem that cannot be solved by ordinary methods [21, 22]. Loading by eccentric tension for which the rate of growth of an initial crack may stabilize seems to be promising in this case [15]. Due to a lack of specimens of required configuration and adequate loading equipment, we evaluate subcritical crack growth under conditions of bending of beam specimens with a sharp lateral notches.

To compare various ceramic materials under long-term static and cyclic loading, we plotted dependences of the crack growth rate on the value of K_{max} (Fig. 8). For classical types of ceramics, we determined the crack growth rate by using the variation in the pliability of the specimens, whereas the motion of the crack tip was directly observed for cermet.

It is worth noting that the kinetic curves of static and fatigue fracture are quite steep for all investigated types of ceramics (alumina, aluminum-zirconium, and silicon-nitride), which hinders separation of three stages on the kinetic curves. For this reason, in comparative analysis of the serviceability of the materials, we use the angular coefficient of the kinetic curves

$$\bar{n} = \frac{\Delta \log v}{\Delta \log K}.$$

Another characteristic of resistance is the value of K_{max} for which spontaneous fracture occurs, namely, K_{sc} and K_{fc} under static and cyclic loading, respectively. Classical ceramic materials largely differ from one another more by K_f than by the coefficient $\bar{n}$ (Fig. 8) despite the inverse proportionality of the parameters $\bar{n}$ and K_f. This is indicated by analysis of our data and those in the literature (Fig. 9). It is significant that the values of K_{sc} and K_{fc} considerably differ for ceramics, namely, K_{sc} is close to K_{Ic}, while K_{fc} is much lower. This is caused, perhaps, by different mechanisms of critical damage in the process of spontaneous fracture rather than by measurement error. Obviously, there exists a similarity between the phenomena of critical coalescence of damage volumes under the conditions of fatigue of ceramics and high-strength steels [23]. Since evaluation of these volumes is an extremely complicated problem, the size of a subcritical crack obtained by the pliability method is considerably underestimated and, hence, the value of K_{fc} is also smaller.

Kinetic curves of cermets differ from typical kinetic curves for ordinary ceramics and more resemble the kinetic curves of conventional metallic materials (Figs. 8 and 10). The kinetic curve of fatigue fracture for Ti–Si cermet consists of three classical sections, and the values of K_{fc} and K_{sc} have a tendency to approach each other.

Mechanisms of Stress Relaxation in Ceramic Materials and the Structural Aspect of the Problem

On the basis of the performed analysis, we can distinguish a number of mechanisms of stress relaxation that form the crack resistance of ceramic materials. For a model simple ceramics of the Al_2O_3 type, fan-like cracking is the main mechanism of stress relaxation. Under conditions of subcritical crack growth, this mechanism can lead to retardation of the growth (see Table 2). The transition of the tetragonal phase to the monoclinic one leads to compressive stresses that cause unloading of crack tips in ceramics of the type Al_2O_3–ZrO_2 (see Table 2). In nitride silicon ceramics, transversely situated elongated grains of the β-phase act as barriers. In cermets, the dislocation mechanism of stress relaxation (typical of ordinary metallic alloys) is strongly manifested.

In addition to the predominant mechanisms, one should also remember the others (see Table 2) that were discussed in the course of evaluation of the crack resistance of ceramics, whose influence on fracture toughness is additive [24]. Among them, the barrier action of particles that are by-passed by a crack and bridging effects are important. The interaction of crack lips in ceramic materials is of special importance. Phenomena of crack closure and their influence on crack resistance in ceramics are much more complicated than in metallic alloys [25]. In particular, there is reason to believe that crack closure in ceramics considerably depends on the length of the crack and also manifests itself under monotonic (noncyclic) loading. However, this requires further analysis.

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