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ИССЛЕДОВАНИЕ ВЛИЯНИЯ НАНОРАЗМЕРНЫХ ЧАСТИЦ TiO_2 НА ФИЗИКО-МЕХАНИЧЕСКИЕ СВОЙСТВА, СТРУКТУРУ И ФАЗОВЫЙ СОСТАВ ($BeO + TiO_2$)-КЕРАМИКИ

Людмила Иосифовна Квеглис[1†], Александр Викторович Павлов[2], Алексей Владимирович Джес[3]

[1] Сибирский федеральный университет, пр. Свободный, 79, 660041, Красноярск, Россия
[2] Восточно-Казахстанский университет им. С. Аманжолова, ул. 30-ой Гвардейской дивизии, 34, 070002, Усть-Каменогорск, Республика Казахстан
[3] Восточно-Казахстанский технический университет им. Д. Серикбаева, ул. Протозанова, 69, 070004, Усть-Каменогорск, Республика Казахстан
[1] kveglis@list.ru[†], https://orcid.org/0000-0002-6850-354X
[2] alexandr_pavlov_1988@mail.ru, https://orcid.org/0000-0002-3514-9476
[3] alexdzhes@gmail.com, https://orcid.org/0000-0002-8172-8073

Аннотация. В настоящем исследовании представлены результаты влияния количества нано размерных частиц TiO_2 (0,1-2,0 мас.%) и температуры обжига на физико-механические свойства, структуру и фазовый состав оксидно-бериллиевой керамики состава ($BeO + TiO_2$) изготовленной из порошков микронного размера. Показано, что присутствие наночастиц TiO_2 способствует увеличению плотности спеченной керамики. Данный эффект достигается вследствие взаимопроникновения фаз $BeO – TiO_2$ и увеличения дефектности структуры. Понижение энергии активации процессов обмена мест в зоне границы зерен может быть объяснено связью между диффузией и дефектами строения решетки. Диффузия вдоль границы зерен происходит быстрее, чем в ненарушенной. Присутствие наночастиц также способствует самозалечиванию микропор, что может быть объяснено блокированием наночастицами определенной доли границ раздела между частицами BeO и созданием диффузионного барьера. Инжекция вакансий внутрь кристалла, повышает свободную энергию системы, делает термодинамически невыгодным его рост, в определенном интервале размеров. Как показано в настоящем исследовании, повышение температуры спекания керамики, способствует трансформации кристаллической структуры TiO_2 в более проводящую Ti_3O_5 с орторомбической структурой. Возникновение электропроводящей фазы, как правило, сопровождается поглощением электромагнитного излучения. Синтезированная керамика актуальна для нужд радио-электронной промышленности: электровакуумные приборы СВЧ – ЭВП, усилители, лампы бегущей и обратной волны, клистроны, клистроды, гироприборы; твердотельные приборы СВЧ; модули СВЧ; комплексированные изделия СВЧ с применением в своем составе ЭВП СВЧ, твердотельных дискретных приборов и модулей СВЧ.

Ключевые слова: наночастицы TiO_2, оксид бериллия, диоксид титана, рутил, керамика, физико-механические свойства, кристалл, микроструктура, спекание, кристаллическое строение, фазовый состав.

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Original article

STUDY OF THE EFFECT OF TiO_2 NANOADDITIVES ON THE PHYSICAL AND MECHANICAL PROPERTIES, STRUCTURE AND PHASE COMPOSITION OF ($BeO + TiO_2$)-CERAMICS

Lyudmila I. Kveglis[1†], Alexander V. Pavlov[2], Alexey V. Jess[3]

[1] Siberian Federal University, Svobodny Pr., 79, Krasnoyarsk, 660041, Russia
[2] East Kazakhstan University named after S. Amanzholov, 30th Guards division Str., 34, Ust-Kamenogorsk, 070002, Republic of Kazakhstan
[3] East Kazakhstan Technical University named after D. Serikbayev, Protozanova Str., 69, Ust-Kamenogorsk, 070004, Republic of Kazakhstan
[1] kveglis@list.ru[†], https://orcid.org/0000-0002-6850-354X
[2] alexandr_pavlov_1988@mail.ru, https://orcid.org/0000-0002-3514-9476
[3] alexdzhes@gmail.com, https://orcid.org/0000-0002-8172-8073

Abstract. This study presents the results of the influence of the number of TiO_2 nanosized particles (0.1-2.0 wt.%) and the firing temperature on the physical and mechanical properties, structure and phase composition of beryllium oxide ceramics of the composition ($BeO + TiO_2$) made from powders of micron size. It is shown that the presence of TiO_2 nanoparticles promotes an increase in the density of sintered ceramics. This effect is achieved due to the interpenetration of the $BeO – TiO_2$ phases and an increase in the defectiveness of the structure. The decrease in the activation energy of place exchange processes in the grain boundary zone can be explained by the relationship between diffusion and defects in the lattice structure. Diffusion along the grain boundary occurs faster than in an undisturbed lattice. The presence of nanoparticles also promotes self-healing of micropores, which can be explained by the blocking of a certain fraction of the interfaces between BeO particles by nanoparticles and the creation of a diffusion barrier. Injection of vacancies into the crystal, increases the free energy of the system, makes its growth thermodynamically unfavorable, in a certain range of sizes. As shown in this study, an increase in the sintering temperature of ceramics promotes the transformation of the crystalline structure of TiO_2 into a more conductive Ti_3O_5 with an orthorhombic structure. The onset of an electrically conductive phase is usually accompanied by the absorption of electromagnetic radiation. The synthesized ceramics are relevant for the needs of the radioelectronic industry: microwave electric vacuum devices - EEC, amplifiers, traveling and backward wave tubes, klystrons, klystrodes, gyro devices; solid state microwave devices; microwave modules; integrated microwave products with the use of microwave electronic devices, solid-state discrete devices and microwave modules.

Keywords: TiO_2 nanoparticles, beryllium oxide, titanium dioxide, rutile, ceramics, physical and mechanical properties, crystal, microstructure, sintering, crystal structure, phase composition.

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Introduction

At present, the widespread development of nanotechnologies makes it possible to synthesize nanophase high-temperature ceramics with increased density, thermal conductivity, and special structural and electrophysical properties [1-2]. The creation of an absorbing material of the conductor-dielectric type with specified high values of dielectric losses and electrical conductivity has become a significant step forward in the development of absorbers for high-power electrovacuum devices. This effect is achieved by controlling the properties of ceramics by thermal diffusion of variable valence ions into it and creating a second phase with increased conductivity [3-4]. Titanium in the form of TiO_2 oxide was chosen as an ion capable of changing valence and satisfying a set of requirements necessary for the synthesis of materials with certain technological properties [5-8].

The absorbing properties of titanate ceramics are due to the presence of nonstoichiometric Ti_3O_5 in its composition, the formation of which is facilitated by firing in a hydrogen medium. This reaction proceeds at a temperature of 1200-1600 °C [9].

The most effective titanate absorber at present is commercial ceramics BT-30, composition BeO + 30 wt. % TiO_2, the electrical properties of which can be improved [10-11]. The sintering temperature of serial ceramics is 1530 °C. An increase in the sintering temperature will promote the transformation of the crystal structure of TiO_2 into a more conductive Ti_3O_5 with an orthorhombic structure. Thus, in this work, it is shown that different ratios of the initial crystals of titanium dioxide and beryllium oxide, their size, and ceramic sintering temperature make it possible to control the degree of its reduction, improve the physical and mechanical properties, and thereby operational characteristics [12-15].

Thus, the main purpose of this work is to establish the mechanisms of structure formation in ceramics based on BeO with the addition of micro- and nanocrystalline TiO_2 powders to form a structure with specified parametric characteristics and properties.

Research methods

The initial micron TiO_2 and BeO powders had an average particle size of 10-15 μm. TiO_2 nanopowder was obtained at the installation for the synthesis of nanoparticles and nanopowders by the method of electrical explosion of a conductor, the average particle size is 15-20 nm. Experimental samples were obtained by hot slip casting. Slip masses containing TiO_2 nanoparticles for each batch were prepared on the basis of an organic binder (wax, paraffin, oleic acid) at the rate of LOI (loss on ignition) = 14.5 wt. %.

Burning of the organic binder was carried out in a muffle furnace with a bogie hearth in graphite filling (graphite grains 0.5-1.0 mm) according to a special regime for 93 hours. The results obtained were sintered in a fore-vacuum in a furnace with a carbon heater, followed by reduction annealing in a hydrogen atmosphere.

The microstructure, particle size distribution, and phase analysis of sintered samples were studied using a scanning electron microscope with an energy-dispersive microanalysis attachment JSM-6390LV, 2007. The apparent density value was determined. Method for determining apparent density, open, total and closed porosity, water absorption. Determination of the microhardness of the samples was carried out using the indentation method using the Vickers method. The X-ray phase analysis of the obtained samples was carried out using an X-ray diffractometer X'PertPRO (PANanalytical, 2005).

Results and their discussion

The interaction of BeO and TiO_2 in a wide temperature range indicates that titanium practically does not form substitutional solid solutions with BeO [16]. However, a weak chemical interaction between them, along the phase interfaces, cannot be ruled out during the sintering of ceramics containing TiO_2^{nano}, where other impurities can also be concentrated. In turn, titanium (Ti) with oxygen (O) forms a large number of oxides: TiO_2, Ti_2O_3, Ti_3O_5, the homologous series of oxides Ti_nO_{2n-1}: Ti_4O_7, Ti_5O_9, Ti_6O_{11}, Ti_7O_{13}, Ti_8O_{15}, Ti_9O_{17}, $Ti_{10}O_{19}$ and possibly others. During ceramic sintering in a reducing gas atmosphere contained in the initial mixture, TiO_2 undergoes reduction according to the reaction:

$$TiO_2 \rightarrow Ti_nO_m + O_2.$$

During sintering of such ceramics in a vacuum furnace with a graphite heater (weakly reducing CO_2 environment), under the influence of carbon and high temperature, TiO_2 reduction proceeds according to the reaction [17-18]:

$$TiO_2 + C = TiO + CO.$$

Let’s take a closer look at some physical processes occurring during the sintering of ceramics based on BeO containing TiO_2 micro- and nanoparticles. The presence of nano- and micropores, boundary segregations, a decrease in the surface energy at the boundaries of crystals, the corresponding morphology and uniformity of their size distribution contribute to the thermal stability of the composite as a whole. The injection of vacancies inside the crystal, increasing the free energy of the system, makes its growth in a certain size range thermodynamically unfavorable. Thus, an increase in the sintering temperature of such ceramics will promote a more efficient transformation of the TiO_2 crystal structure into the conductive Ti_3O_5 of the rutile modification. The appearance of an electrically conductive phase and the possible manifestation of the ferromagnetism of nanoparticles is accompanied by the absorption of electromagnetic energy [19-21]. The absorbing properties of (BeO + TiO_2)-ceramics are due to the presence of non-stoichiometric Ti_3O_5 in its composition, the formation of which is facilitated by firing in a hydrogen environment according to the reaction:

$$3TiO_2 + H_2 = Ti_3O_5 + H_2O.$$

Given that the sintering temperature of serial ceramics is 1530 °C, a study was made of the effect of TiO_2 nanoparticles within (0.1-2.0) wt. %, to increase the sintering temperature of such ceramics.

It has been established that when the sintering temperature reaches 1530 °C, the maximum value of the apparent density is reached, corresponding to 3.22 g/cm^3, which does not decrease with an increase in the sintering temperature up to 1550 °C (Fig.1).

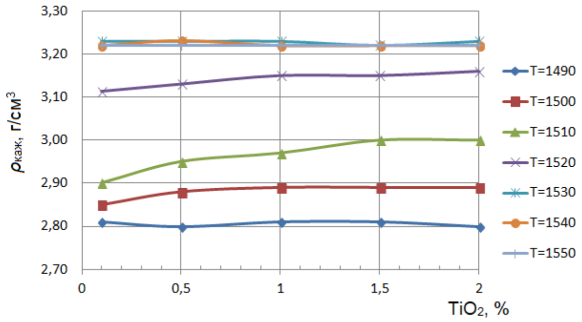

Fig.1. Change in the apparent density of samples depending on the sintering temperature and the amount of TiO_2 nanoparticles

The efficiency of sintering strongly depends on the concentration of TiO_2^{nano}. Two extremes can be distinguished: ^{1}nanoparticles are chemically inert with respect to the micron matrix, as in the case of the BeO + $TiO_2^{\mu m}$ + TiO_2^{nano} system; ^{2}nanoparticles interact with the micron matrix, as in the system $TiO_2^{\mu m}$ + TiO_2^{nano}. It can be seen that during sintering of ceramics with nanoparticles, the sample density at Tc > 1530 °C is not lower than in the case of the initial sample. In the case of TiO_2 nanoparticles that are inert with respect to the BeO matrix, the density of sintered samples does not increase with increasing sintering temperature due to the blocking of grain interfaces. In terms of the combination of properties, the best results are shown by ceramics alloyed with TiO_2 nanoadditives in an amount of 0.5-1.5 wt. %.

The results of studying the microhardness of samples containing nanoparticles in comparison with a serial sample are shown in Fig.2.

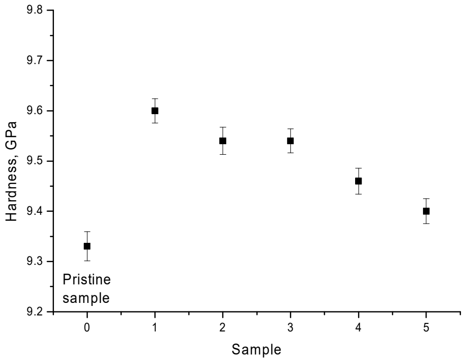

Fig.2. The graph shows the relation of the change in the microhardness of the samples: No. 0 – serial sample (BT-30), No. 1-5 – samples sintered with different concentrations of titanium dioxide nanoparticles (No. 1 – 0.1; No. 2 – 0.5; No. 3 – 1.0; No. 4 – 1.5 and No. 5 – 2.0 wt. %)

The observed improvement in the resistance of the material to destruction is associated with a change in its microstructure under the influence of nanoparticles (Fig.3).

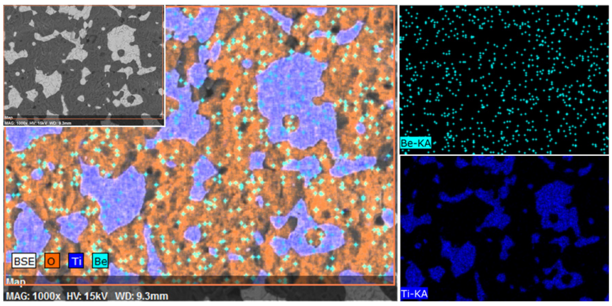

Fig.3. Phase distribution maps in ceramic composition BeO + 28.5% TiO_2 $^{\mu m}$ + 1.5% TiO_2^{nano}, T = 1550 °C

The microstructure of the sample (Figure 3) sintered at a temperature of 1550 °C is represented by relatively large, white fragments of titanium dioxide in a beryllium matrix. Against the background of large, about 10-15 µm TiO_2 structural elements, there are also very small fragments. As can be seen, larger TiO_2 elements have small ~ 1 µm spherical defects formed by grouped TiO_2 nanoparticles, which, during sintering, shrink more than a micron-sized powder. Further, during the sintering process, large fragments of TiO_2 penetrate into the intergranular spaces of BeO through better wettability. Ultimately, the globular pores increase slightly in size and self-heal with a further increase in the sintering temperature of the ceramic. Thus, the mechanism of self-healing is the penetration of the BeO phase into the voids of the TiO_2 structural elements during ceramic shrinkage during sintering. Because the driving force of the sintering process is a decrease in the total surface energy, an increase in the volume fraction of grain boundaries and defect density as a result of nanodispersed additives activates sintering processes. From the distribution maps of chemical elements presented in Fig.3, it can be seen that the addition of TiO_2 nanoparticles leads to a higher density after sintering due to the interpenetration of the TiO_2 and BeO phases, which is due to an increase in the diffusion mobility of atoms due to an increase in the defectiveness of the structure, the proportion of grain boundaries. The presence of nanoparticles contributes to the self-healing of micropores, which is apparently explained by the fact that nanoparticles block a certain fraction of interfaces between BeO particles and create a diffusion barrier. Photographs of the microstructure of serial ceramics in comparison with ceramics doped with nanoparticles are shown in Fig.4.

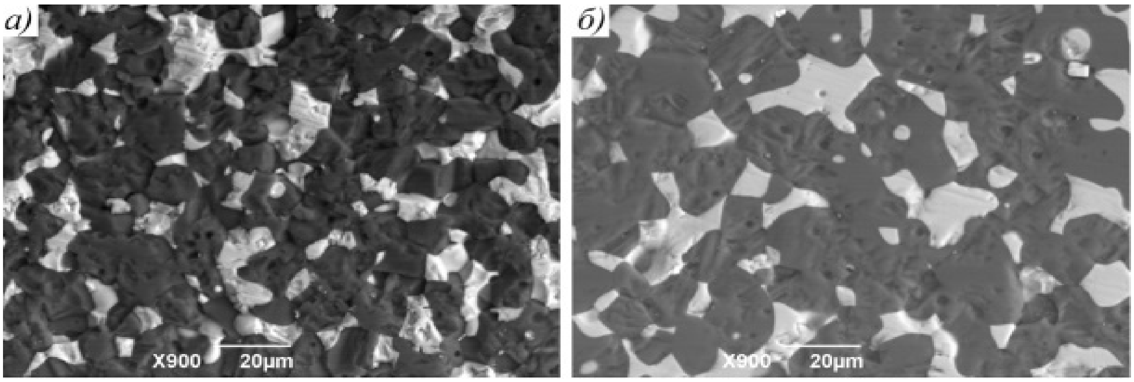

Fig.4. Photographs of the microstructure of $(BeO + TiO_2)$-ceramics, (magnification 900). a – microstructure of serial ceramics sintered at T = 1530 °C; b – $BeO + 28.5\%\ TiO_2^{\mu m} + 1.5\%\ TiO_2^{nano}$, T = 1550 °C

Fig.4a shows the structure of commercial ceramics as a mixture of individual BeO and TiO_2 crystals. Due to the increase in the sintering temperature, the ceramic structure (Fig.4b) looks more ordered, the BeO crystals are much denser packed, and the TiO_2 phase spreads into the intergranular spaces of the BeO micron matrix. In turn, TiO_2 nanoparticles are partially prone to conglomeration with each other and with micron TiO_2 particles (Fig.5).

In correlation to TiO_2, BeO is an inert compound (there is no chemical interaction potential), so the addition of TiO_2 nanoparticles under the same sintering conditions can lead to higher residual porosity. Despite this, the density of $(BeO + TiO_2)$-ceramics with the addition of nanoparticles remains at a high level (≥ 3.2 g/cm^3). On some individual samples, the apparent density value reached 3.33 g/cm^3, which is much higher than the density of serial ceramics.

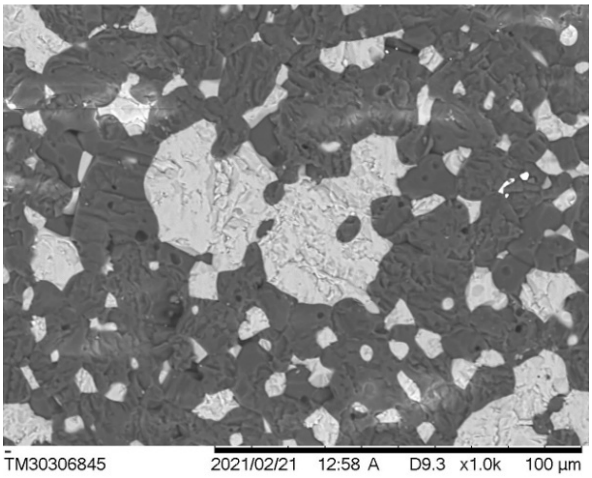

Fig.5. Photograph of the microstructure of the BeO sample + $28.5\%\ TiO_2^{mkm} + 1.5\%\ TiO_2^{nano}$, T = 1550 °C

The obtained diffraction patterns indicate the polycrystalline structure of the samples (Fig.6).

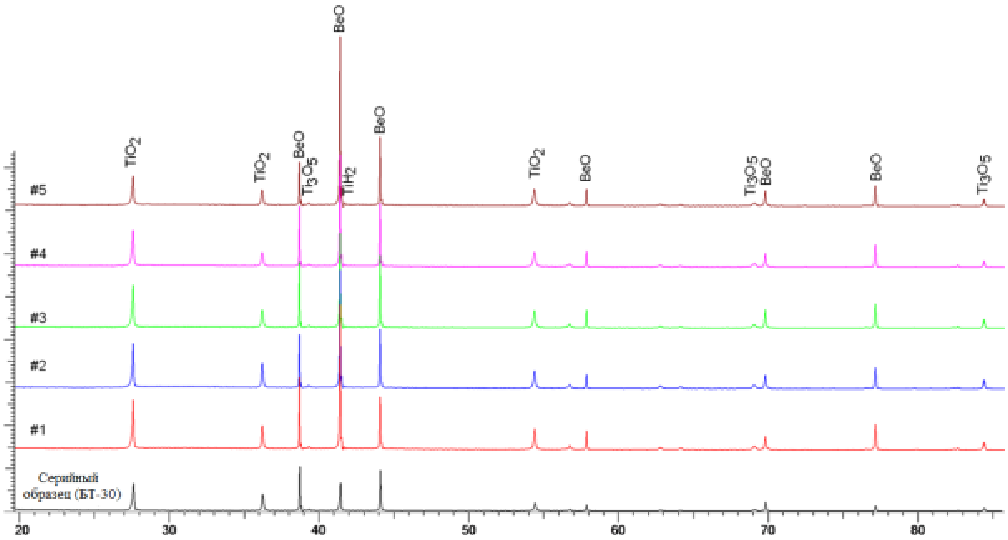

Fig.6. X-ray diffraction patterns of the studied ceramics. BT-30 – serial sample, No. 1-5 – samples sintered at a temperature of 1550 °C with different concentrations of TiO_2 nanoparticles (0.1-2.0) wt. %

Thus, the main contribution to the ceramic structure corresponds to the titanium dioxide (rutile) and beryllium oxide phases. The structure also contains impurity inclusions typical of tetragonal TiH_2 and orthorhombic Ti_3O_5 phases (Table 1), the content of which varies depending on the concentration of nanoparticles. The deviation of the crystal lattice parameters is associated with deformation processes occurring as a result of the formation of ceramics, as well as the presence of impurity phases and solid solutions of substitution and interstitial.

Table 1. Crystal lattice parameters and phase composition of ceramics BeO + 29.0% $TiO_2^{\mu m}$ + 1.0% TiO_2^{nano} sintered at T = 1550 °C in comparison with a serial sample

Phase	Structure type	BeO + 30 % $TiO_2^{\mu m}$		BeO + 29,9 % $TiO_2^{\mu m}$ + 1,0 % TiO_2^{nano}	
		Concentration %	Crystal lattice parameters, Å	Concentration %	Crystal lattice parameters, Å
TiO_2 – rutile	Tetragonal	49.0	a = 4.52321 c = 2.92918 V = 59.93	40.4	a = 4.56844 c = 2.94009 V = 61.36
BeO	Hexagonal	37.1	a = 2.66534 c = 4.33256 V = 26.66	30.0	a = 2.68676 c = 4.35720 V = 27.24
TiH_2	Tetragonal	3.7	a = 3.20137 c = 4.27480 V = 43.81	7.2	a = 3.20576 c = 4.27480 V = 43.93
Ti_3O_5	Orthorhombic	10.2	a = 3.73010 b = 9.44087 c = 9.78548 V = 344.60	22.4	a = 3.73376 b = 9.46864 c = 9.81042 V = 346.83

The main absorbing phase of microwave radiation in the BT-30 material is the semiconductor nonstoichiometric compound Ti_3O_5, which is formed during the reduction of TiO_2 dioxide during the heat treatment of ceramics in a hydrogen medium. Thus, an increase in the sintering temperature of ceramics due to the introduction of TiO_2 nanoparticles in the amount of (0.1-1.5) wt. % contributes to the creation of a reducing atmosphere and special conditions for more efficient transformation of the TiO_2 crystal structure into conducting Ti_3O_5 and TiH_2 compounds.

Chemical reactions that contribute to the formation of the Ti_2O_3 phase during firing:

$$TiO_2 + (CO)_x \rightarrow TiO_{2-x} + (CO_2)_x$$

$$TiO_2 \rightarrow TiO_{2-x} + O_x.$$

Intentional reduction of $Ti^{4+} + e \rightarrow Ti^{3+}$ increases the electrical conductivity of TiO_2. In the (Ti-O)-system, a number of oxide compounds Ti_nO_{2n-1}, where $3 < n < 10$, are known, which have high electrical conductivity. Such an extensive variety of phases leads to the fact that when TiO_2 is heated under various thermodynamic conditions (reducing medium, vacuum), surface and bulk defects appear in its crystal lattice. The main types of intrinsic defects of the TiO_2 crystal lattice are oxygen vacancies, Ti^{3+} and Ti^{4+} interstitial ions, and crystallographic movement of the planes.

Conclusions

The study of the effect of firing temperature on the physical and mechanical properties of ceramics showed that the most effective amount of TiO_2 nanoparticles is their content in the range (0.1-1.5) wt. %, with the same composition it is possible to increase the sintering temperature of ceramics by 30 °C. The addition of TiO_2 nanoparticles leads to an increase in the density and microhardness of the sintered ceramic due to the interpenetration of the TiO_2 and BeO phases, which is due to an increase in the diffusion mobility of atoms due to an increase in the defectiveness of the structure and the proportion of grain boundaries.

The study of the effect of firing temperature on the microstructure of ceramics with the addition of TiO_2 nanoparticles points to the mechanism of self-healing of micropores by the penetration of the

TiO_2 phase into the voids of BeO intergranular spaces during ceramic shrinkage during sintering. According to XRD data, an increase in the sintering temperature of ceramics containing nanoparticles (0.1-1.5) wt. % TiO_2^{nano} up to 1550 °C contributes to the transformation of the crystal structure of TiO_2 into a more conductive Ti_3O_5 with an orthorhombic structure, which may be accompanied by the absorption of electromagnetic radiation.

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Information about the authors

L. I. Kveglis – Doctor of Physical and Mathematical Sciences, Professor of the Siberian Federal University.

A. V. Pavlov – Lecturer, Department of Physics and Technology of the East Kazakhstan University named after S. Amanzholova.

A. V. Jess – Candidate of Technical Sciences, Researcher at the «VERITAS» Center, East Kazakhstan Technical University named after D. Serikbayev.

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Информация об авторах

Л. И. Квеглис – доктор физико-математических наук, профессор Сибирского Федерального университета.

А. В. Павлов – преподаватель кафедры физики и технологий Восточно-Казахстанского университета им. С. Аманжолова.

А. В. Джес – кандидат технических наук, научный сотрудник центра «VERITAS», Восточно-Казахстанский технический университет имени Д. Серикбаева.

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