

DYNAMIC PROPERTIES OF ZIRCONIUM DIOXIDE REFRACTORIES

A. M. Bragov,¹ A. K. Lomunov,¹
V. N. Mineev,² F. A. Akopov,² and G. P. Chernyshov²

UDC 539.3

The results of static and dynamic tests for three groups of ceramic materials based on zirconium dioxide ZrO_2 and for zirconium dioxide concrete are reported. The static and dynamic tests were performed according to the conventional procedure and Kolsky procedure, respectively. The ceramic samples were obtained using different manufacturing technologies and had different initial densities and porosities. In the experiments, static and dynamic strain curves were obtained and strength characteristics were determined. The dynamic strength properties of ceramics and concrete are shown to depend on the stress growth rate. The dynamic curves of ceramics are compared with those of structural and zirconium dioxide concretes.

Introduction. The problem of melt confinement in the active zone is topical for all states that use atomic power stations and experimental nuclear reactors. In middle 1980s, numerous experiments were performed on the interaction between the active zone melt and structural concrete used in mine protection systems. These experiments showed that the existing protection system based on structural concretes is not efficient enough.

It is assumed that the trap material experiences various factors, namely, mechanical and thermal shocks arising when the molten mass and constructional components fall into the trap, steam explosions occurring due to melt–water contact, chemical substances, and radiation.

Preliminary experiments showed that the use of zirconium dioxide ceramics in constructing traps makes it possible to improve significantly their reliability and, thus, solve the problem of safety of nuclear power stations [1].

In operation of a trap may occur, emergency conditions, such as steam explosions accompanied by intense dynamic actions; therefore, the study of the dynamic strength of zirconium dioxide ceramics and concretes under rapid loading becomes a topical problem.

Test Methods. The samples were tested at static (about 10^{-3} sec⁻¹) and dynamic (about 10^3 sec⁻¹) strain rates. In static tests, a UIM-30 hydraulic testing machine with a rated load capacity of 30 tons and a 1958U-10-1 testing machine with a thyristor drive were used.

The Kolsky method that uses the split Hopkinson pressure bar is one of the most developed and widespread procedures for dynamic testing of materials at strain rates of 10^3 sec⁻¹ [2]. This method was used in the present work to study the dynamic curves of ceramics.

Samples Tested. Three groups of zirconium dioxide ceramic samples differing in their grain fractional compositions and also zirconium alumina concrete samples (ZAC) were studied. The fractional compositions of the ceramic samples (α is the fraction size) are listed in Table 1. The ceramic and ZAC samples were prepared from electrically melted zirconium dioxide stabilized with yttrium oxide Y_2O_3 (87% ZrO_2 + 10% Y_2O_3). Barium aluminate (mass fraction 6%) was used as a binder agent for ZAC. Some physicomechanical characteristics of zirconium ceramics and ZAC samples at room temperature are listed in Table 2 (ρ is the density, η is the porosity, and σ_{st} is the ultimate strength in compression).

¹Institute of Mechanics at the State University, Nizhnii Novgorod 603600. ²Research Center for Thermal Physics of Pulsed Treatments, Joint Institute of High Temperatures, Russian Academy of Sciences, Moscow 127412. Translated from *Prikladnaya Mekhanika i Tekhnicheskaya Fizika*, Vol. 42, No. 3, pp. 168–174, May–June, 2001. Original article submitted April 19, 2000; revision submitted July 20, 2000.

TABLE 1

Mass Distribution of Size Fractions in Ceramic Samples (in percent)

Group of samples	α , mm						
	<0.05	0,1–0.2	0.2–0.315	0.315–0.4	0.4–0.63	0.63–1	1–2
1	40	—	20	—	—	40	—
2	25	—	25	20	—	—	30
3	15*	30	—	—	—	—	55

Note. Asterisk denotes the tetragonal phase; all other fractions have a cubic structure stabilized by yttrium oxide Y_2O_3 (molar fraction 10–11%).

TABLE 2

Physicomechanical Characteristics of Ceramic and ZAC Samples

Material	ρ , g/cm ³	η , %	σ_{st} , MPa
Ceramics:			
group 1	4.7–4.9	20	39
group 2	4.82	17	45
group 3	4.56	16	68
ZAC	4.66	13–16	35

For static tests, samples of two sizes were prepared: 20 mm in diameter and height and 30 mm in diameter and height. The choice of different geometric sizes of the samples was motivated by the desire to examine the effect of the scale factor on the strength and strain characteristics of the ceramics.

For dynamic tests, ceramic samples in the form of tablets 20 mm in diameter and 10 mm thick were prepared. To minimize the detrimental influence of inertia and friction, the geometry of the samples was chosen to meet the Davies–Hunter criterion [3]. The zirconium dioxide concrete samples were prepared in the form of 5 mm-thick disks 50 mm in diameter. Prior to tests, the samples were manually polished using a sandpaper with a grain size of 0.01 mm. To diminish friction and improve the acoustic contact, two sheets of thin (10 μ m) fluoroplastic film were placed between the ends of the pressure bars and the sample.

Results of Static Tests. The static tests of ceramic samples of group 2 were performed according to a procedure specified by GOST (National Standard) 10180-90: “Concretes. Procedures for Determining Their Strength Characteristics Using Test Samples.” In the first test series, the strength in compression was determined and static strain curves were obtained. In the second one, tests in tension were performed during which the samples were cleaved. For each loading scenario, four to six tests were made, with subsequent averaging of the results obtained.

It follows from the data obtained that the failure stresses differ appreciably from each other in compression (38–40 MPa) and tension (7–10 MPa), i.e., the strength properties of ceramics are anisotropic. The compression and tension strengths of the samples turned out to be only weakly dependent on their sizes. Thus, for the ceramic of group 2 with the largest grain size smaller than 2 mm (see Table 1), the scale factor is only weakly manifested at static strain rates.

The strain curve in compression is shown in Fig. 1. Curves 1 and 2 correspond to ordinary compression and cyclic deformation of the sample, respectively. The sample was first loaded to a certain stress (below the ultimate strength), then fully unloaded and, finally, loaded again. The data show that the process of active loading is nonlinear. The unloading branch of the strain curve is also nonlinear. The loading and unloading branches of the strain curves differ appreciably. At the initial part of the unloading branch, the slope of the curve is greater than that under loading. The additional loading branch for the unloaded sample behaves very similar to the initial loading branch. The area under the descending part of the strain curve may be interpreted as the specific energy of material failure.

Results of Dynamic Tests and Their Discussion. Compression tests of three groups of ceramic samples and ZAC samples were performed on a setup described in [4], using the Kolsky procedure. Pressure bars 1 m long and 20 mm in diameter were prepared from the D16T alloy. The sample to be tested was placed between the ends

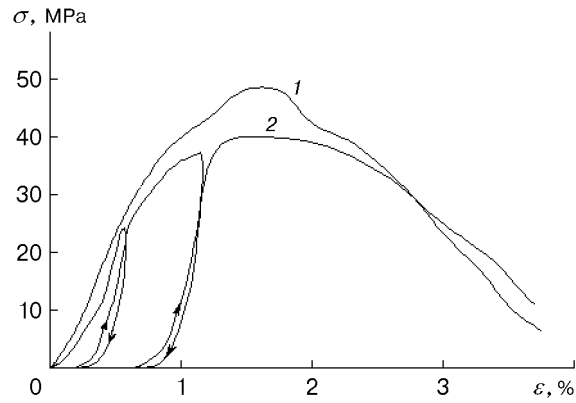


Fig. 1

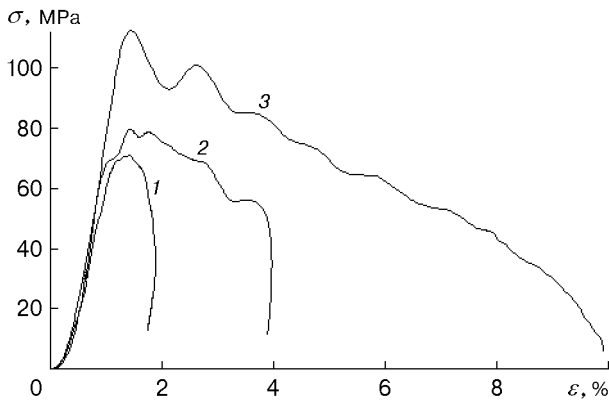


Fig. 2

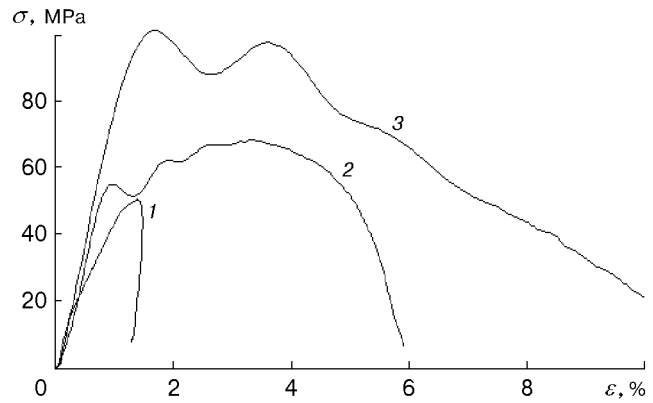


Fig. 3

of the bars. To load the samples, a gas gun 20 mm in diameter was used. During the tests, by adjusting the striker velocity, i.e., the amplitude of the incident wave, loading conditions were found under which the sample either preserved its visual appearance or suffered failure.

The results for groups 1–3 of ceramic samples are shown in Figs. 2–4, respectively. For each group, typical dynamic strain curves are shown. These curves were obtained by averaging data obtained in several experiments performed under identical test conditions. Curves 1 correspond to conditions under which the samples after the tests remained undamaged, curves 2 correspond to conditions under which the samples either displayed some minor (mainly, peripheral) cracks or, remaining intact in appearance, lost their internal integrity, and curves 3 corresponds to conditions of complete failure of the samples.

From the strain curves, the mean moduli of loading branches E_{load} were found, and also the unloading moduli E_{unload} for the cases in which the samples were not destroyed. In addition, the time resistance (ultimate strength) in compression σ_t and the corresponding strain ε_t , and also the time of failure beginning t_t and the corresponding energy capacity W (the area under the σ – ε curve) were determined. The values of these parameters for each curve of Figs. 2–4 are listed in Table 3.

The unloading-branch moduli of the strain curves are 2 to 3 times greater than the loading-branch moduli. It is seen that the ceramic samples of group 3 display a better strength and a lower deformability as compared to groups 1 and 2.

The test results for ZAC samples are shown in Fig. 5 and listed in Table 3. Curve 1 corresponds to the conditions under which the samples remained undamaged, and curves 2–4 to the conditions of failure of the samples. In this case, the first stress maximum corresponds to the onset of concrete failure. However, unlike the curves for ceramics, after attaining the first maximum in the descending branch of the strain curve, a small incremental increase in the strain occurs; afterwards, either further growth of stress is observed (curves 2 and 3) or the stress remains almost unchanged. This behavior was due to the fact that the samples in the form of a disk plate 50 mm

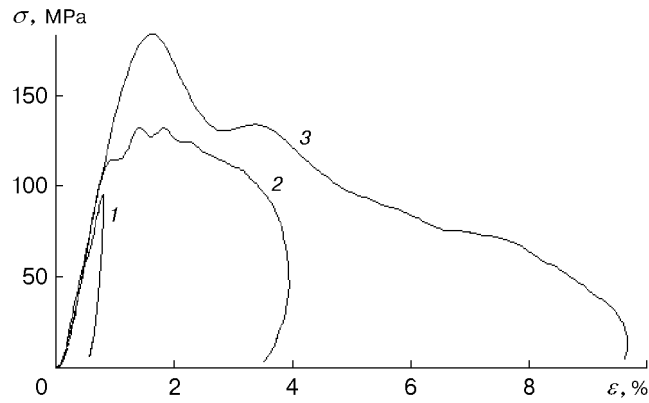


Fig. 4

TABLE 3

Strength Characteristics of Ceramic and ZAC Samples

Material	Curve No. (Figs. 2-5)	E_{load} , MPa	E_{unload} , MPa	$\dot{\epsilon}$, sec^{-1}	$\dot{\sigma}$, $\text{MPa}/\mu\text{sec}$	σ_t , MPa	ϵ_t , %	t_t , μsec	W , MJ/m^3
Ceramics (group 1)	1	7700	13,200	180	2.4	71	1.5	85	0.459
	2	12,300	24,800	400	6.4	80	1.5	43	0.553
	3	10,700	—	1000	10.0	113	1.4	31	0.698
Ceramics (group 2)	1	7700	21,900	150	1.8	48	1.33	109	0.360
	2	7800	—	700	5.2	68	3.37	64	1.700
	3	9000	—	1600	9.5	101	1.60	25	0.974
Ceramics (group 3)	1	14,000	31,000	150	4.6	97	0.78	80	0.334
	2	17,000	20,000	400	9.5	132	1.49	44	1.090
	3	15,500	—	1400	16.7	183	1.55	24	1.656
ZAC	1	10,100	10,300	200	2.4	51	1.98	102	0.754
	2	14,000	23,000	400	4.2	61	0.91	34	0.388
	3	12,100	19,500	900	11.3	129	1.41	29	1.018
	4	12,000	18,000	1600	17.0	206	2.78	24	3.727

in diameter were located between the end faces of the pressure bars 20 mm in diameter. The unloaded parts of the sample plate acted as a restricting yoke that prevented radial deformation of the central zone of the sample which experienced deformation and, on failure, prevented sample fragments from falling out of this zone. Since the duration of the incident pulse in the tests was sufficiently long, the process of deformation was accompanied by compacting of fragments and by an increase in the resistance to deformation.

Consideration of experimental data shows that the stress in the sample after attaining the first strain maximum in the bars decreases, and the strain rate slightly increases. Afterwards, some short-time stabilization of the deformation process is observed, and then (approximately after 50 μsec) the stress in the sample starts increasing again and the strain rate decreases.

As strength characteristics of ceramics and ZAC in compression, we choose the maximum values of the stress and corresponding strain in the strain curves obtained in those tests in which the samples retained their integrity. However, the dynamic loading studies revealed higher ultimate strengths of the materials, which is evidenced by the data in Figs. 2-5. This behavior of the materials is caused by the dynamic character of loading and determined by two competitive processes that occur in the sample: on the one hand, by the formation and growth of microcracks and micropores and their merging into macropores and cracks and, on the other hand, by the wavy character of increasing loading in the material. If the stress growth rate is greater than the intensity of the failure process, then the sample with already formed and developing damage sites may be "overloaded," i.e., can withstand increasing loads for a certain period. Similar phenomena were discussed in [5] in analyzing the time evolution of strength during cleavage.

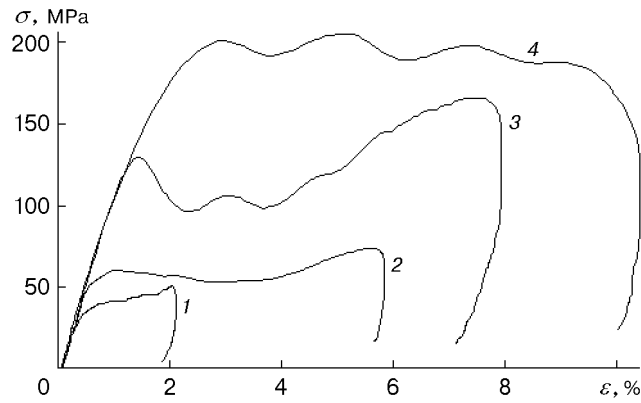


Fig. 5

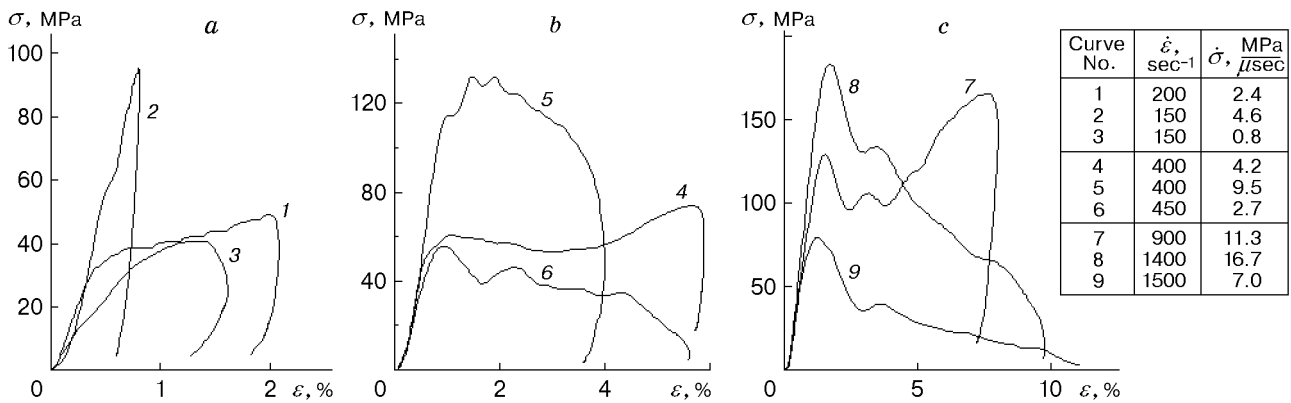


Fig. 6

In treating experimental data obtained in each test, apart from the mean strain rates $\dot{\epsilon}$, we also determined the highest stress growth rates in the sample $\dot{\sigma}$ (Table 3). It should be noted that, as the stress growth rate increases, the failure stress also increases, and this dependence may be approximated by a linear function.

Figure 6 shows the dynamic curves for ceramic samples of group 3 (curves 2, 5, and 8), ZAC samples (curves 1, 4, and 7), and structural microconcrete (curves 3, 6, and 9). The microconcrete was prepared from M400 cement and sand taken in the mass ratio 1:2 (the water-to-cement mass ratio was 1:2). The grain size of sand particles was smaller than 1 mm. For each material, three representative curves are shown for conditions under which the samples retained their integrity (Fig. 6a), were slightly damaged (Fig. 6b), and totally disintegrated (Fig. 6c). The table in Fig. 6 shows the mean strain rates $\dot{\epsilon}$ and the mean stress growth rates $\dot{\sigma}$. The unloading moduli of all the materials studied are greater than the corresponding moduli for the loading branches of the strain curves. The ceramics has a better strength and a lower deformability compared to ZAC and ordinary concrete.

Conclusions. Dynamic tests were performed and dynamic strain curves were obtained for three groups of ceramic samples prepared from zirconium dioxide and differing in their initial physical and mechanical properties, and also for zirconium dioxide concrete samples. The stress growth rate has an appreciable effect on the strength characteristics of the samples. The ceramics of group 3 has the highest strength and the lowest deformability. Ceramics is comparatively inert, and its melting point is about 3000 K. The heat conductivity in materials based on zirconium dioxide is 1.5 to 2 times lower than that of other refractory oxides. Thus, the zirconium dioxide ceramics seems to be a material most suitable for designing traps. The use of this material in constructing traps makes it possible to substantially improve reliability of the latter and, thus, to solve the problem of ensuring safety of nuclear power stations.

The authors thank L. N. Kramarev for performing static tests and A. S. Vlasov and S. S. Lukin for their assistance in measuring the initial physical and mechanical characteristics of the materials.

This work was supported by the International Science and Technology Center (Grant No. 425) within the framework of the Program for Supporting Leading Scientific Schools in Russia (Project No. 00-15-99029).

REFERENCES

1. V. N. Mineev, L. B. Borovkova, F. A. Akopov, et al., "External trap made of zirconium dioxide ceramics and concrete," *At. Énerg.*, **85**, 119–125 (1998).
2. G. Kolsky, "Mechanical studies of materials at high loading rates," *Mekhanika*, **4**, 108–119 (1950).
3. E. D. H. Davies and S. C. Hunter, "The dynamic compression testing of solids by the method of the split Hopkinson pressure bar," *J. Mech. Phys. Solids*, **11**, 155–179 (1963).
4. A. M. Bragov and A. K. Lomunov, "Methodological aspects of studying dynamic material properties using the Kolsky method," *Int. J. Impact Eng.*, **16**, No. 2, 321–330 (1995).
5. N. A. Zlatin, G. S. Pugachev, and V. A. Stepanov, "Failure stresses caused by a short-time impact," *Zh. Tekh. Fiz.*, **49**, No. 8, 1786–1788 (1979).