

Effect of Heat Treatment on the Thermal Conductivity of Single Crystals of ZrO_2 -Based Solid Solutions Stabilized with Scandium and Yttrium Oxides

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Abstract—The effect of heat treatment at 1000°C for 400 h on the thermal conductivity of zirconium dioxide crystals stabilized with scandium oxide $(\text{ZrO}_2)_{1-x}(\text{Sc}_2\text{O}_3)_x$ ($x = 0.08\text{--}0.10$) and simultaneously with scandium and yttrium oxides $(\text{ZrO}_2)_{1-x-y}(\text{Sc}_2\text{O}_3)_x(\text{Y}_2\text{O}_3)_y$ ($x = 0.003\text{--}0.20$, $y = 0.02\text{--}0.025$) has been studied. In the crystals of zirconium dioxide stabilized with scandium oxide, the most noticeable changes in the thermal conductivity are observed in the 9ScSZ crystals, in which the phase composition is changed and a marked content of the rhombohedral phase appears. These changes are less noticeable in the 8ScSZ crystals and they are mainly due to the ordering of oxygen vacancies and the changes in the microstructures of the samples, and there are no changes in the 10ScSZ crystals. The 10ScSZ crystals have the minimum electrical conductivity before and also after annealing, which is determined by the highest content of scandium oxide in the solid solution. Insignificant changes in the thermal conductivity are observed in crystals of partially stabilized zirconium dioxide co-alloyed with scandium and yttrium oxides. The cubic 8Sc2YSZ and 10Sc2YSZ crystals demonstrate only slight changes in the thermal conductivity, the character of the temperature dependence of the thermal conductivity, and the phase compositions. The alloying of the zirconia-based solid solutions with yttrium oxide simultaneously with scandium oxide enables one to increase the stability of their phase compositions and structurally dependent thermal and electrical physical characteristics.

Keywords: zirconium dioxide, single crystals, annealing, thermal conductivity, microstructure changes

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1. INTRODUCTION

High-temperature zirconia-based materials are widely used in engineering as solid electrolytes in various electrochemical devices as heat-resistant coatings of components of gas-turbine and Diesel engines to increase their operating temperature and energy efficiency [7, 8] and as structural materials capable of operating at high temperatures in oxidizing media [1–9]. For many applications of zirconia-based materials, it is necessary to know their thermal-physical characteristics.

The operating conditions of service of such materials are often high temperatures and oxidizing media. There are very many studies devoted to the aging of the materials in conditions which lead to changes in their phase compositions, structures, and physicochemical properties [10–16]. The changes in the thermal-phys-

ical properties of zirconia-based materials under action of high temperatures were studied in [14–16]. Alloying impurities added to modify the structural, mechanical, thermal- and electrophysical properties of such materials for specific applications substantially influence the aging processes.

According to the kinetic theory, in ZrO_2 materials stabilized with Y_2O_3 or simultaneously with Sc_2O_3 and Y_2O_3 , the internal heat transfer is mainly controlled by the directed motion of phonons carrying energy [17, 18]. At increased temperatures, the photon contribution should be also taken into account. The thermal conductivity is very sensitive to the structural features of a material, such as: oxygen vacancies, impurity cations, complexes of vacancies and impurity cations, and locally ordered structures. This fact enables the use the technique of experimental determination of

the temperature dependence of the thermal conductivity as a very useful method of revealing the structural features of crystalline materials and their changes under action of external conditions [17–19]. When studying zirconia-based ceramic materials, the zirconia solid solutions with the tetragonal structure co-alloyed with yttrium and scandium oxides were used [19]. The incorporation of scandium oxide into ZrO_2 stabilized with yttrium oxide enables one to obtain the t phase at relatively wide varying of alloying and to form the larger number of oxygen vacancies to increase the phonon scattering [20]. In addition, Sc^{3+} and Zr^{4+} have a large difference in masses that can significantly increase the phonon scattering and significantly decrease the thermal conductivity. As compared to $(\text{ZrO}_2)_{1-x}(\text{Sc}_2\text{O}_3)_x$, the $(\text{ZrO}_2)_{1-x-y}(\text{Sc}_2\text{O}_3)_x(\text{Y}_2\text{O}_3)_y$ solid solutions have a smaller mean free path of phonons and, correspondingly, lower thermal conductivity that gradually decreases with an increase in the Sc^{3+} content [19]. Such materials exhibit a combination of good mechanical characteristics and the low thermal conductivity, which makes them promising materials for heat-resistant coatings.

Before we studied the thermal conductivity of single-crystal ZrO_2 -based solid solutions co-alloyed with scandium and yttrium oxides [21]. Additional incorporation of yttrium oxide into zirconium dioxide is used to stabilize the cubic and tetragonal modifications of zirconium dioxide. It was shown that the thermal conductivity of crystals of zirconium dioxide stabilized by scandium oxide and co-stabilized with scandium and yttrium oxides decreases as the Sc_2O_3 content increases. This decrease is more intense than that observed with an increase in the stabilizing Y_2O_3 in the crystal, particularly, in the concentration range of scandium oxide more than 9 mol %. The existence of the twin structure and the second-phase inclusions also favors a decrease in the thermal conductivity of zirconia-based solid solutions.

It should be noted that the use of the single crystal samples for studying the influence of the structural features of the material on the thermal conductivity does not enable one to take into account the influence of grain boundaries, the existence of pores and other features inherent in polycrystalline ceramic materials and depending on the synthesis method.

The aim of this work is to study the influence of the heat treatment at 1000°C for 400 h on the thermal conductivity of the ZrO_2 crystals stabilized with scandium and yttrium oxides.

2. EXPERIMENTAL

The solid solution crystals of $(\text{ZrO}_2)_{1-x}(\text{Sc}_2\text{O}_3)_x$ ($x = 0.08\text{--}0.10$) and $(\text{ZrO}_2)_{1-x-y}(\text{Sc}_2\text{O}_3)_x(\text{Y}_2\text{O}_3)_y$ ($x = 0.003\text{--}0.20$, $y = 0.02\text{--}0.025$) were grown by the directional crystallization of melts in a cold container at a “Kristall-407” installation. The cold container

diameter is 120 mm, the high-frequency installation frequency is 5.28 MHz, the working atmosphere is air, and the crystallization rate is 10 mm/h. The initial materials are powder oxides with the purity no less 99.99 wt % of the base material.

The annealing was performed in a Supertherm HT04/16 high-temperature resistance furnace at 1000°C for 400 h in air.

The phase analysis was performed by X-ray diffraction on a Bruker D8 diffractometer using CuK_α radiation and by Raman spectroscopy. The excitation source was a laser with the wavelength 532 nm.

The crystal structure was studied using the optical and the transmission electron microscopy.

The thermal conductivity was measured on the samples cut in parallel to the growth axis having an arbitrary crystallographic orientation, since the anisotropies of the thermal conductivity of the monoclinic and tetragonal modifications of zirconia-based solid solutions are very insignificant [22]. The measurements after annealing were performed on the same samples, the thermal conductivity of which was determined immediately after the growth. The experimental determination of the thermal conductivity in the temperature range 50–300 K was performed by the absolute stationary method of the longitudinal heat flow. The apparatus and the measurement technique are described in [23]. The error of determining the absolute thermal conductivity was no higher than $\pm 6\%$.

3. RESULTS AND DISCUSSION

Table 1 gives the compositions of the heat-treated crystals, their denotations in the text, and the phase compositions.

The grown crystals have different phase compositions (Table 1) and different external views. The solid solutions crystals based on zirconia with scandium oxide were not optically homogeneous and transparent [24]. It is shown that these crystals had a twin structure formed as a result of phase transitions from the high-temperature cubic phase to the low-temperature tetragonal and rhombohedral phases. The additional incorporation of yttrium oxide to the single crystal compositions enabled the obtainment of transparent homogeneous 9Sc2YSZ and 10Sc2YSZ single crystals with the cubic structure [25]. The grown crystals of partially stabilized zirconia co-alloyed with yttrium and scandium oxides 0.3Sc2.5YSZ and 0.8Sc2YSZ had the shape and the sizes similar to the shape and the size of zirconia crystal partially stabilized only with yttrium oxide (PSZ) [26] with the concentration close to the summary concentration of the stabilizing oxides. They also had similar structure of twins in the crystal volume [26]. The appearance of the crystals is not changed after annealing (Fig. 1). Figures 2 and 3 show the results of the measurements

Table 1. Compositions of the study crystals and their corresponding denotations

Composition $\text{ZrO}_2\text{--R}_2\text{O}_3\text{--R}'_2\text{O}_3$, mol %			Denotation	Phase composition
ZrO_2	Sc_2O_3	Y_2O_3		
92	8	—	8ScSZ	<i>t</i> -ZrO ₂
91	9	—	9ScSZ	<i>t</i> -ZrO ₂
90	10	—	10ScSZ	<i>c</i> -ZrO ₂ <i>r</i> -ZrO ₂
97.2	0.3	2.5	0.3Sc2.5YSZ	<i>t</i> -ZrO ₂ <i>t'</i> -ZrO ₂
97.2	0.8	2	0.8Sc2YSZ	<i>t</i> -ZrO ₂ <i>t'</i> -ZrO ₂ <i>m</i> -ZrO ₂
89	9	2	9Sc2YSZ	<i>c</i> -ZrO ₂
88	10	2	10Sc2YSZ	<i>c</i> -ZrO ₂

of thermal conductivity $k(T)$ after annealing of the crystals at 1000°C for 400 h; the numerical data are given in Table 2. Figures 2 and 3 also show, for comparison, the temperature dependences of the thermal conductivity of the same crystal samples measured immediately after their growth.

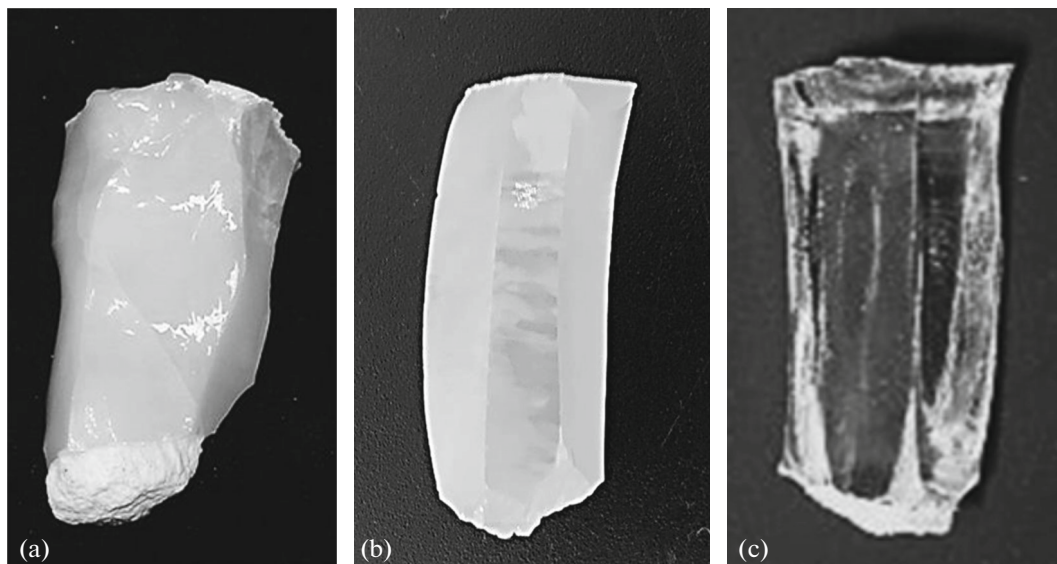
The results of the studies of the thermal conductivity were analyzed before and after annealing taking into account the phase compositions of the single crystals. The phase compositions of some crystals are changed after annealing at 1000°C for 400 h, other some crystals do not change their phase compositions.

In the case of insignificant changes in the phase compositions, the Raman spectroscopy is more sensitive to such changes as compared to the X-ray diffraction method.

Figure 4 shows the Raman spectra of the (8–10)ScSZ crystals measured before and after the annealing.

Before the annealing, the thermal conductivities of the tetragonal ScSZ crystals containing 8 mol % and 9 mol % are close to one other, and $k(T)$ monotonically increases with temperature, which is characteristic of structurally disordered media [3, 21, 27]; however, after the annealing, the difference in the thermal conductivities of these crystals increases, and the thermal conductivity of 8ScSZ is higher than that of the 9ScSZ crystal over entire temperature range. The dependence of the thermal conductivity on temperature becomes more substantial with the change in the slopes of both the curves, since the thermal conductivity of the 8ScSZ and 9ScSZ crystals decreases in the temperature range 50–100 K. This decrease is more substantial for the 9ScSZ crystals in which the phase composition is changed markedly due to the formation of the rhombohedral phase in them. These changes are well seen when comparing the Raman spectra measured before and after the annealing (Fig. 4).

Before annealing, the Raman spectra of the 9ScSZ crystals have the shape characteristic of the Raman spectra of the tetragonal phase [28, 29], the lines of which are significantly broadened, which is not inconceivable that there is a small amount of a second phase, in particular, of the quenched high-temperature cubic phase. After the annealing, the spectra have new lines characteristic of the spectra of the rhombohedral phase (Fig. 4). That is, during the annealing, as

**Fig. 1.** Appearance of crystals: (a) 0.3Sc2.5YSZ, 0.8Sc2YSZ; (b) 8ScSZ, 9ScSZ, and 10ScSZ; (c) 9Sc2YSZ and 10Sc2YSZ.

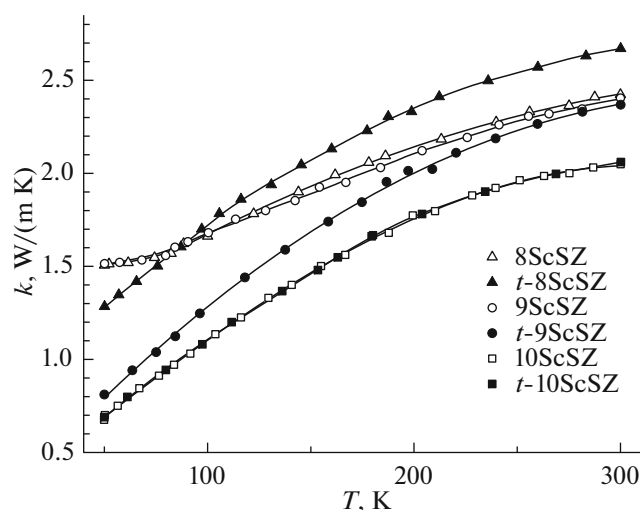


Fig. 2. Temperature dependences of the thermal conductivity of the ScSZ crystals measured before and after annealing.

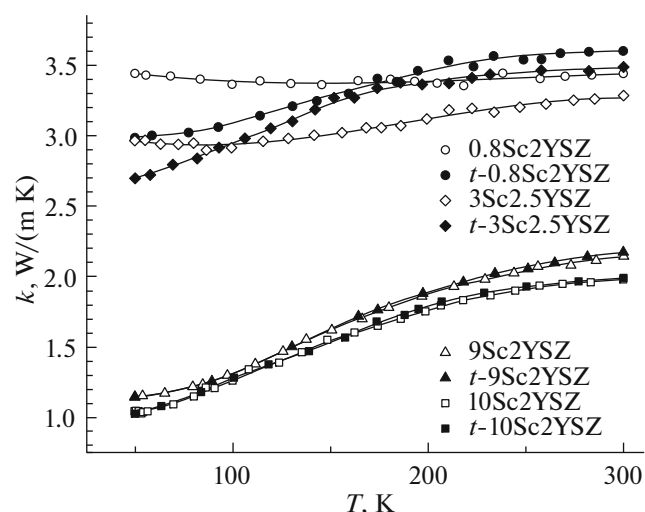


Fig. 3. Temperature dependence of the thermal conductivity of the ScYSZ crystals measured before and after annealing.

a result of phase transformations, the quenched high-temperature cubic phase degrades into the tetragonal and rhombohedral phases, which decreases the thermal conductivity. At a temperature of 300 K, the thermal conductivities of the 8ScSZ and 9ScSZ crystals are almost comparable with those of the crystals before annealing. This fact is due to an increase in the phonon concentration with temperature, which leads to the enhancement of the phonon–phonon scattering and to approaching the phonon mean free path to the minimum value compared to the interstitial distance in the crystal.

It should be noted that, after the annealing, the temperature dependence of the thermal conductivity of the 10ScSZ crystals almost coincides with dependence $k(T)$ of crystals measured before annealing. This result is due to the fact that the phase composition of the material consisting mainly of the rhombohedral phase in a mixture with the cubic phase is

changed insignificantly. The practical absence of the changes in the phase composition is well seen from the Raman spectra of the crystals before and after the annealing (Fig. 4).

Before the annealing, the temperature dependences of the thermal conductivity $k(T)$ of the crystals of partially stabilized zirconium dioxide 0.8Sc2YSZ and 0.3Sc2.5YSZ are very weak. Taking into account the high sensitivity of the thermal conductivity to the disturbance of the periodicity of the crystal field at low temperatures, the abovementioned result can be interpreted as a comparatively less substantial demonstration of the processes of phonon scattering in these crystalline materials in spite of the developed twin structure in them [20]. Unlike the case of co-alloyed crystals with high impurity concentrations (11–12 mol %), the thermal conductivities of the crystals with low summary impurity concentrations is markedly higher. Its temperature dependence is close to

Table 2. Thermal conductivity of the study crystals measured at various temperatures

Sample	k , W/(m K)							
	50 K		100 K		200 K		300 K	
	growth	annealing	growth	annealing	growth	annealing	growth	annealing
8ScSZ	1.50	1.28	1.66	1.71	2.16	2.33	2.42	2.67
9ScSZ	1.51	0.81	1.68	1.25	2.12	2.01	2.40	2.36
10ScSZ	0.67	0.70	1.10	1.10	1.77	1.78	2.05	2.06
0.8Sc2YSZ	3.44	2.99	3.36	3.09	3.38	3.48	3.44	3.60
0.3Sc2.5YSZ	2.96	2.69	2.92	2.95	3.12	3.37	3.28	3.48
9Sc2YSZ	1.15	1.143	1.31	1.32	1.87	1.89	2.14	2.17
10Sc2YSZ	1.05	1.027	1.30	1.28	1.83	1.79	2.20	1.99

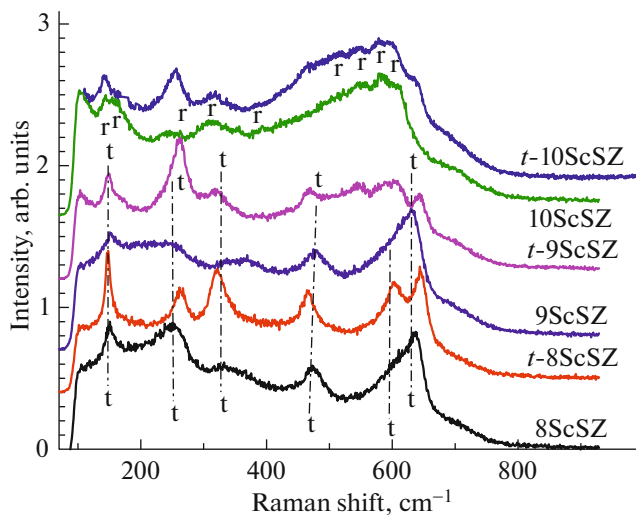


Fig. 4. Raman spectra of the (8–10)ScSZ crystals measured before and after annealing.

$k(T)$ for the 2.5YSZ crystals [22] as k decreases with the increase in temperature and then starts to increase; as a result, the dependence has a minimum shifted to lower temperatures. After the annealing, the thermal conductivities of the crystals are changed insignificantly, but the character of dependence $k(T)$ is changed. At low temperatures 50–100 K, the thermal conductivity is lower than that before annealing, and it increases to the values slightly higher than those before the annealing as the temperature increases to room temperature. The thermal conductivity increases with temperature by analogy with the cases of the crystals with higher impurity concentrations. The character of the temperature dependence is close to $k(T)$ of the compositions with the yttrium oxide content from 3 to 15 mol % [22, 30]. It should be noted that monoclinic phase concentration in the 0.8Sc2YSZ crystals increases, as it is seen from the X-ray diffraction data (Table 2) and the Raman spectra of the crystals after the annealing (Fig. 5).

The thermal conductivity of the monoclinic phase is higher as compared to that of the tetragonal phase [22], and the thermal conductivity of the 0.8Sc2YSZ crystals is higher than that of the tetragonal 0.3Sc2.5YSZ crystals. Clear the annealing of these crystals leads to a decrease in the disorder in the solid solutions due to ordering of oxygen vacancies, as the local environment of zirconium cations approaches the more stable coordination number 7 that is characteristic of the monoclinic ZrO₂ phase due to a partial degradation of the tetragonal phase.

The 9Sc2YSZ and 10Sc2YSZ crystals demonstrate only slight changes in the values of the thermal conductivity, the character of the temperature dependence of the thermal conductivity $k(T)$, and the phase compositions (Figs. 3 and 5, Table 2). Thus, unlike the

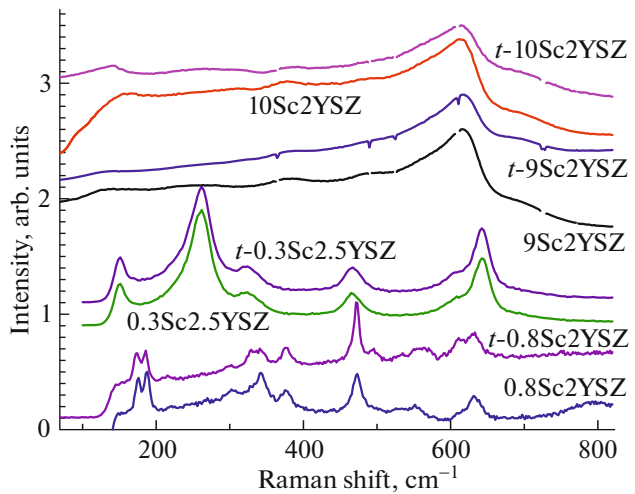


Fig. 5. Raman spectra of the ScYSZ crystals measured before and after annealing.

zirconium dioxide stabilized with scandium oxide, the addition of yttrium oxide along with scandium oxide in the solid solution composition enables us not only to obtain single-phase cubic crystals with the contents of 9–10 mol % Sc₂O₃, but also to increase the stability of its phase composition and the structurally dependent thermal-physical characteristics over entire temperature range. In this case, the lowest thermal conductivity takes place at the scandium oxide content 10 mol % both after the growth and after the annealing.

The electrical conductivity of the crystals, like their thermal conductivity, is a structurally dependent characteristic of the zirconia-based crystals [24, 25, 31]. Figures 6 and 7 show, in the Arrhenius coordinates, the temperature dependences of the electrical conductivity of the single-crystal samples of the ScSZ and ScYSZ solid solutions before and after their annealing at the temperature 1000°C for 400 h.

The electrical conductivity of the 8ScSZ crystals is changed only slightly. The temperature dependences of the electrical conductivity demonstrate well more substantial changes in the electrical conductivities of the (9–10)ScSZ crystals after the annealing. The temperature dependence of the electrical conductivity of the 9ScSZ crystal has a jump related to the formation of the rhombohedral phase in the crystal. The electrical conductivity of the 10ScSZ crystal decreases in the temperature range 823–1173 K, the jump of its temperature dependence is retained, but its value and its temperature range decrease as compared to the electrical conductivity jump after the growth.

The studies of the annealed crystals on an optical microscope showed that, in the (8–10)ScSZ single crystals, the annealing causes the increase in the degree of their inhomogeneity (Fig. 8) due to enlargement of the structural elements and more clear separation of their boundaries in the crystal volume. The

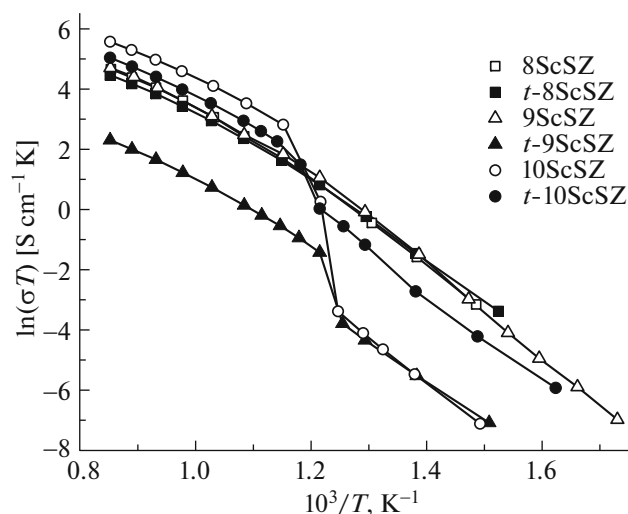


Fig. 6. Temperature dependence of the electrical conductivity of the single-crystal samples of the ScSZ solid solutions measured before and after annealing at 1000°C for 400 h.

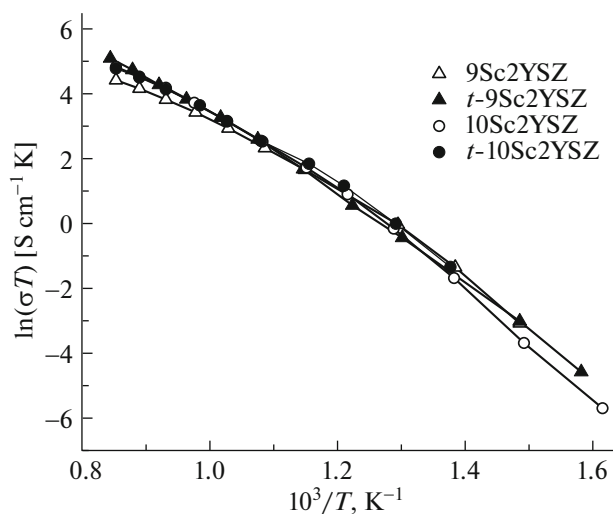


Fig. 7. Temperature dependence of the electrical conductivity of the single-crystal samples of the ScYSZ solid solutions measured before and after annealing at 1000°C for 400 h.

character of the microstructure of the 8CcSZ crystals is retained [24], but the structural elements become coarser and they more clearly seen in an optical microscope (Fig. 8a).

During the annealing, we observe the formation of inclusion groups with more pronounced boundaries. After the annealing, there are semi-transparent regions (Fig. 8c), in which the inclusion elements are coarser and more relief. It is well seen at a larger magnification that these elements are intersected at direct angles (Fig. 8c). In some parts of the crystal, there are twins characteristic of the rhombohedral phase (Fig. 8d) with a small number of fine microcracks. This change in the microstructure of the samples is likely to be due to the separation of the cubic and rhombohedral phases. These features of the microstructure and the existence of the two phases in the 10ScSZ crystal lead to the lowest values of the thermal conductivity among these crystals. On the other hand, the existence of the rhombohedral phase as inclusions in the crystal volume influences weaker the temperature dependence of the electrical conductivity, decreasing the jump and the range of changing the conductivity. It is also possible that the redistribution of scandium between the pseudo-cubic t'' phase and the rhombohedral phase during the annealing also influenced the character of the phase transition in the (9–10)ScSZ crystals.

The annealing leads to ordering of the oxygen vacancies. In the ScSZ crystals, the oxygen vacancies can be arranged either in the first coordination sphere of Zr^{4+} cations giving them the more stable coordination number equal to 7 or placed along-side the Sc^{3+} cations having the ionic radius close to the zirconium ion radius. In the first case, the oxygen mobility

increases and leads to an increase in the electrical conductivity. In the second case, the number of complexes consisting of oppositely charged trivalent cations and vacancies increases, which favors a decrease in the oxygen mobility and a decrease in the electrical conductivity. However, such complexes are capable of decreasing the electrical conductivity. They are the structural defects that cause the phonon scattering and a decrease in the thermal conductivity of the crystals. At temperatures 50–100 K, the thermal conductivity of the 8ScSZ crystals decreases, but the electrical conductivity is changed only slightly, which demonstrates an insignificant prevailing of the ordering by the first variant. At temperatures 50–100 K, the 9ScSZ crystals are characterized by more substantial decrease in the thermal conductivity and a marked decrease in the electrical conductivity. This fact demonstrates an increase in the number of defect complexes. In this case, in addition to the complexes in which a trivalent cation has one oxygen vacancy in its nearest environment, there are the complexes with two oxygen vacancies, which is characteristic of the ordered rhombohedral phase [32]. Moreover, the existence of the rhombohedral phase in the crystal causes the formation of microcracks characteristic of them, which also decreases the thermal conductivity. In the case of the 10ScSZ crystals, the changes in the thermal and electrical conductivities are small after the annealing, since they already contained both types of the defect complexes, and only their proportion was changed.

The annealing of the 9Sc2YSZ and 10Sc2YSZ crystals at 1000°C for 400 h causes only slight changes in the thermal conductivity and the electrical conductivity.

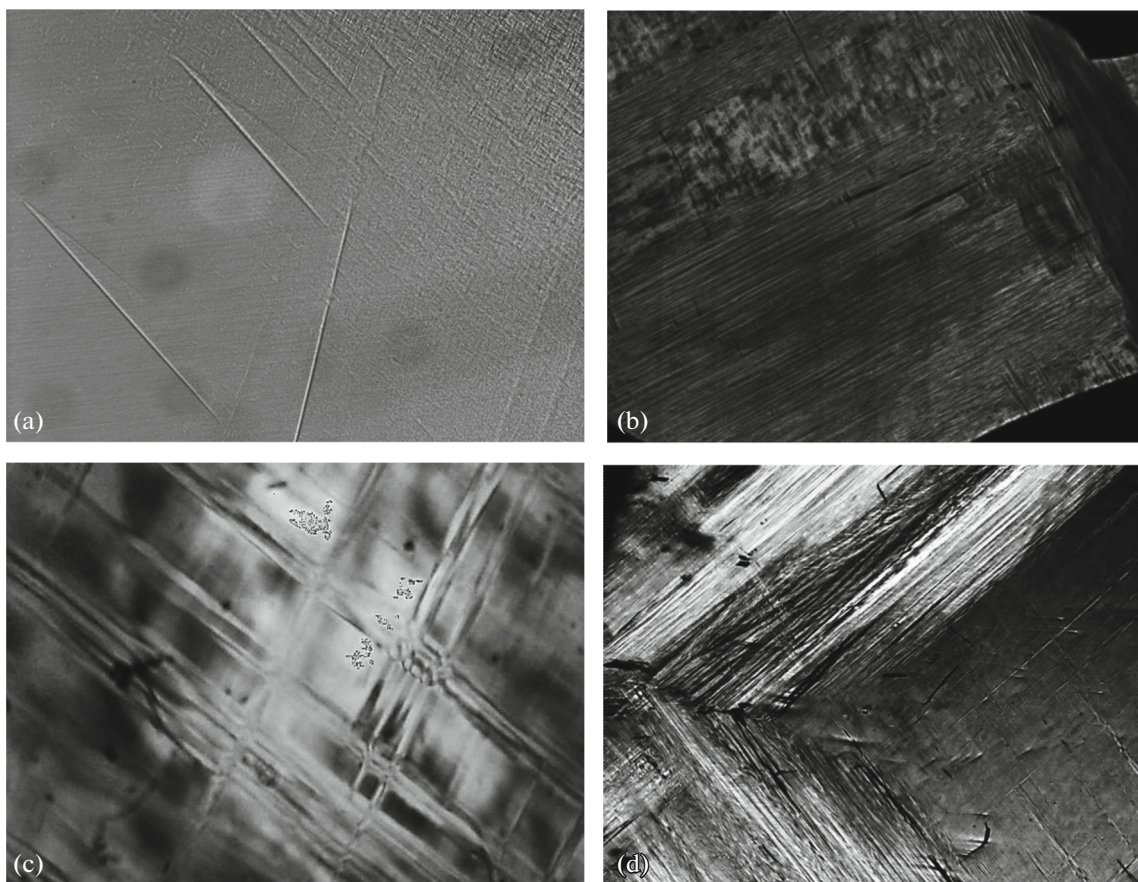


Fig. 8. Microstructures of (a) 8ScSZ, (b) 9ScSZ, and (c, d) 10ScSZ crystals after the annealing.

4. CONCLUSIONS

The effect of heat treatment at 1000°C for 400 h on the thermal conductivity of zirconium dioxide crystals stabilized with scandium oxide $(\text{ZrO}_2)_{1-x}(\text{Sc}_2\text{O}_3)_x$ ($x = 0.08-0.10$) and simultaneously with scandium and yttrium oxides $(\text{ZrO}_2)_{1-x-y}(\text{Sc}_2\text{O}_3)_x(\text{Y}_2\text{O}_3)_y$ ($x = 0.003-0.20$, $y = 0.02-0.025$) has been studied. As a result of the annealing, the thermal conductivities of the 8ScSZ and 9ScSZ crystals decrease in the temperature range 50–100 K and slightly increases at temperatures ~ 300 K. The more substantial changes are observed in the 9ScSZ crystals, which are related to the formation of the rhombohedral phase in them and the change in the microstructure of the crystals. In the 10ScSZ crystals, the change in the proportion of the cubic and rhombohedral phases, the change in the crystal structure, and the appearance of microcracks characteristic of the rhombohedral phase weakly influence the thermal conductivity. The lowest thermal conductivity of this material is mainly determined by the highest content of scandium oxide in the solid solution. The small cation radius of scandium (0.87 \AA) that is close to the cation radius of zirconium (0.84 \AA) leads to a strong disordering of the solid solutions and the formation of the defect complexes consisting of

trivalent cations and oxygen vacancies joined with opposite charges. An increase in the content of the defect complexes with an increase in the Sc_2O_3 concentrations can lead to an increase in the phonon scattering by these complexes, which favors a decrease in the thermal conductivity. The structural changes as a result of the annealing are much more noticeable in the temperature dependence of the electrical conductivity; they lead to a decrease in the electrical conductivity of the crystals.

Unlike zirconium dioxide stabilized only with scandium oxide, an addition of yttrium oxide along with scandium oxide in the solid solution composition enables us to increase the stability of its phase composition and the structurally dependent thermal-physical and electrical-physical characteristics of the compositions under study over entire temperature range. The single-phase cubic 9Sc2YSZ and 10Sc2YSZ crystals demonstrate the maximum stability. The annealing of the partially stabilized crystals changes the shape of the temperature dependence of the thermal conductivity that becomes similar to dependence $k(T)$ of the crystals with 8–15 mol % YSZ with higher concentrations of the alloying oxide. This result is related to the ordering of oxygen vacancies and also to a change in

the phase composition. In the 0.8Sc2YSZ crystals with the high content of scandium oxide, the thermal conductivity is changed due to a partial degradation of the tetragonal phase and the increase in the content of the monoclinic phase with a higher thermal conductivity. In the 0.3Sc2.5YSZ crystals with a lower content of scandium oxide, phase transformations are almost absent, and the ordering of oxygen vacancies plays a main role in the change in the thermal conductivity.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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