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EFFECT OF HIGH-TEMPERATURE INERT GAS ATMOSPHERE ON THE THERMAL STABILITY OF ADVANCED REFRACTORY MATERIALS

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The effect of long-term exposure to a high-temperature gas atmosphere on the stability of ceramic materials has been investigated. Material samples based on ZrO_2 (Zr85, Zr95), Al_2O_3 (Al100), Al_2O_3-SiO_2 (Al63, Al70, Al76, Al96) and SiC (Si100) were exposed to an atmosphere of dry helium and nitrogen (the dew point of water was approximately -70 °C) at a temperature of 900 °C for 1000 hours. Bending-strength tests at 20 °C and Hot modulus of rupture (HMOR) tests at 900 °C were performed on the exposed and unexposed samples. In addition, the chemical composition of the materials tested and their criteria of relative density, i.e., bulk density, apparent porosity and water absorption, were determined together with their changes after the material exposure. The most pronounced change in the bending strength at 20 °C was observed after the Zr95 exposure to nitrogen, with an increase of up to approximately 25 % from the initial state. At 900 °C, the bending strength increased by about 15–20 % for Zr95 and Zr85 after exposure to helium and nitrogen. On the other hand, when Al63 was exposed to helium and nitrogen at 900 °C, its bending strength decreased.

There were no substantial changes in the bending strength of the other materials tested after the exposure. Also, the exposure did not have a significant effect on the change in the criteria of relative density or chemical composition of any of the tested materials.

INTRODUCTION

Refractory materials are indispensable for heavy industry, astronautics and energy applications because of their high melting point and their ability to withstand very high temperatures (1600–2300 °C) even after long-term exposure [1]. Pure oxides, such as SiO_2, Al_2O_3, MgO, CaO, and ZrO_2, or their compounds are typical refractory materials, but they can also be carbides, nitrides, borides, silicides, or even graphite [2-6].

The areas that refractory materials are applied place high demands on them, especially in terms of their thermomechanical stability and corrosion resistance at high temperatures, insulation properties and thermal-shock resistance. Pure oxides have excellent high-temperature properties, but their production is more economically demanding than the manufacturing of products from mineralogically heterogeneous mixtures. In such materials, however, it is necessary to prevent the formation of low-melting eutectics, leading to property deterioration at high temperatures [7, 8].

One of the main indicators of the quality of refractory materials is their mechanical or thermomechanical stability at high temperatures. After prolonged high-temperature exposure, even some high-resistant materials begin to creep. Creep equations describe the dependence of the strain (ε) over time (t) at the stress applied at a temperature (T) and are useful for the evaluation of thermomechanical properties or the long-term stability at high temperatures [9, 10]. The thermomechanical properties and long-term stability at high temperatures are mainly affected by macropores, and also by low-melting phases, glass phases, grain size and crystal boundaries, as well as by the presence of so-called structural dislocations and vacancies in sintered ceramics [11, 12]. The glass phase of SiO_2, which becomes a viscous melt at high temperatures, tends to be the most common cause of low refractory properties in dense sintered materials and is responsible for an overall reduction in the mechanical strength and stability [13].

The high-temperature behaviour of oxide and non-oxide materials has been described in various studies [14-16]. Current research is increasingly focused on the high-temperature behaviour of materials after very long exposure to different environments, with respect to the possible use of these ceramic materials mainly as construction and insulation components, e.g., in new types of nuclear reactors [17, 18].

Advanced nuclear reactors, also known as Generation IV reactors, use primary coolants different from those utilised in current pressurised-water nuclear reactors. Examples include melts of certain metals (Na, Pb, Pb-Bi) or salts, or helium. The use of these coolants will make it possible to achieve higher coolant temperatures (even above 900 °C in the case of helium) and, thus, increase the efficiency and applicability of these systems [19]. The high coolant temperatures place high demands on the mechanical and corrosion resistance of the construction materials used. Special nickel alloys and some types of ceramic materials have been designed for the construction of exposed components of these reactor systems. Among the ceramic materials, in particular, silicon carbide-based materials are mentioned in connection with Generation IV nuclear reactors. They are expected to be used, among others, mainly for fuel cladding and other applications in the core of helium-cooled fast reactors (GFRs) [20]. The use of other high-temperature ceramic materials is also envisaged in advanced reactors, especially for electrical and thermal-insulation components as well as other thermal-circuit components [20, 21]. Last, but not least, Al_2O_3-based ceramic materials and others are envisaged for components of large experimental facilities used for research in advanced nuclear reactors [22].

For economic reasons, ceramics with a high Al_2O_3 content are the most advantageous material for most of the above applications. Corundum-based ceramics have excellent high-temperature properties (e.g., refractoriness under load, hot modulus of rupture, lower creep in compression) and are relatively cheap and available [23]. The disadvantage of these types of ceramics could be their higher thermal expansion (or lower thermal-shock resistance).

This paper focuses on the thermomechanical properties of eight selected ceramic materials with high Al_2O_3, ZrO_2 and SiC contents after exposure to a helium atmosphere, simulating the primary-circuit environment of helium-cooled reactors, at a temperature of 900 °C for 1000 hours. For comparison, samples of these materials have also been exposed to a nitrogen atmosphere at the same temperature.

EXPERIMENTAL

The study focuses on the high-temperature behaviour of ceramic materials with a high Al_2O_3 content and other, mainly sintered, materials from the Al_2O_3-SiO_2 system. These materials are widely available and commonly used as the basis of refractory building components or for thermal insulation. Materials with a high ZrO_2 content have also been studied. SiC-based sintered non-oxide ceramics were the last material included in the experiment.

Materials

Commercially available ceramic materials for special applications were used for the experiment. ZrO_2-based ceramics and corundum Al100 (single-component Al_2O_3) are relatively pure systems. Sample Zr95 is a binary ZrO_2-MgO system and sample Zr85 is a ternary ZrO_2-Y_2O_3-Al_2O_3 system. More impurities and sintering additives are contained in the corundum ceramics designated as Al96. Sintered mullite ceramics (Al63) also contain a relatively high amount of admixtures to the basic Al_2O_3-SiO_2 system (ca 4 wt. % of impurities or sintering additives). In contrast, the mullite–corundum samples, Al70 and Al76, have a reduced impurity content (ca 98 wt. % is formed by the Al_2O_3-SiO_2 system). SiC-based sintered ceramics contain a minimum amount of impurities (below 0.4 %).

Table 1. The chemical composition of the samples before exposure.

Material designation	Al63	Al70	Al76	Al96	Al100	Zr95	Zr85	Si100
Composition [wt. %]								
Al_2O_3	62.86	69.01	76.11	95.58	99.56	0.12	3.35	0.08
SiO_2	32.99	28.95	21.9	2.17	0.10	-	-	-
K_2O	1.76	0.60	0.59	-	-	-	-	0.06
Na_2O	0.23	0.17	0.16	-	-	-	-	0.14
MgO	0.25	0.20	0.19	0.92	0.24	2.63	-	-
CaO	0.26	0.11	0.11	1.21	-	-	-	0.05
Fe_2O_3	1.01	0.49	0.51	-	-	-	-	-
TiO_2	0.52	0.38	0.39	-	-	-	-	-
ZrO_2	-	-	-	-	-	95.03	87.26	-
HfO_2	-	-	-	-	-	1.83	1.69	-
Y_2O_3	-	-	-	-	-	0.15	7.56	-
SiC	-	-	-	-	-	-	-	99.53

Table 2. Selected properties of the measured samples.

Material designation		Al63	Al70	Al76	Al96	Al100	Zr95	Zr85	Si100
Type of ceramics		mullite	mullite–corundum	mullite–corundum	corundum	corundum	zirconia	zirconia	carbide
Main component content	%	60.0	70.0	80.0	95.0	99.5	99.7	99.7	99.9
Ceramic body characteristic	-	sintered	porous	porous	sintered	sintered	sintered	sintered	sintered
Imporosity	-	perfect	-	-	perfect	perfect	perfect	perfect	perfect
Water absorption	%	0	10–16	5–10	0	0	0	0	0
Bulk density	$kg\cdot m^{-3}$	$2.5\cdot10^3$	$2.3\cdot10^3$	$2.4\cdot10^3$	$3.8\cdot10^3$	$3.8\cdot10^3$	$5.7\cdot10^3$	$5.6\cdot10^3$	$3.2\cdot10^3$
Bending strength at 20 °C	MPa	120	10	30	375	300	500	400	450
Mean coefficient of thermal expansion	K^{-1}	$5–6\cdot10^{-6}$	$5–6\cdot10^{-6}$	$3–6\cdot10^{-6}$	$6–8\cdot10^{-6}$	$7–8\cdot10^{-6}$	$9–10\cdot10^{-6}$	$9–11\cdot10^{-6}$	$3.7–4.4\cdot10^{-6}$
Thermal-shock resistance	K	150	400	250	140	150	-	-	180

Sample exposure

Long-term exposure to a gas atmosphere was tested in the laboratory apparatus shown in Figure 1, which included two drying columns. The first column was filled with silica gel, which dries the gas to a water dew point of approximately -40 °C. The second column was filled with a molecular sieve, drying the gas to a water dew point of roughly -70 °C. The experiments were performed in an inert atmosphere of helium and nitrogen. The samples were placed in a reactor holder in a tubular furnace that provided a constant temperature over the entire sample surface. At the beginning of the experiment, the temperature in the furnace was increased steadily at a rate of 1 °C per minute up to a temperature of 900 °C. This temperature was maintained for 1000 hours. Subsequently, the temperature was gradually reduced at a rate of 1 °C per minute to ambient temperature (approximately 25 °C), at which point the exposure was terminated. The gas flow through the reactor was about 0.03 $l\cdot min^{-1}$. The overpressure in the whole apparatus was controlled by a manometer (filled with glycerine) placed next to the tubular furnace.

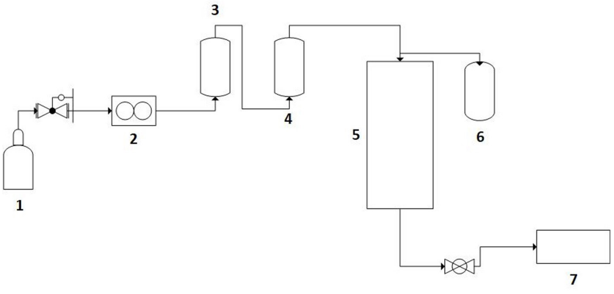

Figure 1. Diagram of the laboratory apparatus for the sample exposure: *1 – tested gas, 2 – flow regulator, 3 – drying column with silica gel, 4 – drying column with a molecular sieve, 5 – tubular furnace with a reactor, 6 – manometer, 7 – fume hood.*

Sample Testing Methods

Bending Strength and Hot Modulus of Rupture (HMOR)

The bending strength and HMOR were tested according to the procedure described below at 20 °C and 900 °C on the cylindrical samples (6 mm in diameter and 160 mm long), unexposed and exposed to an atmosphere of helium (1000 h/900 °C) or nitrogen (1000 h/900 °C). The samples were heated to the test temperature at a rate of 5 °C·min and loaded with increasing pressure. The distance of the supports during the test was 125 mm. The HMOR was measured after being held at 900 °C for 1 h. The temperature of 900 °C was chosen for the application in nuclear reactors because it is close to their operating temperature.

To test the strength by the three-point bending method at high temperatures, a special device (see Figure 2) was created by modifying a high-temperature furnace with eight Super Kanthal heating elements (see Figure 3). A lever load mechanism was inserted into the top opening of the furnace to continuously achieve increasing pressure on the specimen during the strength testing at the selected temperature. The cylindrical sample was placed on two supports

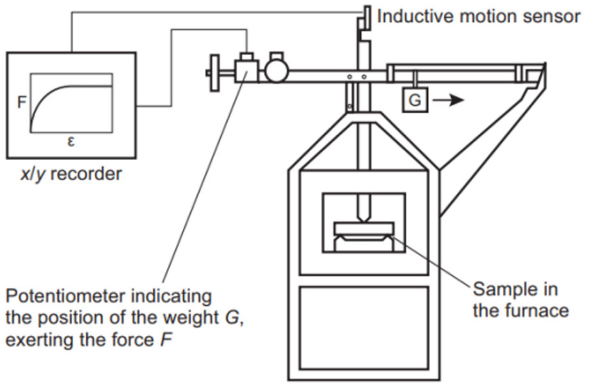

Figure 2. Furnace diagram for the bending-strength and HMOR testing [17].

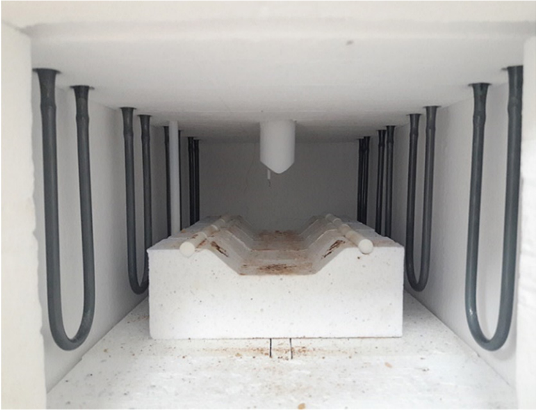

Figure 3. The inside of a furnace for the bending-strength and HMOR testing [17].

and uniformly loaded with a continuously increasing pressure in the centre until rupture. The bending strength test was carried out according to the standard EN 993-6 (Methods of Test for (Dense) Shaped Refractory Products – Part 6: Determination of Modulus of Rupture at Ambient Temperature) and the HMOR test according to EN 993-7 (Methods of Test for (Dense) Shaped Refractory Products – Part 7: Determination of Modulus of Rupture at Elevated Temperatures).

Criteria of relative density

The term "criteria of relative density" refers to the degree to which the space is filled with solid phases, and the density is, thus, expressed as the ratio of the volume of the solid phase to the total volume. This test consists of the saturation of the sample (or open pores) with a liquid followed by weighing. The samples are cleaned and dried at 105 °C for 1 hour. Their weight is determined to the nearest 0.01 g. The samples are placed in a container of liquid and saturated with the liquid by boiling or under vacuum. The saturated samples are weighed again on a balance in the air and then on a hydrostatic balance submerged in water. The criteria of relative density, i.e., bulk density, water absorption and apparent porosity, were determined on the sample residues after the bending-strength measurements. The measurements were carried out on both the unexposed and exposed samples in the helium and nitrogen atmospheres (900 °C/1000 h) according to the standard CSN 72 5010 (Determination of Water Absorption, Apparent and True Porosity, Bulk Density and Apparent Solid Density of Fired Ceramic Materials and Products).

RESULTS AND DISCUSSION

Exposure to Helium

The chemical composition of all types of the exposed samples was determined by X-ray fluorescence analysis (on the ground sample residue after the bending-strength measurements). The results of the measurements are summarised in Table 3. The chemical composition of the materials tested is not affected by their exposure to helium (900 °C/1000 h) – the observed deviations are within standard statistical errors (see Table 3).

Table 3. Chemical composition after exposure to helium.

Material designation	Al63	Al70	Al76	Al96	Al100	Zr95	Zr85	Si100
Composition [wt. %]								
Al_2O_3	62.82	70.77	77.12	95.82	99.6	0.19	3.44	0.18
SiO_2	33.49	27.26	21.12	1.98	0.08	-	-	-
K_2O	1.59	0.59	0.44	-	-	-	-	-
Na_2O	0.21	0.17	0.09	-	-	-	-	0.11
MgO	0.20	0.19	0.18	0.91	0.23	2.60	-	-
CaO	0.23	0.11	0.11	1.21	-	-	-	0.08
Fe_2O_3	0.86	0.48	0.48	-	-	-	-	-
TiO_2	0.47	0.37	0.41	-	-	-	-	-
ZrO_2	-	-	-	-	-	94.97	87.17	-
HfO_2	-	-	-	-	-	1.81	1.69	-
Y_2O_3	-	-	-	-	-	0.16	7.62	-
SiC	-	-	-	-	-	-	-	99.60

Exposure to Nitrogen

The chemical composition of all types of the samples exposed to nitrogen was determined by the same method as that used for the samples exposed to helium. The results of the analyses are shown in Table 4. The chemical composition is not affected by the exposure to nitrogen atmosphere (900 °C/1000 h) either.

Table 4. Chemical composition after exposure to nitrogen.

Material designation	Al63	Al70	Al76	Al96	Al100	Zr95	Zr85	Si100
Composition [wt. %]								
Al_2O_3	63.29	70.39	76.45	95.56	99.44	0.28	3.23	0.11
SiO_2	33.01	27.50	21.58	2.09	0.23			
K_2O	1.56	0.63	0.57					
Na_2O	0.26	0.16	0.14					0.11
MgO	0.22	0.21	0.18	0.92	0.23	2.46		
CaO	0.23	0.12	0.11	1.34				0.06
Fe_2O_3	0.85	0.52	0.51					
TiO_2	0.46	0.37	0.40					
ZrO_2						94.99	87.42	
HfO_2						1.75	1.66	
Y_2O_3						0.25	7.54	
SiC								99.56

Bending Strength at Laboratory Temperature

Among the unexposed samples at the laboratory temperature, the highest bending strength was found for the ZrO_2-based sintered ceramics partially stabilised with MgO, for the ZrO_2-based sintered ceramics partially stabilised with Y_2O_3 and for the SiC-based sintered ceramics. These are followed by two types of corundum ceramics. The sintered mullite ceramics showed almost half the strength in comparison with the corundum ceramics. The lowest strength was measured for the porous mullite–corundum ceramics. When comparing the bending strength at the laboratory temperature (20 °C) between the unexposed and exposed samples (1000 h/900 °C) to helium (or nitrogen), it can be noted that there is no significant loss of strength (see Figure 4). The differences are within the experimental errors of the bending-strength assessment (ca 10 %). An increase in the bending strength at the temperature of 20 °C after long-term exposure for some sintered materials with a high bulk density is discussed below.

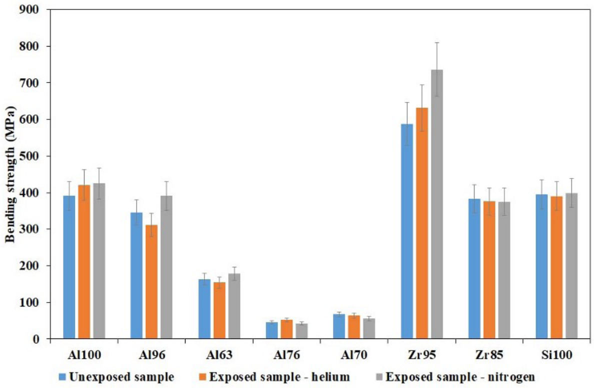

Figure 4. Bending-strength values of the unexposed ceramic samples at the laboratory temperature (20 °C).

Hot modulus of rupture at a temperature of 900 °C

When the modulus of rupture was measured at the elevated temperature (900 °C), the values for both the unexposed and exposed Si100 samples remained completely unchanged (relative to the bending-strength measurements at a temperature of 20 °C) as the Si100 properties are constant up to 1400 °C [24]. In the case of the pure corundum ceramics, Al100 and Al96, there is a slight decrease in the bending strength due to the low amount of sintering additives forming a eutectic melt, which softens the matrix [17]. At this temperature (900 °C), the HMOR of ZrO_2-based ceramics decreases as well, with the values being almost the same as those of corundum-based ceramics. In contrast, at the laboratory temperature (20 °C), these values are up two times higher, especially in the case of Zr95. It has also been found that Zr95 partially stabilised with MgO has a higher bending strength than Zr85 partially stabilised with Y_2O_3, which is not common due to the coarser structure in the case of the stabilisation with MgO, but a significant decrease in the bending strength at the higher temperatures may be caused by the ternary character of the system and the degree of stabilisation (only 2 % of MgO), as well as by the associated transformation changes. Due to the dissipation of energy for the grain modification changes, the partially stabilised ZrO_2 has much better mechanical properties than the fully stabilised ZrO_2. However, the degree of stabilisation of these commercial materials was not the subject of this research [24-26]. In the sintered mullite ceramic, Al63, no strength loss was observed despite its very heterogeneous composition, which can be attributed to the ability of the solid grains in the plastic matrix to absorb the energy required for the crack propagation and subsequent deformation [27]. The lowest strength was measured for the porous mullite–corundum samples. The results of the HMOR measurements show that even after long-term exposure, there is no crystal growth, which would reduce the strength of the samples. The X-ray diffraction also showed no changes in the modifications or phase composition.

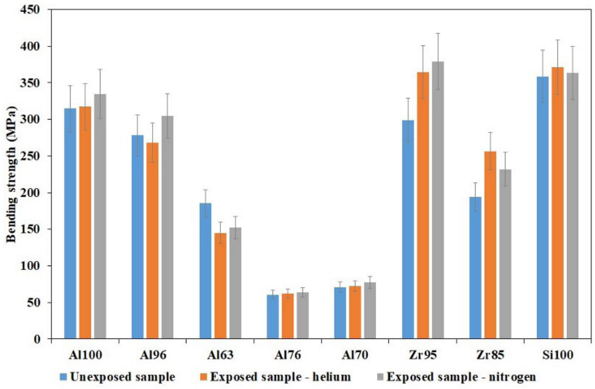

Figure 5. Bending-strength values of the unexposed ceramic samples at a temperature of 900 °C.

An interesting behaviour was observed in the exposed samples (1000 h/900 °C) of the sintered Zr95 zirconia ceramics partially stabilised with MgO, where prolonged exposure at higher temperatures can, by certain mechanisms, harden the structure, which then achieve a slightly higher strength than in the case of the samples not exposed for long periods [28]. For the samples of sintered corundum ceramics, Al100 and Al96, exposed to N_2, the HMOR values are higher only due to the higher bulk density of the material, which is explained in the following paragraph (AlN formation on the sample surface was excluded by the X-ray diffraction). For the other types of ceramics, no difference in the strength was observed between the unexposed and exposed samples at the laboratory temperature (20 °C) or elevated temperatures (900 °C), where the exposure did not affect their properties at all (see Figure 5).

Criteria of relative density

The highest bulk density is achieved by the ZrO_2-based samples, which is logically caused by the higher density of ZrO_2 in comparison with Al_2O_3 or SiO_2. For both types of corundum-based ceramics, the bulk density is almost the same. In the case of the sintered mullite ceramics, the bulk density naturally decreases further. For the porous samples of the mullite–corundum ceramics, the apparent porosity reaches values of 25.3 % for Al176 and 30.4 % for Al170 following the firing temperature of the ceramics (the higher the firing temperature, the lower the porosity). The SiC-based sintered ceramics have a bulk density of approximately 3200 kg·m^{-3}, which corresponds to its cubic modification, stable up to a temperature of 1700 °C. There were

Table 5. Relative density criteria for the unexposed and exposed ceramic samples.

Material designation	Al100	Al96	Al63	Al76	Al70	Zr95	Zr85	Si100
Unexposed sample								
Bulk density [kg·m^{-3}]	3710	3730	2600	2450	2410	5870	5830	3190
Apparent porosity [%]	0	0	0	25.3	30.4	0	0	0
Water absorption [%]	0	0	0	10.3	12.6	0	0	0
Exposed sample – He								
Bulk density [kg·m^{-3}]	3750	3720	2640	2460	2370	5740	5890	3120
Apparent porosity [%]	0	0	0	25.4	33.1	0	0	0
Water absorption [%]	0	0	0	10.4	13.9	0	0	0
Exposed sample – N_2								
Bulk density [kg·m^{-3}]	3830	3840	2690	2390	2390	5790	5840	3110
Apparent porosity [%]	0	0	0	25.6	31.3	0	0	0
Water absorption [%]	0	0	0	10.7	13.1	0	0	0

no significant changes either in the relative density criteria or bulk density after the exposure to helium or nitrogen (900 °C/1000 h), which corresponds with the findings that there were no changes in the chemical composition or the porosity of the samples (see Table 5). The slight changes in the bulk density between the unexposed and exposed sintered corundum ceramic samples, Al100 and Al96, are caused by the measurement of the different production batches, because the measurement of the bending strength is a destructive method, and it was not possible to expose the already cracked measured samples.

CONCLUSIONS

According to the results of these tests, the following has been found:

- There is no change in the chemical composition or relative density criteria of the tested materials during exposure.
- The highest bending-strength of the materials tested was exhibited by Zr95 ceramics – more than 600 MPa at 20 °C with an HMOR value around 300 MPa at 900 °C. After 1000-hour exposure to helium at 900 °C, the bending-strength values increased by about 10 %.
- Higher bending-strength values were found for the corundum ceramics Al100 and Al96 as well as for the materials Zr85 and Si100. The bending-strength values at 20 °C are about two-thirds that of Zr95, whereas the HMOR of the materials Si100, Al100, and Al95 at 900 °C is comparable to that of Zr95 at this temperature. There was no significant change in the bending-strength values of these materials after exposure to the helium and nitrogen atmosphere at 900 °C.
- The lowest strength values were found for the materials Al63, Al76, and Al70. Exposure of these materials to helium and nitrogen at 20 °C and 900 °C does not significantly affect the bending strength or the HMOR either.

Based on these results, it can be concluded that the tested materials are stable in a helium and nitrogen atmosphere at 900 °C. There was no significant decrease in the bending strength (and HMOR) or change in the chemical composition of any of the tested materials during exposure. The suitability of these materials for use as components of advanced gas-cooled nuclear reactors and other equipment will depend on the specific requirements for the components under consideration. It will also be necessary to consider different parameters, such as the electrical or thermal conductivity, thermal expansion, etc. For components intended directly for use in the core of a nuclear reactor, the resistance of the materials to neutron-radiation flux will also need to be considered. Nevertheless, the investigation into the effect of neutron radiation on material properties was not the aim of this experimental work.

Further research in this area could focus on, among other things, verifying the resistance of the tested materials in different atmospheres with different chemical compositions, or with exposure times longer than 1000 h.

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