Damage Formation, Fatigue Behavior and Strength Properties of ZrO_2-Based Ceramics

A. A. Kozulin[1,a)], A. S. Narikovich[2], S. N. Kulkov[1,3,b)], V. N. Leitsin[2], and S. S. Kulkov[1]

[1] *Tomsk State University, Tomsk, 634050 Russia*
[2] *Immanuel Kant Baltic Federal University, Kaliningrad, 236041 Russia*
[3] *Institute of Strength Physics and Materials Science SB RAS, Tomsk, 634055 Russia*

a) Corresponding author: kozulyn@ftf.tsu.ru
b) kulkov@ispms.ru

Abstract. It is suggested that a non-destructive testing technique using a three-dimensional X-ray tomography be applied to detecting internal structural defects and monitoring damage formation in a ceramic composite structure subjected to a bending load. Three-point bending tests are used to investigate the fatigue behavior and mechanical and physical properties of medical-grade ZrO_2-based ceramics. The bending strength and flexural modulus are derived under static conditions at a loading rate of 2 mm/min. The fatigue strength and fatigue limit under dynamic loading are investigated at a frequency of 10 Hz in three stress ranges: 0.91–0.98, 0.8–0.83, and 0.73–0.77 MPa of the static bending strength. The average values of the bending strength and flexural modulus of sintered specimens are 43 MPa and 22 GPa, respectively. The mechanical properties of the ceramics are found to be similar to those of bone tissues. The testing results lead us to conclude that the fatigue limit obtained from 10^5 stress cycles is in the range 33–34 MPa, i.e. it accounts for about 75% of the static bending strength for the test material.

Keywords: porous ceramics, three-point bending test, fatigue strength, bending strength, flexural modulus, X-ray tomography

INTRODUCTION

Recent years have seen wide diversity of manufacturing techniques for structural oxide ceramics. This raises the question as to what materials of this class produced by what technologies are most suitable for particular applications in vital structural members and critical parts. The high degrees of biological compatibility and chemical inertness allow for using the ceramics in medical engineering applications (for example, as osteoreplacing materials) [1–4]. Among the variety of structural ceramic materials, investigations of partially stabilized zirconia-based ceramics are of prime interest due to a comprehensive impact of porosity, grain structure, and phase composition on their mechanical behavior. It is known that different molding forces and sintering temperatures provide materials with different porosities [4]. The foregoing parameters affect the physical and mechanical properties of the oxide ceramics, ranging from low performance characteristics, as in bone tissues, to record high characteristics, as in structural steels and alloys. Nowadays a large body of research is devoted to the relation between internal structure of zirconia-based ceramics and their mechanical properties, like compressive strength, bending strength, fracture toughness, and microhardness [2–5]. However, little is known about the fatigue behavior of porous ceramic materials [6, 7]. That is why studying the fatigue behavior of this type of ceramics and establishing the association of this characteristic and internal structure of the material is one of the topical areas in current research because of the need for long-term operation and trouble-free service of ceramic products employed in a variable load environment.

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It is the objective of this work to investigate the internal structure and nature of microdamage of porous zirconia-based ceramics and to evaluate the strength characteristics and fatigue life of the ceramic specimens subjected to three-point bending tests.

MATERIALS AND METHODS

Prismatic specimens with a final size of 5 mm in width, 6 mm in height, and 40 mm in length were produced for experiments. The source material was a fine-crystalline powder of the MgO-partially stabilized ZrO_2 system made up of hollow spherical particles of average size 0.5–2 μm and crystallites of size 20–30 nm [4]. The powder was compacted in molds, and the resulting green pellets were subjected to high-temperature sintering at $T = 1600°C$ for 1 h.

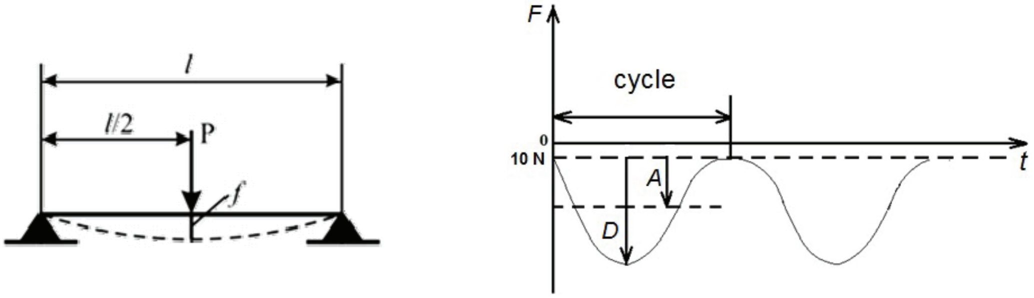
The physical and mechanical properties of the specimens were investigated by the three-point bending technique (see the scheme in Fig. 1a), using an Instron ElectroPulse E1000 electrodynamic testing machine. The span between two supports of the specimen holder was 30 mm. The supports and the loading piston were configured as knives with edges rounded off to a radius of 2.5 mm. Load was applied at the midpoint of the specimen with a rate of 0.2 mm/min. Bending strength σ_b and flexural modulus E_b of the specimen material undergoing static three-point bending were determined as in [2, 8] by the following formulas:

$$\sigma_b = \frac{3Pl}{2bh^2}, \quad E_b = \frac{Pl^3}{4fbh^3},$$

where P is the ultimate load, N is found in testing the specimens, l is the support span, mm, b is the specimen width, mm, h is the specimen height in the midspan, mm, and f is the specimen deflection, mm.

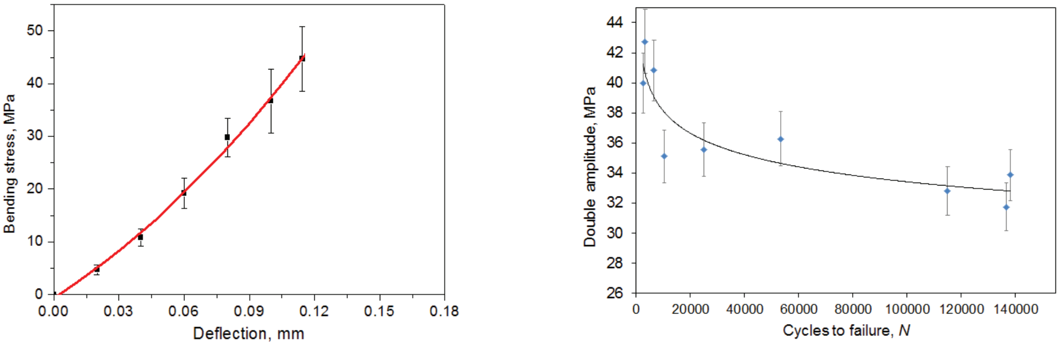
To measure the fatigue life, loading conditions were assigned in terms of unipolar loading waves from 10 N with double amplitude D of the cycle corresponding to three stress ranges: 0.91–0.98, 0.8–0.83, and 0.73–0.77 MPa of the static bending strength at a frequency of $\nu = 10$ Hz (see the scheme in Fig. 1b). The parameters of the fatigue tests were chosen in such a way as to provide the required stiffness of the testing system with reasonable accuracy in the fulfilment of the specified conditions in the loading cycle.

Using 3D X-ray tomography by means of a *Y*. Cheetah inspection system provided by YXLON Company, we have performed non-destructive testing of the ceramic materials for internal structural defects, porosity, voids, and microcracks in the bulk of the test specimens. A fine-focus open-type X-ray tube equipped with a continuous air exhaust unit—a high-power bremsstrahlung source (up to 15 W at the target) with a voltage of 25–160 kV and a current of 1 mA—was located in the system used. An X-ray beam focusing unit inside the tube formed a 1–2 μm focal spot at an anode target of the transmission type, which allowed for high-contrast imaging of the object with micron resolution. Given an adequate choice of scanning regimes, the proposed investigation technique made it possible to conduct a high-accuracy structural examination of the materials. The same scanning conditions were used for all test specimens: voltage of 110 kV, current of 70 μA, 12× magnification, and 4 projections per degree of specimen rotation around the longitudinal axis with an exposure time of 2 s.



(a) (b)

FIGURE 1. Three-point bending test (a) and unipolar cyclic loading schemes (b), with *A* and *D* denoting the load amplitude and double amplitude of the loading cycle *N*, respectively



(a) (b)

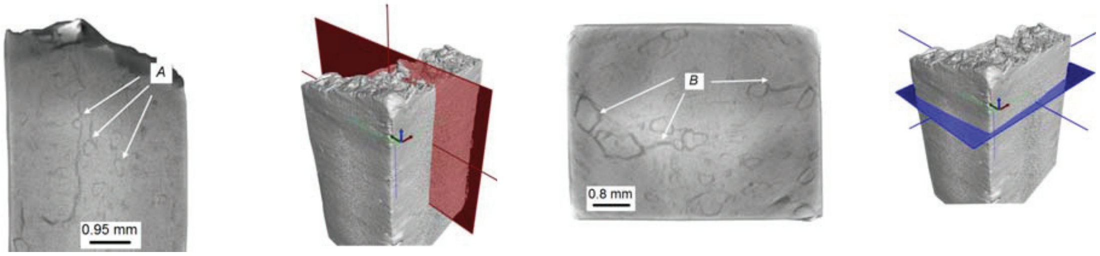
FIGURE 2. Experimental relationship between bending stress and specimen deflection (a) and fatigue bending test results (b) for specimens made from the zirconia ceramics under study

RESULTS AND DISCUSSION

The average ultimate bending strength of the test material was calculated to be σ_b = 43 MPa for a maximum specimen deflection of 0.12 mm and flexural modulus E_b = 22 GPa. The experimental relationship between bending stress and specimen deflection is shown in Fig. 2a. The foregoing characteristics were calculated as the mean of 5 values.

Experimental data points for the fatigue bending test and their approximation curve are presented in Fig. 2b. The plot is known as a Wohler curve or an *S–N* diagram constructed for the assessment of the fatigue life [2]. The data about the specimens which exhibited spontaneous fracture and those which survived the fatigue test are omitted from the curve fitting. The fatigue limit was found to lie in the range from 33 to 34 MPa for 1.3×10^5 loading cycles, which accounted for about 0.75 of σ_b. The value of the fatigue limit derived from the *S–N* diagram equals the stress corresponding to the horizontal asymptote of the fitted curve for the fatigue life. The fatigue test results for the ceramic specimens exhibit a large spread in the experimental fatigue data, which is likely to be due to special manufacturing features and is consistent with the data about the fatigue life of this class of zirconia ceramics subjected to four-point bending tests reported in [6].

It will be recalled that inspection for internal microstructural defects in the bulk of the test material and for cracks and damage in the specimen were performed by the non-destructive testing technique with the use of an X-ray tomography system before and after the fatigue test. The result of the investigation was a voxel model for 3D imaging of the object under study. The model allows for imaging any surface or internal section of the specimen under study (Fig. 3). The as-sintered specimens are found to have a porosity of 15–30%. This class of oxide ceramics was examined in [4] by methods of scanning electron microscopy. The results obtained revealed a porosity level as high as 45%. The difference is attributable to higher resolution of the microscope used in [4] in which case micropores smaller than 3 μm make a major contribution. This falls outside of the resolving power of the X-ray tomography system used in our investigations.



(a) (b)

FIGURE 3. Voxel models of a ceramic specimen subjected to the fatigue test: longitudinal (a) and transverse sections (b) near the fracture surface

Figure 3 presents a voxel model for sections of part of the specimen that failed during the fatigue test. The specimen has structural defects, microdamage, and cracks in the fracture zone. The internal structural defects of the test specimen are represented as dense agglomerates of zirconia of size up to 1.5 mm. Longitudinal (*A*) and transverse (*B*) cracks are located in the agglomerate surrounding area. The agglomerates prevent further crack growth thus increasing the structural strength of the specimen without violating its integrity. Non-uniform distributions of spherical pores, microcracks, and zirconia agglomerates in the bulk of the test specimen are the cause of a large spread in the experimental data.

CONCLUSIONS

The bending strength (σ_b = 43 MPa) and flexural modulus (E_b = 22 GPa) of MgO-stabilized ZrO_2 specimens were evaluated by three-point bending tests. The low values of σ_b and E_b approach those found for bone tissues, which is a favorable condition for the survivability of implants produced from the test oxide ceramics.

Experiments were performed to investigate the fatigue behavior of the test specimens subjected to three-point bending at different levels of cyclic stresses. The fatigue life for each stress range studied as a function of the number of loading cycles to failure was plotted and approximated by a Wohler curve. The fatigue limit was found to lie in the range from 33 to 34 MPa for 1.3×10^5 loading cycles. Bending tests revealed a large spread in the fatigue values, in agreement with the results reported by other researchers [6]. This is due to the nature of the test material: the ceramic specimens produced by methods of powder metallurgy had a relatively large number of defects, such as zirconia agglomerates, pores, and microcracks. The latter are sources of fatigue cracks that impair the fatigue strength of the ceramic materials.

A 3D X-ray tomography investigation has revealed that the crack growth ceases in the neighborhood of the dense zirconia agglomerates in the bulk of the specimens. The agglomerates prevent further crack propagation thus increasing the structural strength of ceramic products.

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The study reported in this article was conducted according to accepted ethical guidelines involving research in humans and/or animals and was approved by an appropriate institution or national research organization. The study is compliant with the ethical standards as currently outlined in the Declaration of Helsinki. All individual participants discussed in this study, or for whom any identifying information or image has been presented, have freely given their informed written consent for such information and/or image to be included in the published article.

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