



Superplastic Properties of Zirconia-Hafnia Composites Doped with Various Oxides

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Abstract

The influence of 1 mol% dopant of various oxides on the superplastic flow of yttria stabilized tetragonal zirconia ceramics and zirconia – 10 mol% hafnia composite was examined in the temperature range 1553–1773 K. It was claimed that doping caused the increase of superplastic strain rate in zirconia and in zirconia-hafnia composite in comparison with undoped materials. The maximum strain rate for zirconia was measured for GeO₂ addition; it was 16x higher than in pure ceramics, but for zirconia-hafnia the maximum strain rate was for SiO₂ dopant (4x higher than in pure composite). There is a linear relationship between the grain boundary diffusion coefficient of Zr⁴⁺ and superplastic strain rate for zirconia ceramics. For zirconia-hafnia composite this relationship is not so obvious because of high strain rate for SiO₂ addition.

Keywords: Zirconia, Hafnia, Superplasticity, Dopant, Diffusion

WŁAŚCIWOŚCI NADPLASTYCZNE KOMPOZYTÓW ZrO₂-HfO₂ DOMIESZKOWANYCH RÓŻNYMI TLENKAMI

Zbadano wpływ dodatku 1. % mol. różnych tlenków na nadplastyczne płynięcie ceramiki tetragonalnego dwutlenku cyrkonu stabilizowanego tlenkiem itru i kompozytu ZrO₂-10 % mol. HfO₂ w zakresie temperatury 1553–1773 K. Stwierdzono, że domieszkowanie spowodowało wzrost szybkości nadplastycznego odkształcania dwutlenku cyrkonu i kompozytu ZrO₂-HfO₂ w porównaniu z materiałami bez domieszek. Maksymalną szybkość odkształcania zmierzono w przypadku dodatku GeO₂; była ona 16x większa niż w czystej ceramice, ale w przypadku ceramiki ZrO₂-HfO₂ maksymalna szybkość odkształcania pojawiła się dla domieszki SiO₂ (4x większa niż dla czystego kompozytu). Zależność pomiędzy współczynnikiem dyfuzji po granicach Zr⁴⁺ i nadplastyczną szybkością odkształcania ceramiki cyrkonowej była liniowa. W przypadku kompozytu ZrO₂-HfO₂ zależność ta nie była tak oczywista z powodu wysokiej szybkości odkształcania zmierzonej dla dodatku SiO₂.

Słowa kluczowe: ZrO₂, HfO₂, nadplastyczność, domieszka, dyfuzja

1. Introduction

One of the most interesting structural materials is 3 mol % yttria stabilized tetragonal zirconia (ZrO₂) polycrystals (3Y-TZP). Simultaneously, 3Y-TZP with submicrometer grain size exhibits superplastic deformation at temperatures above 1500 K [1-6]. Superplasticity creates an opportunity of easy shaping and joining of ceramic elements. However, at present the effect of cavitation followed by cracking of the ceramics during superplastic deformation is a serious limitation for a broad application of this phenomenon in the ceramic technology. During the last years several studies have been reported in order to improve the situation. For example, an addition of some oxides (Al₂O₃, SiO₂, GeO₂, TiO₂, MgO) to 3Y-TZP significantly enhances the superplastic flow in this material [7, 8]. Superplastic flow in 3Y-TZP is attributed to the grain boundary sliding controlled by the grain boundary diffusion of Zr⁴⁺ ions [4]. The dopant cations added to 3Y-TZP tend to segregate to the grain boundaries, thus they could affect grain boundary diffusion of Zr⁴⁺ and hence they could change the superplastic flow of the ceramics.

Prime mineral sources for ZrO₂ such as baddeleyite and zircon typically contain 1.5-3 wt% Hf with respect to Zr content. Most of the commercially available zirconium compounds (such as ZrO₂ powders used in the ceramic industry) contain this level of hafnium content as an expected impurity. It is caused by the remarkable similarity of hafnium compounds to those of zirconium both in structure and in chemistry. For example, the structures of HfO₂ and ZrO₂ are so close to each other that it is difficult to detect their individual presence using conventional powder X-ray diffraction. This great similarity is related to the electron configuration of Hf and Zr, respectively: 4f¹⁴ 5d⁵ 6s² and 4d² 5s². In the periodic table, the rare-earth elements preceding hafnium (from cerium to lutetium) add electrons to the inner 4f shell. Because no outer electrons have been added to compensate for the increased nuclear charge, there is a contraction in the atomic size to such an extent that element 72, hafnium, has a slightly smaller atomic size than element 40, zirconium. This results in the so-called lanthanide contraction. The atomic radii of Hf and Zr are close to each other; 0.1442 and 0.1454 nm, respectively,

so are their ionic radii (M^{4+}), 0.084 nm for Hf and 0.083 for Zr [9]. The electronegativity values are 1.23 for Hf and 1.22 for Zr [9]. These values are indicative of the almost identical chemistries exhibited by Hf and Zr and explain the origin of similarity between HfO_2 and ZrO_2 .

HfO_2 exhibits the following transformation with the increasing temperature:



Similarly, ZrO_2 exhibits:



It was shown above that a technologically important monoclinic \leftrightarrow tetragonal transformation starts and completes in HfO_2 at about 600 K higher than in ZrO_2 .

There is rather little information in the literature about mechanical properties of HfO_2 and $HfO_2 - ZrO_2$ ceramics, especially at high temperature. The aim of this work is the determination of superplastic flow parameters of 3Y-TZP with natural content of HfO_2 (about 1.4 mol%) and with 10 mol% HfO_2 doped with 1 mol% of SiO_2 , GeO_2 , TiO_2 , Al_2O_3 , MgO and $MgAl_2O_4$ in the temperature range from 1553 to 1773 K.

2. Experimental

The 3Y-TZP and $ZrO_2 + 10 \text{ mol\% } HfO_2$ composite materials were prepared. The following powders were used:

- 3 mol% yttria stabilized tetragonal ZrO_2 from Zhongshun Sci. & Tech. China. Besides HfO_2 (1.9 wt%) other impurity content did not exceed 0.05 wt%, crystallite size is 20 – 30 nm.
- HfO_2 from Alfa Aesar GMBH Germany. It contained 98.5 wt% HfO_2 and about 1.5 wt% ZrO_2 in a monoclinic phase, crystallite size is $0.5 \pm 0.3 \mu\text{m}$.
- Al_2O_3 (AKP-53 type) from Sumitomo Japan, impurity content was about 130 ppm, crystallite size was $0.32 \mu\text{m}$.
- $MgAl_2O_4$ (spinel S30CR type) from BaikaloX France, impurity content was about 45 ppm and crystallite size was about $0.2 \mu\text{m}$.
- SiO_2 , MgO , GeO_2 , TiO_2 supplied by various producers, of purity 99.999 %.

The batches of samples with 1 mol% of oxides (Al_2O_3 , SiO_2 , MgO , $MgAl_2O_4$, GeO_2 , TiO_2), pure 3Y-TZP and zirconia-hafnia composite (containing 10 mol% of HfO_2), with and without dopants were made. In order to obtain the samples ZrO_2 powder was mixed with chosen oxide powders by the ball-milling method for 24 h in water. Subsequently, the powders were uniaxially pressed under pressure of 20 MPa and then isostatically pressed at 120 MPa. Plates obtained by this method were sintered for 2 h at 1623 K in air at a heating and cooling rate of $2 \text{ K} \cdot \text{min}^{-1}$. The samples of $2.5 \times 2.5 \times 5 \text{ mm}^3$ for compression tests for superplasticity measurements were cut from the sintered plates. Some samples were polished and next thermally etched for 1 h at 1573 K in vacuum. The electron scanning microscope micrographs of the etched samples were used for measurements of average grain size. The results of grain size measurements are shown in Table 1. Densities of 3Y-TZP samples were larger than 97% of theoretical density which equals $6.1 \text{ g} \cdot \text{cm}^{-3}$, but for zirconia-hafnia composites densities were larger than 94 % of theoretical density which equals $6.45 \text{ g} \cdot \text{cm}^{-3}$.

Table 1. Grain size d in μm for doped 3Y-TZP and $ZrO_2 + 10 \text{ mol\% } HfO_2$ samples.

Dopant oxide	3Y-TZP	$ZrO_2 - HfO_2$
*	0.30 ± 0.06	0.25 ± 0.04
Al_2O_3	0.36 ± 0.09	0.28 ± 0.09
SiO_2	0.27 ± 0.07	0.32 ± 0.07
MgO	0.28 ± 0.06	0.31 ± 0.09
$MgAl_2O_4$	0.32 ± 0.04	0.32 ± 0.11
GeO_2	0.34 ± 0.05	0.33 ± 0.09
TiO_2	0.34 ± 0.08	0.31 ± 0.11

* - means the undoped sample.

The superplasticity tests were performed in compression geometry under constant load in air at 1553 to 1773 K and in the stresses range of 20-50 MPa using Zwick 1446 universal testing machine. Strain rate $d\varepsilon/dt$ versus stress σ was calculated from experimental data according to [10].

3. Results and discussion

In Fig. 1 some plots of the applied stress and strain in the function of time for undoped 3Y-TZP at 1773 K are presented as an example. From these types of plots (for five selected temperatures mentioned in Fig. 2) the relationship $d\varepsilon/dt = f(\sigma)$ was calculated according to [10] (Fig. 2). The strain rate $d\varepsilon/dt$ could be expressed as [1-5]:

$$\frac{d\varepsilon}{dt} = \frac{A_0 \cdot D \cdot G \cdot b}{k \cdot T} \cdot \left(\frac{b}{d}\right)^p \cdot \left(\frac{\sigma}{G}\right)^n, \quad (1)$$

where A_0 is a dimensionless constant, D is the diffusion coefficient ($= D_0 \exp(-Q/RT)$, where D_0 is the frequency factor, Q the activation energy and R the gas constant), G is the shear modulus, b is the Burgers vector, d is the grain size, k is Boltzmann's constant, p is the exponent of the inverse grain size and n is the stress exponent.

Relationships, shown in Fig. 2, were also evaluated for doped 3Y-TZP samples and $ZrO_2 - HfO_2$ composites. On the logarithmic scale these relationships are linear. The values of stress exponent n (Eq. 1) calculated by the least square method (LSM), were collected in Fig. 3.

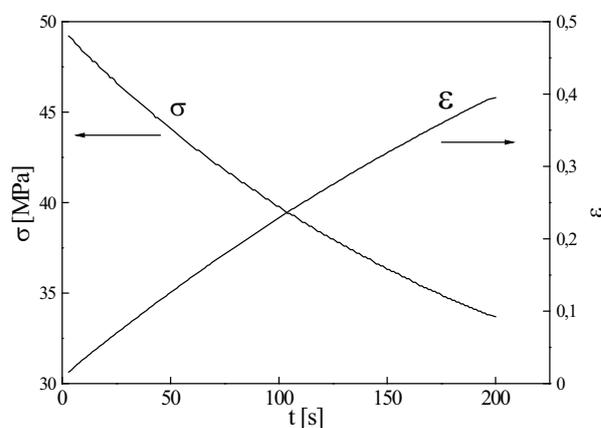


Fig. 1. Stress σ and strain ε in function of time t for undoped sample of 3Y-TZP in compression test at 1773 K. Initial stress was 50 MPa.

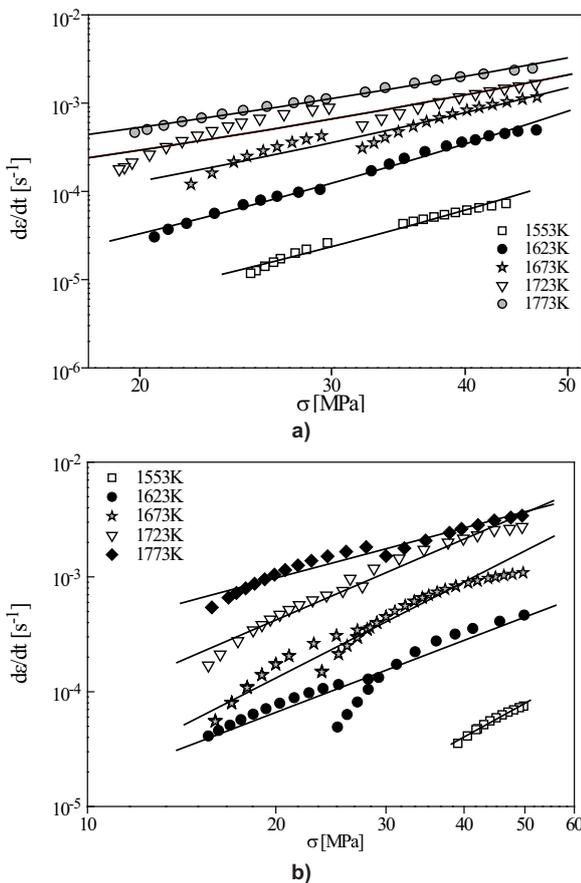


Fig. 2. Strain rate de/dt in function of stress s on a logarithmic scale for undoped materials for various temperatures: a) 3Y-TZP and b) ZrO_2 -HfO₂ composite.

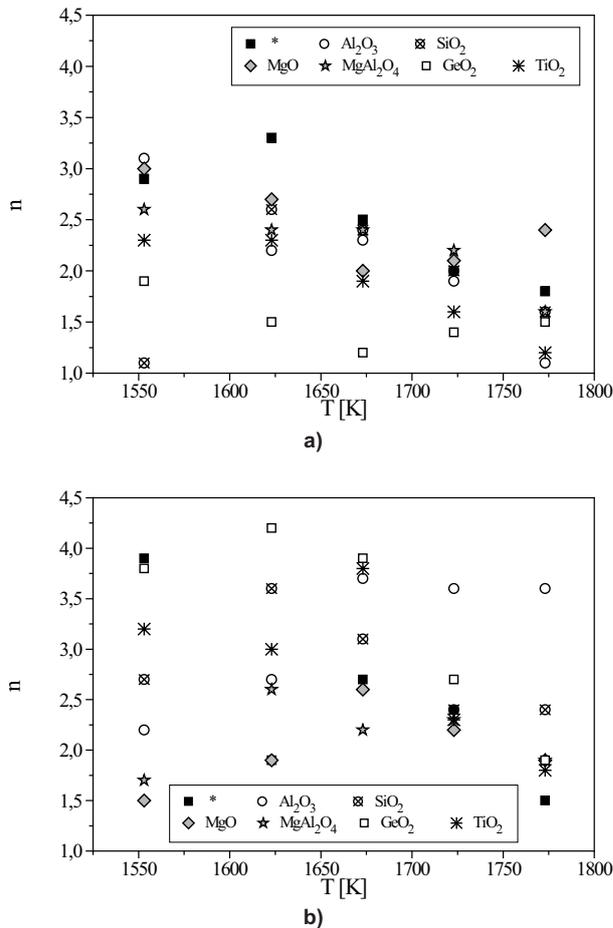


Fig. 3. Values of stress exponent n in function of temperature T for materials undoped and doped with various oxides: a) 3Y-TZP and b) ZrO_2 -HfO₂ composites.

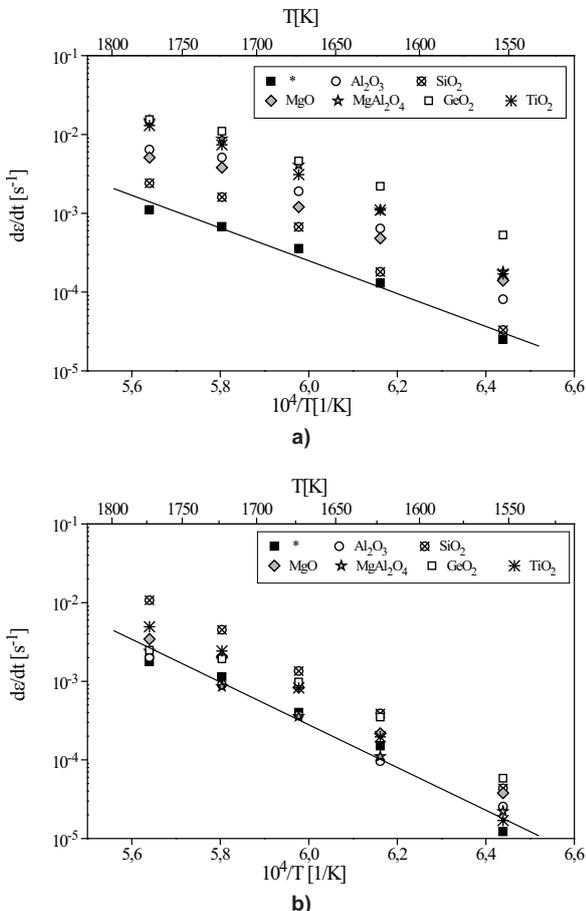


Fig. 4. Variation of strain rate de/dt with inverse temperature at 30 MPa: a) 3Y-TZP and b) ZrO_2 -HfO₂ composite. The solid lines concern undoped 3Y-TZP and ZrO_2 -HfO₂.

Next the effect of temperature on the deformation rate (for the mentioned above materials) is shown in Fig. 4 as an Arrhenius plot of strain rate vs reciprocal temperature for $\sigma = 30$ MPa. The values of activation energy Q were calculated by LSM and collected in Table 2.

Table 2. Activation energy Q for 3Y-TZP and ZrO_2 -HfO₂ composites in $kJ \cdot mol^{-1}$.

dopant oxide	3Y-TZP	ZrO_2 -HfO ₂
*	398 ± 32	520 ± 56
Al ₂ O ₃	468 ± 52	465 ± 18
SiO ₂	462 ± 38	576 ± 22
MgO	394 ± 28	482 ± 33
MgAl ₂ O ₄	470 ± 37	486 ± 8
GeO ₂	358 ± 24	399 ± 47
TiO ₂	455 ± 31	597 ± 43

As it is seen in Table 1, the grain sizes of studied ceramics are similar and equal to about 0.3 μm . Therefore, it is possible to evaluate the influence of dopants on the strain rates of 3Y-TZP and ZrO_2 -HfO₂ (Fig. 4) or the influence of HfO₂ on the strain rates of materials doped with the same oxides (Fig. 5).

The analysis of the presented data give us some interesting observations:

- The values of n decrease in the function of temperature

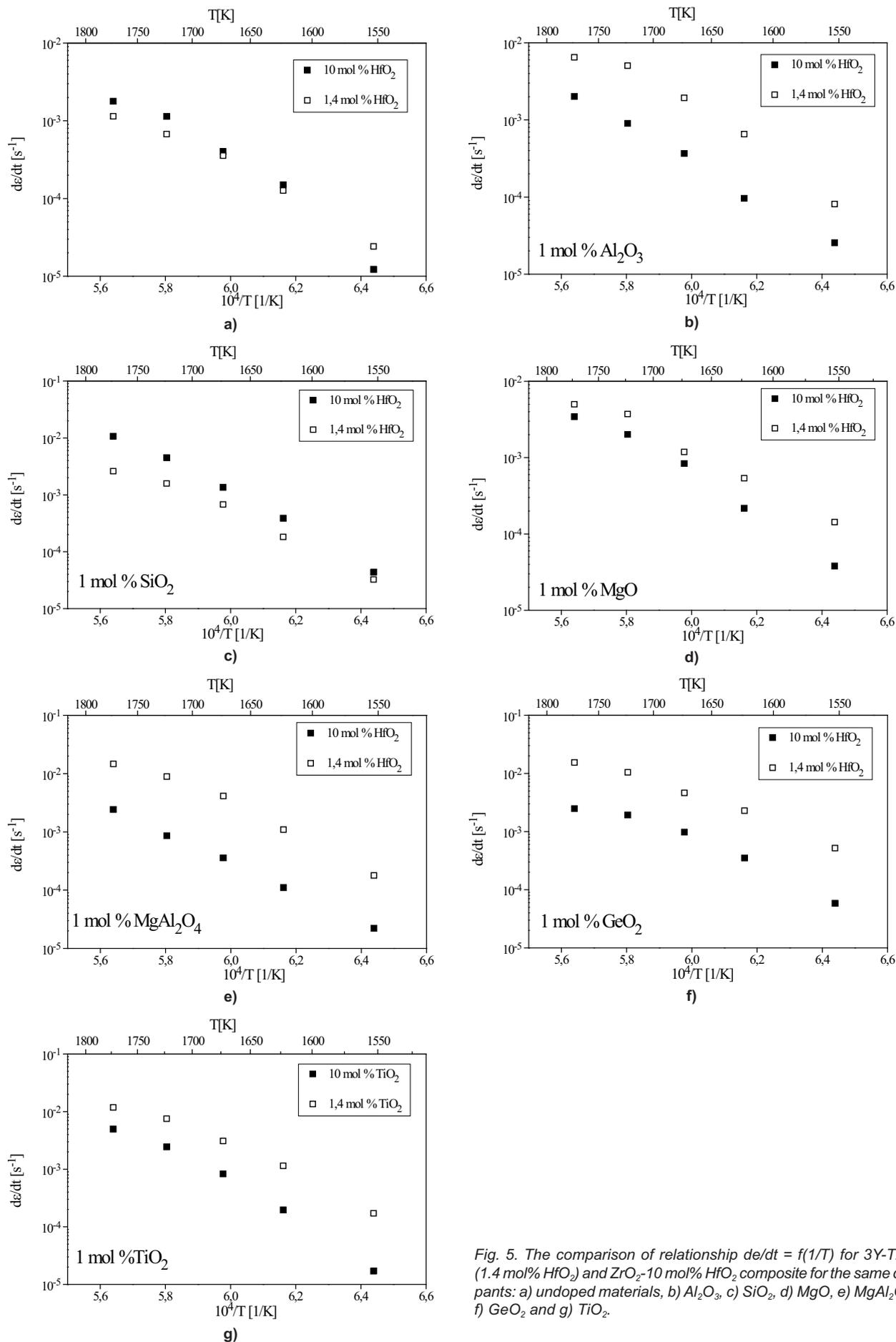


Fig. 5. The comparison of relationship $de/dt = f(1/T)$ for 3Y-TZP (1.4 mol% HfO₂) and ZrO₂-10 mol% HfO₂ composite for the same dopants: a) undoped materials, b) Al₂O₃, c) SiO₂, d) MgO, e) MgAl₂O₄, f) GeO₂ and g) TiO₂.

and the lowest values n at each temperature are for GeO_2 addition to 3Y-TZP (Fig. 3a) but there are no such effects in the case of $\text{ZrO}_2\text{-HfO}_2$ (Fig. 3b).

- The significant increase of strain rate is observed for all doped ceramics in comparison with the undoped one. The largest increase is for GeO_2 doped 3Y-TZP (for example at 1673 K for 30 MPa it is about 16x) and for SiO_2 doped $\text{ZrO}_2\text{-HfO}_2$ (about 4x at 1673 K for 30 MPa). Thus the influence of dopants is more significant in the case of 3Y-TZP (Fig. 4).
- For undoped 3Y-TZP and $\text{ZrO}_2\text{-HfO}_2$ the strain rate values are similar at each temperature. For almost all dopants (with the exception of SiO_2) the strain rates for 3Y-TZP are higher than for $\text{ZrO}_2\text{-HfO}_2$ (Fig. 5).
- For almost all dopants (with the exception of Al_2O_3) the values of activation energy Q are higher for $\text{ZrO}_2\text{-HfO}_2$ than for 3Y-TZP (Table 2).

Based on Eq. (1) the strain rate of superplastic flow is proportional to the diffusion coefficient D . The Zr^{4+} cation is regarded as the rate controlling ionic species for the superplastic flow in yttria-stabilized tetragonal zirconia [4]. The accommodation process for superplastic flow is usually explained in terms of grain boundary diffusion [4] or lattice diffusion [11]. The following experimental facts indicate that this is grain boundary diffusion rather than lattice diffusion that controls the superplastic flow:

- A small amount of cations significantly influences the superplastic flow (Fig. 4).
- The dopant cations tend to segregate along the grain boundary [7, 8, 12, 13] (Fig. 6).

There is no second phase particle or amorphous layer along the grain boundary; two grains are directly bonded at the boundary [7, 8, 12, 13] (Fig. 6).

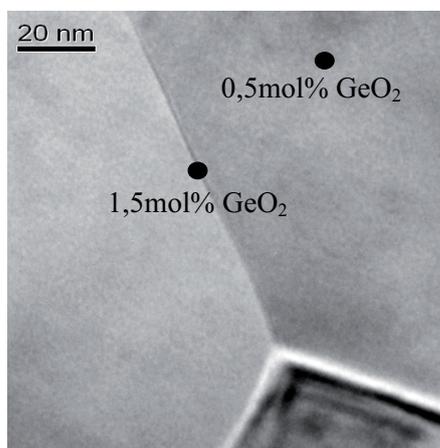


Fig. 6. High resolution transmission electron microscopy image of the area around a triple junction in GeO_2 doped 3Y-TZP. The GeO_2 concentration was measured by EDS with focused electron beam size about 5 nm. The GeO_2 concentration near grain boundary was three times higher than in grain interior.

The solubility limits of doping oxides in the tetragonal zirconia matrix are as follows: Al_2O_3 - 0.3 mol% at 1573 K [14], SiO_2 - 0.6 mol% at 1673 K [15], MgO - 2.3 mol% at 1023 K [16], GeO_2 - 3.5 mol% at 1623 K [17], TiO_2 - 13.8 mol% at 1573 K [18]. Strict information about the solubility limit of MgAl_2O_4 in ZrO_2 was not found. However, basing on some reports, for instance [19], one can conclude that it is close

to Al_2O_3 . Because the oxide content is 1 mol% thus in the case of Al_2O_3 , SiO_2 and MgAl_2O_4 the second phase locates at multiple grain junctions such as alumina or spinel grains [14] or glass pockets [15]. Then the grain boundary diffusion of Zr^{4+} ions in doped with SiO_2 , Al_2O_3 and MgAl_2O_4 zirconia should not depend on the second phase precipitation. Only the dopant cation which segregates in the grain boundaries could influence the diffusion.

The measurements of diffusion coefficient of Zr^{4+} were made in [20]. The results were combined with strain rate values of superplastic flow (Fig. 7).

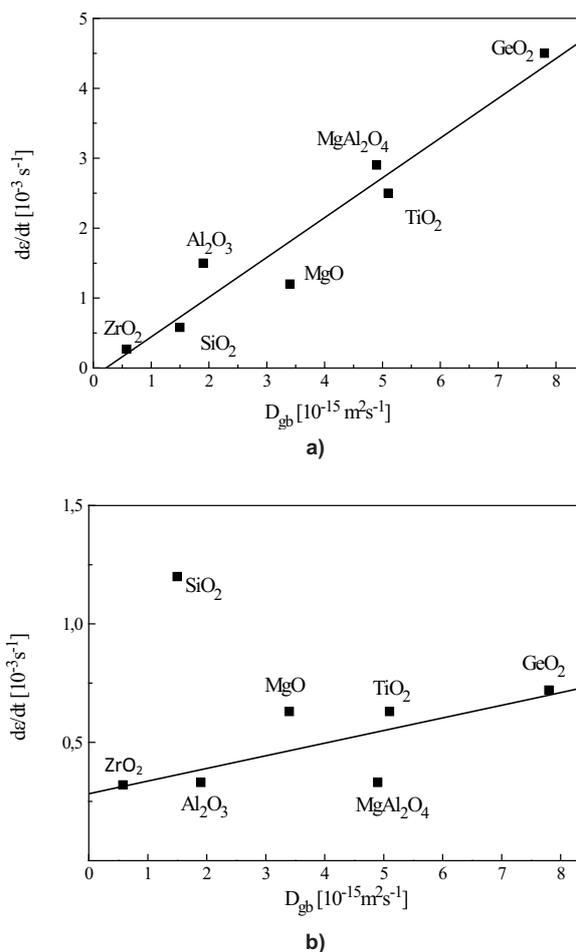


Fig. 7. The strain rate $d\epsilon/dt$ versus grain boundary diffusion coefficient D_{gb} of Zr^{4+} for undoped and 1 mol% selected oxide doped zirconia ceramics at $T = 1673$ K and $s = 30$ MPa: a) 3Y-TZP and b) $\text{ZrO}_2\text{-HfO}_2$; D_{gb} was measured with aid of an electron microprobe analyser by scanning of a cross section of the diffusion couple (3Y-TZP and ZrO_2+10 mol% HfO_2) (Zr-Hf interdiffusion) in [20].

The linear relationship between $d\epsilon/dt$ and D_{gb} for 3Y-TZP (Fig. 7a) confirms a thesis that grain boundary diffusion of Zr^{4+} plays the main role in the superplastic deformation of zirconia. The more complicated situation is in the case of $\text{ZrO}_2\text{-HfO}_2$ (Fig. 7b) because of the maximum of $d\epsilon/dt$ which appears for SiO_2 doped composite. It will be discussed later. Besides SiO_2 the strain rate is rather proportional to the grain boundary diffusion coefficient for the zirconia-hafnia composites (Fig. 7b).

The observed in [20] increase of the grain boundary diffusion is caused by a change in chemical bonding due to dopant cation segregation. The chemical bonding state

in TiO_2 and/or GeO_2 doped 3Y-TZP was investigated in [12, 21, 22] and in Al_2O_3 , SiO_2 , MgO , MgAl_2O_4 , GeO_2 and TiO_2 in [20] by the first-principles molecular orbital calculation. According to this calculation, doped cations and the surrounding O^{2-} anions form strong covalent bond and reduce the ionic bonds between the O^{2-} and Zr^{4+} . It enhances the Zr^{4+} diffusion and thereby increases the strain rate of superplastic flow.

The standard Nabarro-Herring and Coble diffusional creep models [23], involving vacancy diffusion, either through the lattice or along grain boundaries, respectively, lead to a stress exponent n of one, which is not consistent with the present observations (Fig. 3). These models are based on continuum models in which it is assumed that the grain boundaries act as perfect sources and sinks for vacancies. In practice, however, the grain boundaries contain dislocations which are discrete vacancy sources and sinks through their motion in the boundary planes. The presence of these sources and sinks leads to a significant modification to the continuum models for diffusion creep by introducing an interface-controlled behaviour and creep rates which may be significantly lower than in continuum calculations [4]. The interface-controlled diffusion creep is said to be "mobility-limited" because its rate is determined by local processes taking place at the interface (grain boundary) rather than by the kinetics of long-range diffusion [24]. One can say that the time for vacancy emission and absorption is substantially larger than taken for vacancy diffusion across the grain. In consequence, n values are usually larger than 1. At higher temperature and for some dopants the amount of the vacancy sources and sinks increases and $n \rightarrow 1$ (Fig. 3a). In $\text{ZrO}_2 - 10 \text{ mol\% HfO}_2$ sintered for 2 h at 1673 K monoclinic hafnia particles are dispersed at the tetragonal zirconia matrix. For undoped materials diffusion coefficients of zirconia and hafnia are the same (because of similarity of chemistry between Hf and Zr) and hence the strain rate values at the same temperature for 3Y-TZP and $\text{ZrO}_2\text{-HfO}_2$ are almost the same (Fig. 5). As it was shown in Fig. 4 and 5, the addition of other oxides to the zirconia-hafnia composite does not change the strain rate of superplastic flow as much as for 3Y-TZP (with exception of SiO_2). It seems that the dopants do not enhance the diffusion coefficients in HfO_2 . In SiO_2 doped composite the bigger strain rate increase is probably caused by creation of a new phase (hafnon – HfSiO_4) during sintering; it could accelerate the strain rate of the composite. The bigger values of activation energy in case of zirconia-hafnia than in 3Y-TZP (Table 2) mean that an enthalpy needed for vacancy creation in the first material is higher than in the second one.

It was shown [7, 8] that the flow stress at 10 % deformation at 1673 K under the strain rate 1.1 and $1.3 \cdot 10^{-4} \text{ s}^{-1}$, respectively, was roughly proportional to the ionic radius of dopant cation in 3Y-TZP. In Fig. 8 the relationship between the superplastic strain rate at 1673 K for 30 MPa and the ionic radius of dopant cations is presented.

There is no proportionality between strain rate and ionic radius of the dopant cation. Only some maxima are observed for Ge^{4+} doped 3Y-TZP and for Si^{4+} doped $\text{ZrO}_2\text{-HfO}_2$.

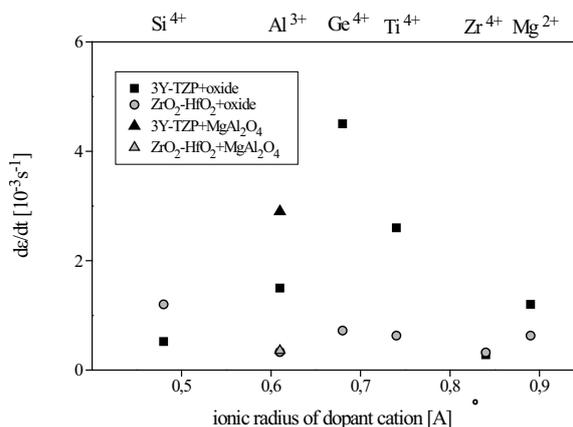


Fig. 8. A plot of strain rate de/dt at 1673 K and for 30 MPa against ionic radius of dopant cation in 3Y-TZP and $\text{ZrO}_2\text{-HfO}_2$. The ionic radius is for the cation of 8-fold coordination [25]. For MgAl_2O_4 , Al^{3+} ionic radius was taken.

4. Conclusions

The influence of 1 mol% dopant of various oxides on superplastic flow of 3 mol% yttria stabilized tetragonal zirconia ceramics and zirconia – 10 mol% hafnia composite was examined in the temperature range 1553–1773 K. It was claimed that:

- The values of stress exponent n in the creep equation (1) are in the range 1.1–3.3 and 1.5–4.2 for $\text{ZrO}_2\text{-HfO}_2$, respectively. For 3Y-TZP n decreases in the function of temperature and it is the smallest for GeO_2 doped material.
- The significant increase of strain rate is observed for all doped ceramics in comparison with the undoped one. The biggest increase is for GeO_2 doped 3Y-TZP (for example at 1673 K for 30 MPa it is about 16x) and for SiO_2 doped $\text{ZrO}_2\text{-HfO}_2$ (about 4x at 1673 K for 30 MPa). Thus the influence of dopants is more significant in the case of 3Y-TZP. It is supposed that this increase is caused by a change in chemical bonding between Zr and O ions due to dopant cation segregation to the grain boundary.
- For undoped 3Y-TZP and $\text{ZrO}_2\text{-HfO}_2$ the strain rate values are similar at each temperature. For almost all dopants (with the exception of SiO_2) the strain rates for 3Y-TZP are higher than for $\text{ZrO}_2\text{-HfO}_2$.
- For almost all dopants (with the exception of Al_2O_3) the values of activation energy Q are higher for $\text{ZrO}_2\text{-HfO}_2$ than for 3Y-TZP.
- There is a linear relationship between strain rate of superplastic flow and grain boundary diffusion coefficients of Zr^{4+} for 3Y-TZP. In the case of $\text{ZrO}_2 - \text{HfO}_2$ there is a strain rate maximum for SiO_2 doped samples.
- There is no linear relationship between strain rate of superplastic flow and ionic radius of dopant cation for both studied materials.

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