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A PROCEDURE FOR THE INVESTIGATION OF MECHANICAL AND PHYSICAL PROPERTIES OF CERAMICS UNDER THE CONDITIONS OF BIAXIAL BENDING OF A DISK SPECIMEN ACCORDING TO THE RING–RING SCHEME

B. D. Vasyliv

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A procedure and instruments are developed for the strength analyses of disk ceramic specimens under the conditions of biaxial bending according to the ring–ring scheme at high temperatures (up to 800°C) in a working hydrogen-containing medium of a solid-oxide fuel cell with continuous measuring of the physical characteristics of materials (electric resistivity and conductivity) as functions of time.

Keywords: ceramics, fuel cell, disk specimen, biaxial tension, hydrogen-containing medium, high-temperature, conductivity, strength.

The contemporary investigations of materials intended for elements of solid-oxide fuel cells are aimed at reducing the operating temperature to 550–600°C and elevation of their functional (conductivity) and operating (strength and durability) characteristics [1, 2]. For the anodes of fuel cells, it seems to be quite promising to use ceramics of the ScCeCZ system [zirconium oxide (ZrO_2) stabilized by adding 10 mol.% Sc_2O_3 and 1 mol.% CeO_2] modified with nickel oxide (NiO). In these anodes, NiO is reduced in the atmosphere of hydrogen, which may lead to significant changes in the physicomechanical properties of the material of the anode. In the literature, the results of investigations of the kinetics of reduction and changes in the mechanical properties of ScCeCZ–NiO ceramics in an atmosphere of high-temperature hydrogen (600–800°C) are practically absent.

This is why, to justify the changes in the mechanical and physical properties of the ceramics of anodes under the operating conditions, we developed a procedure and instruments for the investigation of strength and durability of disk ceramic specimens under the conditions of biaxial bending at high temperatures (up to 800°C) in the hydrogen-containing working medium of fuel cells with continuous recording of the parameters of loading (stresses and strains) and physical characteristics of the material (electric resistivity and conductivity) as functions of time.

There are two typical schemes of tests of this sort, namely, a ball–balls scheme (the load is applied along the axis by a ball punch to a specimen placed on three spherical supports) [3, 4] and a ring-ring scheme (the load is applied along the axis by an annular punch to a specimen placed on an annular support) [5]. The second scheme seems to be preferable because it gives a larger contact area (faces of the ring) that can be measured with high accuracy. By measuring the resistance of the specimen with a teraohmmeter, we find its resistivity and conductivity. On the basis of these observations, we developed a special device for the strength analyses of disk ceramic specimens under the conditions of biaxial bending according to the ring–ring scheme at high temperatures (up to 800°C) in the working hydrogen-containing medium of fuel cells (Fig. 1a) with continuous measuring of resistivity and conductivity as functions of time.

Specimen 1 (Fig. 1b) is concentrically held by stoppers 2 on the lower supporting ring 3 and loaded by the upper supporting ring 4.

Karpenko Physicomechanical Institute, Ukrainian National Academy of Sciences, Lviv, Ukraine; e-mail: vasyliv@ipm.lviv.ua.

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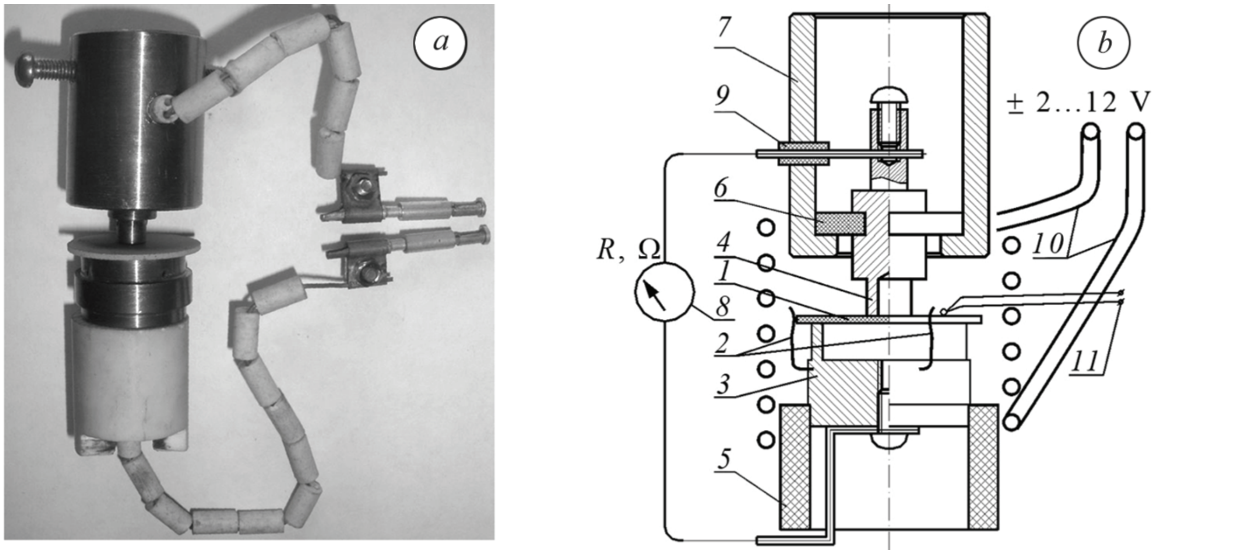

Fig. 1. General view of a ring–ring installation for loading ceramic disk specimens by biaxial bending (a) and its design (b): (1) specimen, (2) stoppers, (3, 4) lower and upper supporting rings, (5) holder of the lower ring, (6, 9) insulating inserts, (7) holder of the upper ring, (8) E6-13 teraohmmeter, (10) heating element, (11) thermocouple.

The lower ring is fixed in holder 5 and the upper ring is fixed through the insulating insert 6 in holder 7. The wire of the electric circuit goes to teraohmmeter 8 (E6-13 model), on one side, from the upper supporting ring 4 through the insulating insert 9 in holder 7 and, on the other side, from the lower supporting ring 3 through holder 5. In the process of loading of the specimen, the end faces of the supporting rings close contacts of the electric circuit with a voltage of 100 V applied by teraohmmeter 8. The proposed scheme makes it possible to realize continuous recording of current values of the electric resistance of the specimen as a function of time.

The heating element 10 in the form of a nichrome coil is mounted in the chamber. The heating temperature is measured with thermocouple 11. The installation is energy-saving because its working space is localized inside the heater 10 of small volume with a power of 300 VA, which heats the specimens to 800°C for 5–10 min (Fig. 2). The uniformity of heating of the specimen in hydrogen is monitored at its nominal temperature equal to 600°C. Since the thickness of specimens is smaller than 1 mm, the temperature gradient across the thickness is insignificant. Two thermocouples were brought in contact with the upper surface of the specimen. One of these thermocouples (central) was connected to the system of control of heating and located at the center of the circle and the second (outer) thermocouple was located near the periphery. The temperature of the gas in the chamber outside the heater was kept equal to 185 ± 3°C by balancing the mode of heating of the coil with the mode of cooling of the casing of the chamber.

According to the results of measurements, the system of control of heating guaranteed the temperature range 600 ± 2.5°C (measured by the central thermocouple). In the peripheral regions, the temperature was equal to 605 ± 2.5°C (outer thermocouple). Since the indicated range satisfies the conditions of modeling of the operating mode of the fuel cell, for batch testing of the materials, the thermocouple was brought in contact with the specimen at a point located at the center of radius of the disk specimen. At this point, the temperature was kept within the range 602 ± 2.5°C. At the center and on the periphery of the specimen, the nominal temperatures were equal to 600 and 605°C, respectively, with identical deviations equal to ± 2.5°C.

The stresses and strains are continuously recorded as functions of time by analyzing the signals of force and displacement gauges mounted outside the chamber. In the course of the tests, all signals of the gauges passing through an E14-140/D analog-to-digital converter to a computer are processed by the ACTest-Light software.

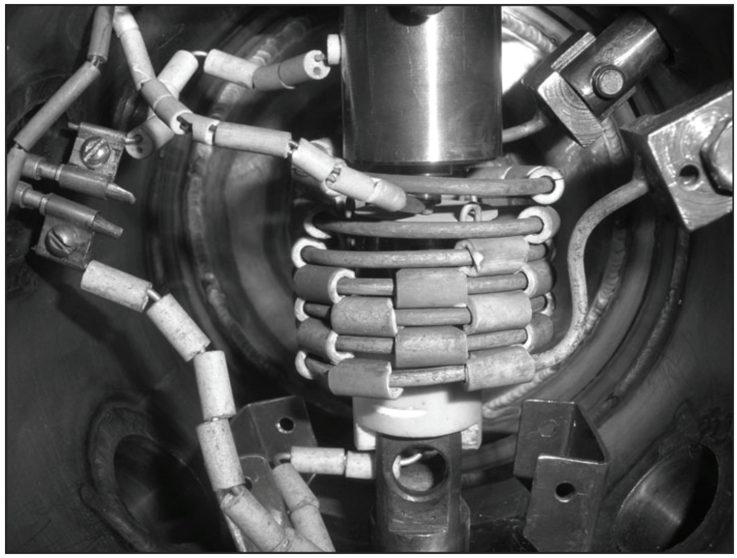

Fig. 2. Location of the ring–ring installation in the chamber with hydrogen-containing medium.

The stresses (MPa) are computed by using the formula [5]

$$\sigma_{max} = \frac{3P}{2\pi t^2}\left[(1-\nu)\frac{D_S^2 - D_L^2}{2D^2} + (1+\nu)\ln\frac{D_S}{D_L}\right], \quad (1)$$

where P is the load acting upon the specimen (N), ν is Poisson's ratio, t is the thickness of the specimen (mm), D_S is the diameter of the supporting ring (mm), D_L is the diameter of the loading ring (mm), and D is the diameter of the specimen (mm).

The radial and tangential components of bending stresses can also be found by using the following analytic relations [6]:

$$\sigma_r = \frac{M_r z}{t^3/12} \quad \left(\text{for } z = \frac{t}{2}, \text{ we have } \sigma_r = \frac{6M_r}{t^2}\right), \quad (2)$$

for the radial stresses (MPa), where

$$M_r = \frac{P}{4\pi}(1+\nu)\ln\frac{R}{r} \quad (3)$$

is the radial moment of forces (N·mm),

$$R = \frac{D_S}{2},$$

z is the coordinate of the line of action of stresses (mm), and r is a current computed distance from the axis of the specimen (mm);

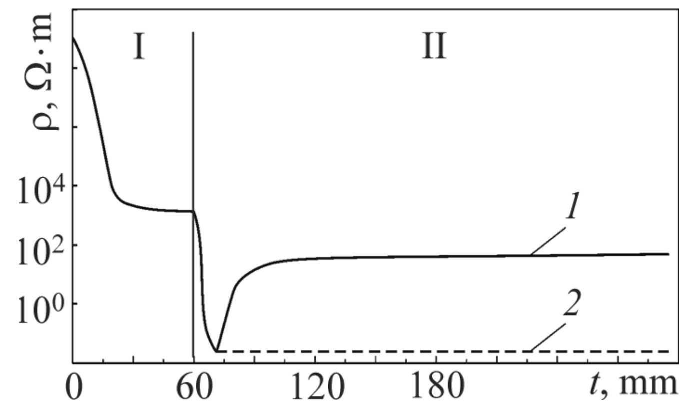

Fig. 3. Time dependences of the electric resistivity of the ceramic anode: (I) in air; (II) in a hydrogen-containing medium under the conditions of incomplete (1) and complete (2) reduction.

Table 1. Comparison of the Computed Fracture Stresses in Disk Specimens

Load P, N	Fracture stresses given by different relations, MPa		
	(1)	(2)	(4)
20	24.9	21.3	30.4
40	49.8	42.6	60.6
60	74.7	63.9	91.2

for $r = R$, we have $M_r = 0$;

for $r \le \frac{D_L}{2}$, we have $M_r = \frac{P}{4\pi}(1+\nu)\ln\frac{D_S}{D_L}$;

$$\sigma_\Theta = \frac{M_\Theta z}{t^3/12} \quad \left(\text{for } z = \frac{t}{2}, \text{ we have } \sigma_\Theta = \frac{6M_\Theta}{t^2}\right) \tag{4}$$

for the tangential stresses (MPa), where

$$M_\Theta = \frac{P}{4\pi}\left[(1+\nu)\ln\frac{R}{r} + (1-\nu)\right] \tag{5}$$

is the tangential moment of forces (N·mm);

for $r = R$, we have $M_\Theta = \frac{P}{4\pi}(1-\nu)$;

for $r \le \frac{D_L}{2}$, we have $M_\Theta = \frac{P}{4\pi}\left[(1+\nu)\ln\frac{D_S}{D_L} + (1-\nu)\right].$

The results of evaluation of stresses are presented in Table 1.

The highest tangential stresses in the specimen are given by relation (4). The radial stresses given by relation (2) are lower than the tangential stresses (4) and close to the stresses given by relation (1). Thus, for the subsequent evaluation of the fracture stresses in disk specimens and finding the ultimate strength of materials, it is necessary to use relation (1) [or (2)].

It is reasonable to study the joint influence of high temperatures and hydrogen on the conductivity of ScCeCZ–NiO ceramics by making the mode and of testing and the medium as close to the operating conditions of the fuel cell as possible. Therefore, the proposed procedure includes two consecutive steps (Fig. 3), namely, the preliminary step of heating of the specimen from the room temperature to a temperature of 600°C in air (corresponds to the startup mode of the fuel cell) and the step of operation with evacuation of air from the chamber followed by the delivery of hydrogen-containing gaseous mixtures or purified hydrogen (corresponds to the operating mode of the fuel cell).

The time dependences of the conductivity and strength characteristics of the anode ceramics with various contents of NiO obtained by using the proposed procedure can be used for the optimization of their chemical compositions.

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