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S. M. LOGVINKOV, O. M. BORYSENKO, N. P. KLOCHKO, H. M. SHABANOVA, V. M. SHUMEIKO

QUESTION OF THE ZrO_2 – TiO_2 PHASE DIAGRAM

Ceramic materials based on zirconium dioxide (ZrO_2) occupy leading positions among modern structural and functional materials due to their unique physical and chemical properties. These properties are due to the possibility of controlled stabilization of polymorphic modifications of ZrO_2 (monoclinic, tetragonal and cubic). Partially stabilized zirconia exhibits high chemical inertness, low thermal conductivity, exceptional corrosion resistance and thermal shock resistance. These properties make ZrO_2-based ceramics promising for use in a wide range of fields, including biomedical, electronic, structural and functional ceramics, as well as abrasives, refractories and insulation materials. One of the key problems when using pure ZrO_2 is the phase transition from the tetragonal to the monoclinic modification, accompanied by a significant change in the volume of the crystal lattice, which can lead to the destruction of the material. To prevent this transition, modifiers are used that form solid solutions with tetragonal ZrO_2, providing a metastable state due to distortions in the crystal structure. Among such modifiers, titanium dioxide (TiO_2) occupies a special place. Joint doping of ZrO_2 with TiO_2 allows achieving specific effects, especially in the field of electroceramics, where unique dielectric properties of finished materials based on these oxides are manifested, which makes the ZrO_2 – TiO_2 system an object of increased interest for researchers and engineers. The phase diagram of the ZrO_2 – TiO_2 system has been studied since the 1950s, and during this time it has undergone significant refinements. Modern research continues to improve it, but the use of a small scale to display the full diagram over the entire temperature range has led to graphical inaccuracies, which complicates its application in technological practice. In this paper, a comprehensive analysis of the phase diagrams is performed, based on the generalization of data from various studies. This made it possible to identify the most reliable and reproducible elements of the phase structure of the system. To improve the ease of interpretation and practical use, the diagram was conditionally divided into two temperature ranges: low-temperature (800 – 1600 °C) and high-temperature (1600 – 2400 °C). This division facilitates the understanding of phase equilibria and their dependence on temperature and composition, which is critically important for optimizing the synthesis of materials in this system. The obtained data on the phase structure of the ZrO_2 – TiO_2 system create the basis for the targeted synthesis of ceramic materials with a given phase composition and performance characteristics.

Keywords: zirconium dioxide, titanium dioxide, phase equilibria, modification, solid solutions, ceramics, phase diagram, synthesis of materials.

С. М. ЛОГВІНКОВ, О. М. БОРИСЕНКО, Н. П. КЛОЧКО, Г. М.ШАБАНОВА, В. М. ШУМЕЙКО

ДО ПИТАННЯ ПРО ДІАГРАМУ СТАНУ ZrO_2 – TiO_2

Керамічні матеріали на основі діоксиду цирконію (ZrO_2) займають лідируючі позиції серед сучасних конструкційних та функціональних матеріалів завдяки своїм унікальним фізико-хімічним властивостям. Ці властивості обумовлені можливістю контрольованої стабілізації поліморфних модифікацій ZrO_2 (моноклінної, тетрагональної та кубічної). Частково стабілізований діоксид цирконію демонструє високу хімічну інертність, низьку теплопровідність, виняткову корозійну стійкість та стійкість до термічних ударів. Ці якості роблять кераміку на основі ZrO_2 перспективною для застосування в широкому спектрі областей, включаючи біомедичну, електронну, конструкційну та функціональну кераміку, а також як абразивні, вогнетривкі та ізоляційні матеріали. Однією з ключових проблем при використанні чистого ZrO_2 є фазовий перехід з тетрагональної в моноклінну модифікацію, що супроводжується значною зміною об'єму кристалічних ґрат, що може призводити до руйнування матеріалу. Для запобігання цьому переходу застосовуються модифікатори, які утворюють тверді розчини з тетрагональним ZrO_2, забезпечуючи метастабільний стан за рахунок виникнення дефектів у кристалічній структурі. Серед таких модифікаторів особливу увагу займає діоксид титану (TiO_2). Спільне допування ZrO_2 з TiO_2 дозволяє досягти специфічних ефектів, особливо в галузі електрокераміки, де проявляються унікальні діелектричні властивості готових матеріалів на основі даних оксидів, що робить систему ZrO_2 – TiO_2 об'єктом підвищеного інтересу для дослідників та інженерів. Діаграма стану системи ZrO_2 – TiO_2 вивчалася з 50-х років, і за цей час вона зазнала значних уточнень. Сучасні дослідження продовжують удосконалювати її, проте використання дрібного масштабу для відображення повної діаграми у всьому температурному діапазоні призвело до графічної неточності, що ускладнює її застосування у технологічній практиці. У даній роботі проведено всебічний аналіз діаграм стану, заснований на узагальненні даних із різних досліджень. Це дозволило виділити найбільш достовірні та відтворювані елементи фазової будови системи. Для підвищення зручності інтерпретації та практичного використання діаграма була умовно поділена на два температурні діапазони: низькотемпературний (800 – 1600 °C) та високотемпературний (1600 – 2400 °C). Такий поділ полегшує розуміння фазових рівноваг та їх залежності від температури та складу, що критично важливо для оптимізації процесів синтезу матеріалів у даній системі. Отримані дані про фазову будову системи ZrO_2 – TiO_2 створюють основу для спрямованого синтезу керамічних матеріалів із заданим фазовим складом та експлуатаційними характеристиками.

Ключові слова: діоксид цирконію, діоксид титану, фазові рівноваги, модифікація, тверді розчини, кераміка, діаграма стану, синтез матеріалів

Introduction. Zirconium dioxide (ZrO_2) ceramics occupy an important place among modern structural and functional materials [1]. This is due to the possibility of controlled stabilization of various polymorphic modifications of ZrO_2, which allows controlling the structure and properties. Zirconium dioxide has a number of outstanding properties, including high mechanical strength, elastic modulus, hardness, fracture toughness, corrosion and wear resistance, good tribological properties, and high-temperature ionic conductivity.

Ceramics obtained from partially stabilized ZrO_2 exhibit excellent physicochemical properties such as chemical inertness, low thermal conductivity, high corrosion resistance and excellent thermal shock resistance [1, 2]. Due to these properties, ceramics obtained from partially stabilized ZrO_2 are considered as a promising replacement for pure ZrO_2 in the field of high-performance materials, including electronic, functional, biomedical and structural ceramics [3–5].

In addition, ZrO_2-based ceramics have found wide application as abrasive, refractory and insulating materials [6, 7]. Before the appearance of this type of ceramics, pure ZrO_2 ceramics occupied a dominant position in the market. However, its further use has been limited due to

less stable performance characteristics. One of the main reasons for this is the significant volumetric changes during phase transitions caused by temperature fluctuations during the manufacturing process, which leads to internal stresses and the formation of destructive defects. These factors negatively affect the physical properties of pure ZrO_2 and significantly reduce its potential for use as a structural and functional material [8, 9].

When producing ceramic materials for various purposes (cutters, low-temperature dielectrics, high-temperature oxygen concentration sensors in gas mixtures, etc.), zirconium dioxide needs to be modified due to differences in the volumes of the elementary crystal lattices of its polymorphic modifications (monoclinic, tetragonal and cubic). The main danger is the modification transition of the tetragonal modification to the monoclinic one, which is accompanied by a significant (more than 6 %) expansion of the material and leads to its destruction upon cooling. Therefore, they strive to prevent the phase transition to the monoclinic modification using various additives – modifiers capable of forming solid solutions with tetragonal ZrO_2.

At present, many oxides (CrO, MgO, Y_2O_3, CeO_2) are known and have already become traditionally used, capable of forming solid solutions with ZrO_2 and providing a metastable state due to certain distortions in the tetragonal structure [10–15].

Zirconium dioxide – monoclinic modification (baddeleyite) according to the symmetry of the crystal lattice belongs to the space group $P2_1/c$, which changes during the phase transition (about 1140 °C) to tetragonal ($P4_2/mmc$), stable up to 2333 °C with a subsequent change to cubic (Fm3m). At high pressures, the existence of two phases of ZrO_2 with an orthorhombic crystal lattice was established [12].

Crystallochemical regularities in ZrO_2 (hereinafter, the abbreviations t – tetragonal, m – monoclinic and c – cubic modification of ZrO_2 will be used) determine the specifics of the formation of solid solutions and the possibilities of regulating the degree of stabilization. Divalent oxides (CaO, MgO) have a relatively low solubility in t-ZrO_2, which limits the achievable level of stabilization of solid solutions. Trivalent oxides (Y_2O_3, Gd_2O_3, Ga_2O_3) are capable of better solid-phase solubility in t-ZrO_2 due to filling anion vacancies in the crystal structure. Tetravalent oxides (CeO_2, GeO_2, TiO_2) with solid-phase solubility in t-ZrO_2 provide significant concentrations due to isovalent cation substitution, and mixed complex oxides based on tri- and pentavalent elements ($YTaO_4$, $YNbO_4$) are capable of exchange-compensatory heterovalent isomorphism and also provide high concentrations in the resulting solid solutions.

For economic reasons, biocompatibility conditions and the crystalline proximity of the parameters of the crystal lattices of t-ZrO_2 and rutile, TiO_2 is often used as a modifier. An additional advantage, especially in the manufacture of electroceramics, is the manifestation of specific effects during the combined doping of t-ZrO_2, in particular, MgO and TiO_2. In addition, researchers [12] have expanded the range of possible TiO_2 concentrations in homogeneous solid solutions based on t-ZrO_2 to 25 mol. %. In this case, a significant role in the stabilization of t-ZrO_2 is given to the formation of clusters close to the composition of Zr_3TiO_8, which play a specific role in the processes of ordering – softening of solid solutions with the formation of domains. This circumstance complements the relevance of the analysis of the ZrO_2 – TiO_2 system.

Modern data on the phase diagram of ZrO_2 – TiO_2. The phase diagram of ZrO_2 – TiO_2 has been studied since the 1950s by many researchers and has undergone significant refinements to date. The issues of the existence of $ZrTiO_4$, $ZrTi_2O_6$, $Zr_5Ti_7O_{24}$ compounds, the temperature ranges of their thermodynamic stability, deviations of srilankite ($ZrTi_2O_6$) from the stoichiometric composition, etc. have been discussed for a long time. The relevance of research into materials of the ZrO_2 – TiO_2 system is due to their extensive use in various fields of technology and modern technological processes, especially with the use of ultra-high-frequency devices. Integrated information on the achievements and problems in the studies of the ZrO_2 – TiO_2 phase diagram can be obtained from the results of works [16, 17], in which the controversial compound $Zr_5Ti_7O_{24}$ is no longer considered due to the absence of confirmation of its synthesis by the sol-gel method since 1998.

In the study [16], the emphasis was placed on examining $ZrTi_2O_6$ in the form of a solid solution (the formula is $(Zr,Ti)_2O_4$), capable of ordering-disordering the crystal lattice. From this point of view, the experimental results of phase changes in mixtures of initial oxides in the presence of fluxing additives (CuO, mixtures of $LiMoO_4$ and MoO_3 in a mass ratio of 1:1.6) are considered. The starting mixtures were pressed into platinum capsules and fired for a long time (in particular, 96 hours at 800 °C) in a furnace with a controlled temperature of ±1 °C, and then cooled to room temperature for 1 minute: experiments were carried out at 800 – 1650 °C, the majority of experiments were carried out in the range of 1000 – 1200 °C, which is most important for controlling the technological parameters of obtaining ceramic materials.

According to the results of experiments in the solid-phase solution after firing at 800 °C, the TiO_2 concentration was 64.9 mol. % (in srilankite the TiO_2 content is 66.7%), and after firing at 1060 °C, the TiO_2 concentration decreased to 60.4 wt. %, which is explained by the process of ordering-disordering of the $(Zr,Ti)_2O_4$ cations. In the temperature range of 1060 – 1160 °C, a sudden change in the parameter “b” of the crystal lattice of the solid solution was noted (at 1160 °C, the TiO_2 content was 51.5 wt.%), and the closest correspondence of the composition of the solid solution to the $ZrTiO_4$ compound was noted after firing at 1080 °C (49.0 wt. % TiO_2). Above 1160 °C, the prevalence of disordering processes (more uniform distribution of titanium and zirconium cations in the corresponding positions of the crystal lattice) was noted, which is reflected by the formation of two steeply ascending boundaries of the crystallization fields from the $ZrTiO_4$ composition on the

constructed phase diagram in the studied temperature range (Fig. 1).

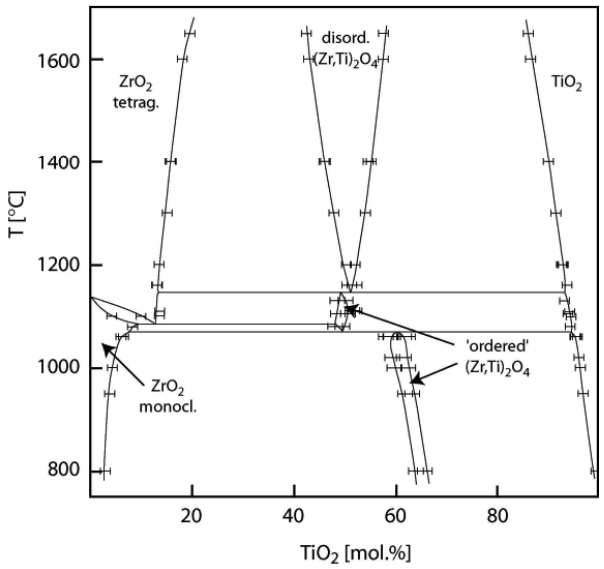


Figure 1 – The ZrO_2 – TiO_2 phase diagram at atmospheric pressure based on experimental data [16]

The diagram contains a tie line connecting the points of the solid solution compositions that participate in the eutectoid interaction at about 1080 °C. The experimental results do not substantiate the reliable presence of this tie line, which is within the permissible temperature deviations of the tie line at ~ 1060 °C, corresponding to the peritectoid interaction.

The reasons for the deviations of the solid solution compositions in the range of 1060 – 1160 °C from the stoichiometry of the $ZrTiO_4$ composition were not analysed; they may be due to the use of fluxes in the experiments and errors in determining the TiO_2 concentrations. The noted reasons, along with a significant manifestation of kinetic inhibition of lower-temperature processes in the studied samples, could also cause significantly greater deviations of solid solutions below 1060 °C from the $ZrTi_2O_6$ composition.

The authors of [17] correct some temperatures of phase equilibria in the ZrO_2 – TiO_2 system in the range of 1000 – 1500 °C, as well as the compositions of solid solutions. However, the $ZrTi_2O_4$ compound is displayed with a stoichiometric composition existing in the low-temperature region up to ~ 1170 °C, when it disproportionates according to the mechanism of peritectoid reaction with the formation of two solid solutions: based on ZrO_2 and $ZrTiO_4$. In this case, $ZrTi_2O_4$ is designated as α′-$ZrTiO_4$, and a solid solution with a composition close to $ZrTiO_4$ is designated as α-$ZrTiO_4$. These designations do not bring additional information to the essence of the processes occurring, as does the designation of the region of solid solutions based on $ZrTiO_4$ – β-$(Zr_xTi_{1-x})_2O_4$.

The temperature dependence of the heat capacity for $ZrTiO_4$ established in [15] made it possible to use thermodynamic calculations to predict the parameters of phase equilibria in the system and to conduct confirmatory experiments, including in the high-temperature region up to 1850 °C.

In the phase diagram of ZrO_2 – TiO_2 (Fig. 2), the structural elements of the low-temperature region are close to those in [16].

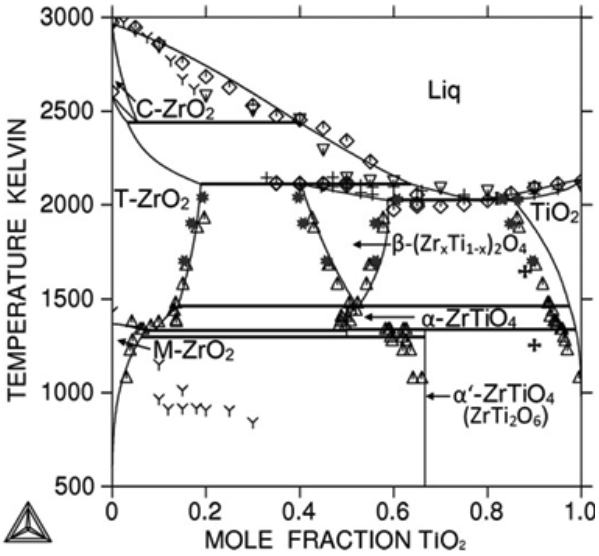


Figure 2 – Calculated phase diagram of the ZrO_2 – TiO_2 system [17]

At the same time, a small scale was chosen to display the entire temperature range of the state diagram, which introduced some graphical inaccuracies and made it inconvenient to use it in technological practice.

Results and discussion. The results of the analysis of the phase diagrams of ZrO_2 – TiO_2 according to the studies [16–19] allow us to generalize them with the preservation of the most reliable elements of the structure and in a scale of the temperature axis convenient for technological practice, dividing it into two ranges of values: low-temperature (800 – 1600 °C) and high-temperature
(1600 – 2400 °C) – Fig. 3a and 3b, respectively. In the temperature range of 1060–1084 °C, the only tie line is preserved at 1070 °C, which combines two processes:

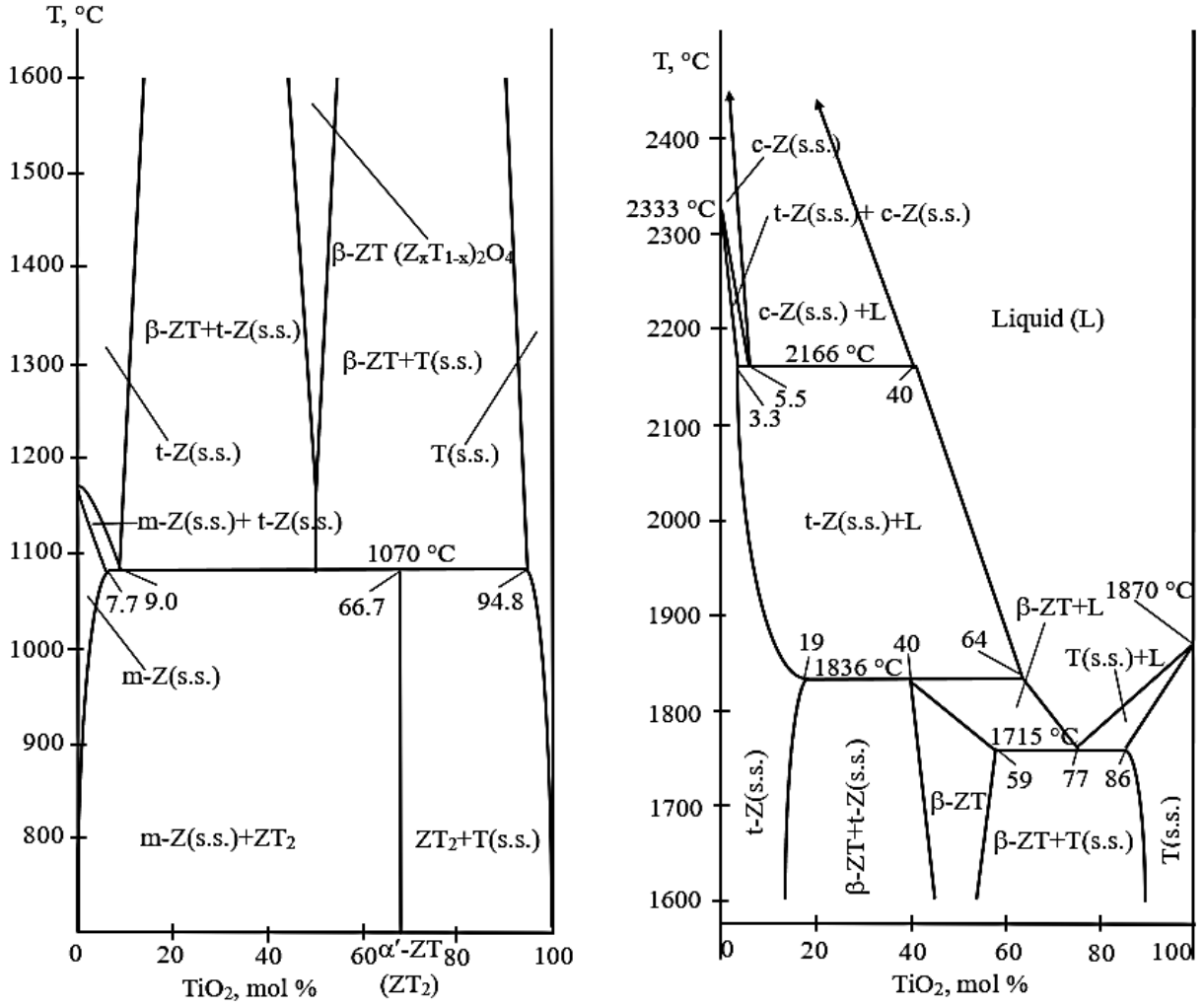
1. disproportionation of $ZrTi_2O_6$ (α′-$ZrTiO_4$ – here and in Fig. 3a, b the designations are retained according to [17]) by the eutectoid mechanism

$$ZrTi_2O_6 \leftrightarrow \alpha\text{-}ZrTiO_4 + \text{s.s. } TiO_2 \text{ (94.8 mol. \% } TiO_2);$$

2. eutectoid interaction by mechanism:

$$\alpha\text{-}ZrTiO_4 + \text{m-}ZrO_2 \text{ (7.7 mol. \% } TiO_2) \leftrightarrow \text{t-}ZrO_2 \text{ (9.0 mol. \% } TiO_2).$$

The konnoda (about 1129 °C according to calculations [17]) is not preserved due to its lack of information, since in fact it only reflects the temperature of the onset of formation of a homogeneous solid solution of β-$ZrTiO_4$ ($(Zr_xTi_{1-x})_2O_4$) based on α-$ZrTiO_4$ with an excess or deficiency of TiO_2. The boundary curves formed above this temperature highlight the region of homogeneous solid solutions of β-$ZrTiO_4$ and simultaneously determine the coexisting compositions based on t-ZrO_2 and TiO_2, respectively (Fig. 3a).



a b

Figure 3 – Phase diagram of the ZrO_2 – TiO_2 system:
a – low-temperature region (800 – 1600 °C),
b – high-temperature region (1600 – 2400 °C)

In the high-temperature region of the ZrO_2 – TiO_2 phase diagram (Fig. 3b) at 1751 °C, a single eutectic point is noted (TiO_2 content 77 mol.%), when interaction occurs between a homogeneous solid solution of β-$ZrTiO_4$ (59 mol. % TiO_2) and a solid solution based on TiO_2 (86 mol. % TiO_2) with the formation of a melt.

At the eutectic temperature, β-$ZrTiO_4$ and the solid solution based on TiO_2 have the maximum concentration of TiO_2 in their compositions. The konnoda at 1836 °C was adopted based on the calculated results [17] and reflects the peritectic decomposition of the β-$ZrTiO_4$ solid solution into a solid solution of t-ZrO_2 (19 mol. % TiO_2) and a melt containing 64 mol. % TiO_2. The temperature of 2166 °C was taken as the minimum temperature for the possible formation of c-ZrO_2 according to the peritectic mechanism of interaction:

s.s. t-ZrO_2 (3.3 mol. % TiO_2) + liquid (40.0 mol. % TiO_2) ↔ c-ZrO_2 (5.5 мол. % TiO_2).

The corresponding tie line unites the above-mentioned points of the compositions at 2166 °C and this temperature determines the maximum solubility of TiO_2 with c-ZrO_2 – 5.5 mol. %. For better visual perception, the boundary lines in Fig. 3b above 2490 °C are not drawn, since in real technological practice higher

temperatures are extremely rarely used, and if necessary, they can be easily extrapolated to the melting point of zirconium dioxide without any significant loss in the accuracy of the construction. The general appearance of the phase diagram is also not difficult to visualize by combining Fig. 3a and 3b along the 1600 °C isotherm.

Conclusions. The results of the analysis of the phase diagrams of the ZrO_2 – TiO_2 system, performed on the basis of data presented in different studies, allowed us to generalize them while preserving the most reliable and stably reproducible elements of the phase structure. For the sake of ease of interpretation and subsequent use of the phase diagram of the ZrO_2 – TiO_2 system, the temperature range was conditionally divided into two characteristic intervals: low-temperature (800 – 1600°C) and high-temperature (1600 – 2400°C).

Thus, the obtained data on the structure of the ZrO_2 – TiO_2 system will be used for the targeted synthesis of functional ceramic materials with specified phase composition and operational characteristics. This allows the development of materials with improved mechanical, thermal and electrical properties adapted for specific applications.

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Відомості про авторів / About the Authors

Логвінков Сергій Михайлович (Logvinkov Sergey) – доктор технічних наук, професор, професор кафедри охорони праці та безпеки життєдіяльності, Харківський національний університет міського господарства імені О. М. Бекетова, м. Харків, Україна; ORCID: https://orcid.org/0000-0001-5957-2386 ; e-mail: Sergii.Logvinkov@m.hneu.edu.ua

Борисенко Оксана Миколаївна (Borysenko Oksana) – доктор технічних наук, професор, професор кафедри технології кераміки, вогнетривів, скла та емалей, Національний технічний університет «Харківський політехнічний інститут», м. Харків, Україна; ORCID: https://orcid.org/0000-0002-2746-6797 ; e-mail: oksana.borysenko@khpi.edu.ua

Клочко Наталя Петрівна (Klochko Natalja) – кандидат технічних наук, доцент, доцент кафедри мікро- та наноелектроніки, Національний технічний університет «Харківський політехнічний інститут», м. Харків, Україна; ORCID: https://orcid.org/0000-0002-0852-4373 ; e-mail: klochko.np16@gmail.com

Шабанова Галина Миколаївна (Shabanova Halyna) – доктор технічних наук, професор, професор кафедри технології кераміки, вогнетривів, скла та емалей, Національний технічний університет «Харківський політехнічний інститут», м. Харків, Україна; ORCID: https://orcid.org/0000-0001-7204-940X ; e-mail:gala-shabanova@ukr.net

Шумейко Віта Миколаївна (Shumeiko Vita) – кандидат технічних наук, старший науковий співробітник кафедри технології кераміки, вогнетривів, скла та емалей, Національний технічний університет «Харківський політехнічний інститут», м. Харків, Україна; ORCID: https://orcid.org/0000-0003-0567-0946 ; e-mail: shum-vita@ukr.net