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STUDY OF THE STRUCTURAL EVOLUTION AND STRENGTHENING OF Li_2TiO_3 -BASED CERAMICS DOPED WITH LiNbO_3

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Ceramic materials based on lithium metatitanate (Li_2TiO_3) modified with lithium niobate (LiNbO_3) in the concentration range of 0.05–0.25 M were studied. The synthesis was carried out by mechanical activation in a planetary mill at grinding speeds of 150...600 rpm with a step of 50 rpm, followed by thermal stabilization at 700 °C for 5 hours. The objective of the work was to determine the relationship between the dopant concentration, the energy of mechanical processing and the structural and mechanical properties of the obtained ceramics.

The phase composition and structural parameters were studied by X-ray diffraction, and the morphological features of the ceramics were studied using scanning electron microscopy. The formation of a two-phase system, including the monoclinic Li_2TiO_3 phase and the rhombohedral LiNbO_3 phase, can be seen. The increase in the dopant content occurs simultaneously with changes in the parameters of the crystal lattice and a decrease in the size of the coherent scattering regions. SEM analysis showed pronounced changes in the morphology of ceramics upon the addition of LiNbO_3 , depending on the dopant concentration. Increasing the mechanical activation rate to 600 rpm and increasing the alloying additive concentration to 0.25 M lead to the creation of a fine-grained composite microstructure, where an improvement in the strength characteristics is observed compared to the unalloyed material.

The obtained data show that the combination of changing the LiNbO_3 content and mechanical activation parameters is one of the most effective tools for controlling the phases and strength of lithium-containing ceramic materials, which opens up enormous opportunities for optimization in high-temperature energy applications.

Key words: *lithium-containing ceramics; lithium metatitanate; lithium niobate; X-ray phase analysis; crystal structure.*

INTRODUCTION

The need for stable materials for tritium production is leading to increased attention to lithium-containing ceramics for inclusion in thermonuclear reactor blanket assemblies [1-2]. Among the promising breeder compounds, lithium metatitanate (Li_2TiO_3) occupies a special place, possessing high thermal stability, chemical inertness and good compatibility with the structural materials of reactor systems [3-4]. At the same time, Li_2TiO_3 is characterized by relatively low mechanical strength, grain growth during heat treatment, and brittleness, which limits its operation and requires targeted modification of the structure [5-6].

Lithium niobate (LiNbO_3), widely used in piezoelectric, acousto-optic and nonlinear optical devices due to its pronounced ferroelectric properties, is considered as a promising modifying additive [7-8]. The material is characterized by high chemical resistance and structural stability, but is also brittle and sensitive to microstructural defects [9-10].

The difference in the parameters of the crystal lattices of Li_2TiO_3 and LiNbO_3 contributes to the formation of internal stresses and interphase boundaries, which, when

doping and mechanical activation are applied, provide dispersion strengthening and inhibition of grain growth [10-12]. Changing the speed of planetary grinding allows one to control the degree of particle grinding, the accumulation of defects and the distribution of the dispersed phase in the matrix, thereby most likely increasing the strengthening of interphase grain boundaries and crack resistance [13-15].

In this work, ceramics based on Li_2TiO_3 , modified with LiNbO_3 additives in the concentration range up to 0.25 M, obtained by mechanochemical synthesis, were investigated. The aim of the study was to establish the patterns of the influence of lithium niobate concentration and mechanical activation modes on the phase composition, structural parameters and mechanical properties of ceramics. The research aims to develop reinforced lithium-containing ceramics with enhanced mechanical stability, thereby improving the reliability of breeder materials for fusion power systems. The scientific novelty of the study is to determine the relationship between the concentration of LiNbO_3 , synthesis conditions, structural evolution and mechanical characteristics of the obtained Li_2TiO_3 – LiNbO_3 ceramics.

METHODS

Ceramics based on lithium metatitanate (Li_2TiO_3 powder) with the addition of a dopant in the form of lithium niobate (LiNbO_3 powder) were selected as the initial samples. The dopant concentration ranged from 0.05 to 0.25 M in the composition. All starting components were purchased from Sigma Aldrich, with a stated chemical purity of approximately 99.95%. Mechanochemical solid-phase synthesis for the preparation of two-phase lithium-containing ceramics was used to mix the starting components and obtain a homogeneous and isotropic distribution of lithium-containing ceramics. Mixing was carried out in a PULVERISETTE 6 planetary mill (Fritsch, Berlin, Germany), at grinding speeds from 150 to 600 rpm in 50 rpm increments to obtain a finely dispersed fraction to initiate dispersion strengthening processes.

The resulting powders were then pressed into tablets of 10 mm diameter and 1 mm thickness, which were then used for structural characterization and mechanical strength for resistance to external influences under single compression. Tablet pressing was carried out using the standard method at a pressure of 250 MPa in a cylindrical press mold, with a pressing time of 30 minutes.

The pressed tablets were annealed at 700 °C for 5 hours in a Nabertherm LE 4/11/R6 furnace (Nabertherm, Lilienthal, Germany), followed by gradual cooling under furnace conditions until they reached room temperature. It was found that the addition of a dopant lithium niobate in ceramics based on lithium metatitanate results in an increase in the content of finely dispersed fraction in the composition, from which it follows that the grinding processes under mechanical impact action depend not only on the grinding speed but also on concentration of impurities, the presence of which leads to an intensification fragmentation processes of the main phase, as a result of

which the grinding processes can be carried out at lower grinding speeds.

Microstructural characteristics were determined using scanning electron microscopy with a Phenom ProX microscope (Thermo Fisher Scientific, Eindhoven, Netherlands). The phase composition and structural parameters of the studied samples were estimated depending on variations in the percentage content of the components in the composition of the materials studied by X-ray diffraction method. The diffraction patterns were recorded using a D8 ADVANCE ECO X-ray diffractometer (Bruker, Karlsruhe, Germany) in the angular range of $2\theta = 20\text{--}80^\circ$ in the Bragg-Brentano geometry, with a diffraction pattern recording time of 1 s at one point and a step of 0.03° . The interpretation of diffraction patterns was carried out using the DiffracEVA v.4.2 software; the determination of the phase composition, as well as the refinement of the parameters for all identified phases, was performed using card values from the PDF-2 database.

The determination of resistance to external influences, in particular resistance to single compression, was carried out using the testing machine LFM-L 10kH (Walter + Bai AG, Lönigen, Switzerland). The experiments were carried out by compressing the samples in a special holder and recording the maximum load, under which cracking of the ceramic samples occurred, indicating a loss of strength and resistance to external loads. Loading was applied at a constant rate of 0.05 mm/s. The critical failure load was determined by a reduction in pressure of at least 75% from the maximum value achieved. All obtained load values were averaged from at least four samples tested for each composition. All samples had identical geometry and similar linear dimensions, ensuring the accuracy of the test results.

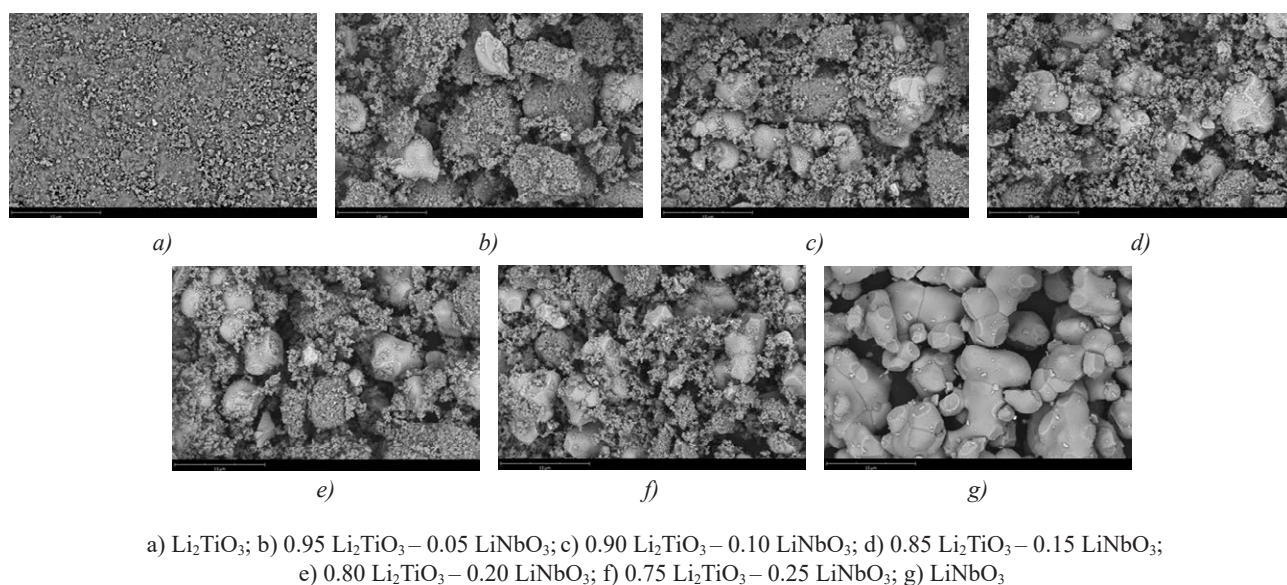


Figure 1. SEM images of the surface of Li_2TiO_3 based ceramics with different LiNbO_3 contents

RESULTS AND DISCUSSION

Figure 1 shows images of the surface of the studied ceramics with the addition of LiNbO_3 .

Figure 1a) shows that Li_2TiO_3 ceramics are characterized by a finely dispersed structure with agglomerated particles of irregular shape. When a dopant is added to the ceramics (Figures 1b-f), a more heterogeneous structure is formed, with larger agglomerates with a finely dispersed structure distributed throughout the sample being observed with an increasing LiNbO_3 content. The obtained results demonstrate that the introduction of LiNbO_3 into the ceramic structure leads to a rearrangement of the morphology of Li_2TiO_3 ceramics depending on the dopant concentration. The formation of a morphologically inhomogeneous structure can contribute to the redistribution of local stresses and a change in the trajectory of crack propagation, which, along with other structural factors, can contribute to an increase in the mechanical stability of the ceramics.

The evolution of phases in the manufactured ceramic composites was studied using X-ray diffraction methods. The corresponding diffraction patterns are shown in Figure 2. The diffraction pattern data reflect a change in the ratio of 2 phases – Li_2TiO_3 with a monoclinic type of crystal structure (PDF-00-033-0831) and lithium niobate with a rhombohedral crystal structure (PDF-01-070-08452), an increase in intensity and weight fraction proportional to the weight fraction of the dopant used to modify lithium metatitanate. The data are presented for samples ground at a grinding speed of 600 rpm to assess the effect of variations in the variation of components in the composition of the material under study on its structural properties. The general appearance of the presented results of X-ray phase analysis indicates a high degree of structural ordering of the sample, regardless of the content of the alloying impurity in the ceramic composition. At the same time, such changes in the morphology of the primary Li_2TiO_3 phase, accompanying the increase in the concentration of the rhombohedral lithium niobate within the sample, lead to a reduction in the size of coherent scattering domains; this indicates the influence of the dopant on the degree of fragmentation of the primary phase while its degree of structural ordering is preserved.

Table 1 presents the structural parameters of the studied lithium-based ceramics depending on changes in concentration in the sample composition.

An analysis of the structural parameters of the studied ceramics, obtained using the Rietveld method, showed that changing the ratio of components in the ceramics leads to different effects on the structural distortion of the crystal lattices of both phases established in the ceramics. Thus, the addition of LiNbO_3 to the Li_2TiO_3 composition at concentrations of 0.05–0.15 M initially leads to a pronounced strain-induced distortion of the tensile type (as evidenced by an assessment of the changes in the parameters and volume of the crystal lattice of the Li_2TiO_3 phase), followed by ordering and partial remov-

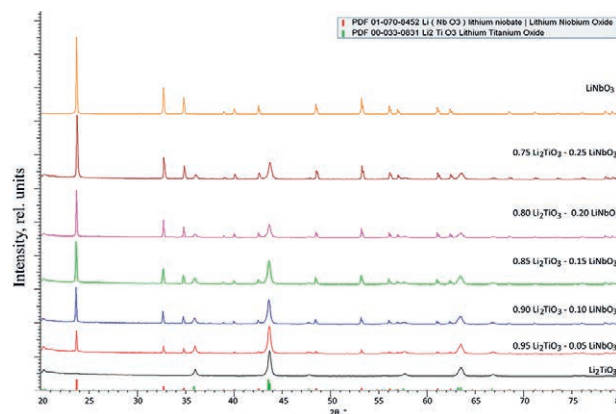


Figure 2. X-ray diffraction results of lithium-based ceramic materials with the addition of lithium niobate to the composition.

al of strain-induced distortions caused by mixing the two phases. At the same time, the parameters of the LiNbO_3 phase increase with an increase in concentration from 0.05 to 0.15 M while at higher concentrations stabilization of the LiNbO_3 phase is observed, characterized by a decrease in the parameters and volume of the crystal lattice, indicating a partial removal of strain-induced distortions and structural stresses. A decrease in the concentration of structural deformation-induced distortions is indicated by a change in the degree of structural ordering, the value of which is presented in Table 1. This value was assessed based on changes in the shape of diffraction reflections associated with the formation of structural distortions caused by mechanical stress and recrystallization processes occurring during sintering. According to the presented data, the increase in the LiNbO_3 concentration in the ceramics leads to an increase in the degree of structural ordering, which directly indicates the decrease in the concentration of deformation-induced distortions in the ceramics due to enhanced recrystallization processes.

Table 1 also presents the results of the crystallite size assessment (coherent scattering regions), which were determined based on the intensity and shape changes of the diffraction lines for each phase using the Scherrer formula. According to the presented data, each phase has significant differences in crystallite size, as evidenced by the obtained assessment results. For the Li_2TiO_3 phase, the broadened shape of the diffraction reflections, which is preserved for all studied concentrations of the component ratios, the average crystallite size is approximately 25...35 nm with a pronounced decrease in size with the increase in the LiNbO_3 concentration in the ceramics, for which the crystallite size increases from 67 ± 4 nm to 92 ± 5 nm in the case of single-component ceramics. Such changes are associated with recrystallization processes that occur during sintering of ceramics, as a result of which structural ordering in the presence of different phases leads to more intense crystallization of the LiNbO_3 phase into larger agglomerates, while smaller grains of Li_2TiO_3 surround

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Table 1 – Structural parameters of the studied lithium-based ceramics depending on the change in concentration of LiNbO_3 in the sample composition

Phase	Crystal lattice parameters						
	Concentration of LiNbO_3 component, M						
	0.0	0.05	0.10	0.15	0.20	0.25	1.0
Li_2TiO_3	a=5.0601 Å, b=8.7697 Å, c=9.6921 Å, $\beta=100.534^\circ$, V=422.84 Å ³	a=5.0799 Å, b=8.7697 Å, c=9.7341 Å, $\beta=99.630^\circ$, V=427.54 Å ³	a=5.0621 Å, b=8.8111 Å, c=9.7073 Å, $\beta=99.905^\circ$, V=426.51 Å ³	a=5.0501 Å, b=8.7731 Å, c=9.7456 Å, $\beta=100.573^\circ$, V=424.45 Å ³	a=5.0581 Å, b=8.7835 Å, c=9.7977 Å, $\beta=100.062^\circ$, V=428.15 Å ³	a=5.0581 Å, b=8.7801 Å, c=9.7303 Å, $\beta=99.945^\circ$, V=425.63 Å ³	-
LiNbO_3	-	a=5.1564 Å, c=13.8384 Å, V=318.64 Å ³	a=5.1564 Å, c=13.8709 Å, V=319.99 Å ³	a=5.1604 Å, c=13.8982 Å, V=320.52 Å ³	a=5.1523 Å, c=13.8819 Å, V=319.14 Å ³	a=5.1271 Å, c=13.8384 Å, V=315.04 Å ³	a=5.1473 Å, c=13.8381 Å, V=317.52 Å ³
Phase	Size of crystallites (coherent scattering regions), nm						
	Concentration of LiNbO_3 component, M						
	0.0	0.05	0.10	0.15	0.20	0.25	1.0
Li_2TiO_3	35±2	32±3	32±2	30±3	29±2	25±3	
LiNbO_3		67±4	70±5	74±4	80±5	87±4	92±5
Phase	Degree of structural ordering, %						
	Concentration of LiNbO_3 component, M						
	0.0	0.05	0.10	0.15	0.20	0.25	1.0
	84.3±2.3	86.7±2.5	88.3±2.4	89.8±2.1	91.1±2.3	91.9±2.5	92.3±3.2

larger particles of LiNbO_3 (see data shown in Figure 1), thereby forming structures with a large number of grain boundaries formed by different phases that have clearly expressed differences in size.

The critical load, which corresponds to the failure of the specimen under single compression, is shown in Figure 3.

The addition of lithium niobate to lithium metatitanate and an increase in the speed of grinding lead to a pronounced strengthening of the material due to several interrelated mechanisms. As the concentration of

LiNbO_3 increases, the critical load on the failure of the samples increases, which may be associated with dispersion strengthening: lithium niobate particles and regions may be distributed throughout the Li_2TiO_3 matrix. The difference in the parameters of the crystal lattices of LiNbO_3 and Li_2TiO_3 may contribute to local elastic distortions and create additional obstacles to the movement of dislocations, increasing the resistance to deformation. The interfaces between phases may act as barriers to the initiation and propagation of cracks, thereby contributing to an

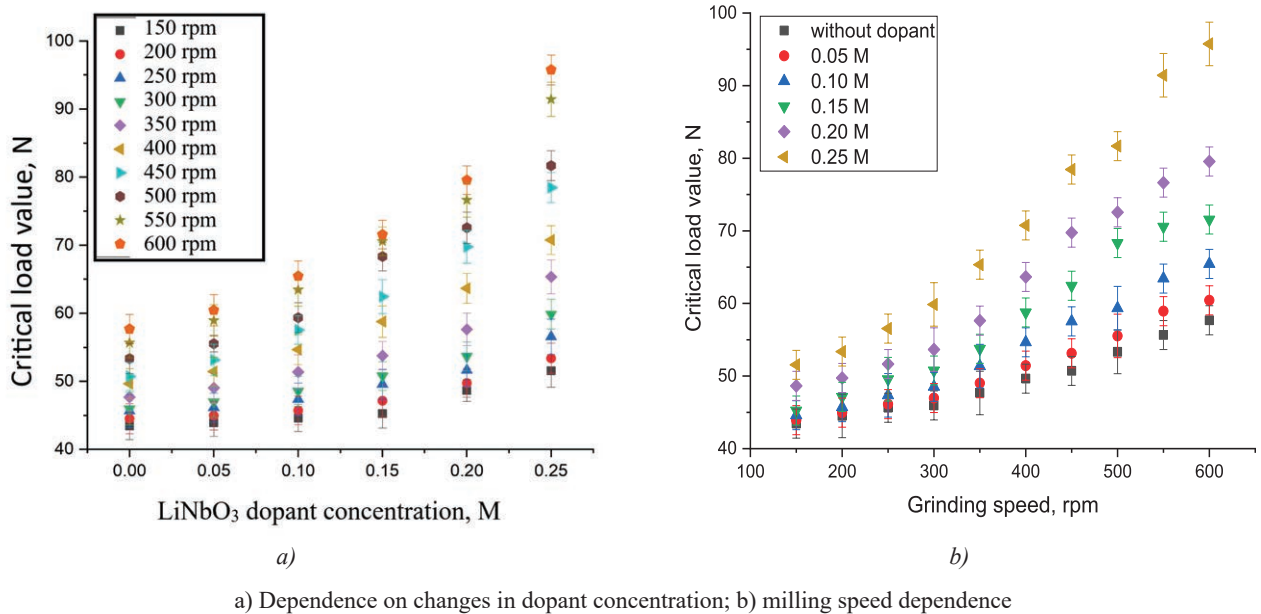


Figure 3. Results of the assessment of the strength properties of the studied ceramics depending on the variation of grinding conditions and dopant concentration

increase in fracture energy. At the same time, microstructural analysis confirms morphological changes following the introduction of the dopant. Depending on the dopant concentration, the compositions exhibit a finely dispersed fraction combined with coarse structures, which may further contribute to increased fracture resistance.

Increasing the milling speed enhances the strengthening effect by reducing the grain size of the main phase. The finer the grain, the greater the proportion of grain boundaries that impede plastic deformation, which is consistent with the possible contribution of the Hall-Petch mechanism. As the rotation speed (milling energy) increases, strong mechanical processes occur in the powder: intense particle refinement, defect accumulation, and surface activation. Higher speeds lead to the formation of smaller grains of the main Li_2TiO_3 phase and fine grinding of LiNbO_3 particles. The formation of a fine-grained structure can enhance the role of grain boundaries as obstacles capable of disrupting and dissipating localized mechanical effects.

The synergistic effect is particularly pronounced when combining high LiNbO_3 concentration with a high grinding speed. This mode creates a microstructure that produces maximum critical loads under mechanical stress. The increased mechanical resistance of the ceramics is likely due to the simultaneous influence of several factors: structural refinement, the formation of a two-phase system, and changes in the morphology of the samples. Consequently, the increase in strength with increasing alloying impurity concentration is enhanced by the high-energy refinement effect, facilitating the formation of a dense, stable, and mechanically strong composite structure.

The obtained results are confirmed by a number of studies of lithium-containing ceramics, where changes in the phase composition and microstructure of the material affect the mechanical properties of the ceramics. Thus, for two-phase ceramics of the Li_2TiO_3 – Li_2ZrO_3 type it was confirmed that the formation of a homogeneous structure with agglomerations of different sizes leads to an increase in resistance to crack propagation and mechanical load [16]. A group of scientists showed a relationship between the formation of a finely dispersed structure and mechanical stability for $\text{Li}_2(\text{Ti,Zr})\text{O}_3$ – Li_4SiO_4 ceramics, for which a grain size of less than 2 μm was combined with a high critical failure load of more than 70 N [17]. Similarly, in a study of two-phase Li_2TiO_3 – Li_2ZrO_3 ceramics high mechanical parameters were also obtained under certain synthesis conditions: during heat treatment at 1100 °C the critical failure load reached 67.6 N [5].

CONCLUSION

The study provided a comprehensive assessment of the effects of lithium metatitanate modification with lithium niobate and mechanical activation parameters on the structural and performance properties of the ceramics. The data obtained confirmed the effectiveness of com-

bining phase alloying and high-energy milling as a tool for targeted microstructural control of lithium-containing materials. Thus, the volume of the unit cell of Li_2TiO_3 varies within the range of 422.8–428.2 Å³ depending on the concentration of LiNbO_3 .

It has been shown that changing the LiNbO_3 concentration and milling rate is an effective technological factor that allows obtaining a stable controlled structure that provides increased mechanical stability without disturbing the phase ordering of the matrix, which indicates the contribution of structural factors to the strengthening of ceramics.

The proposed modification principles may be of interest for the future development of promising lithium-containing ceramics as breeder materials for fusion power plants. Further research should focus on the long-term thermal stability of the resulting compositions, their radiation resistance, and the kinetics of tritium release, which will expand our understanding of the mechanisms of structural evolution and confirm their potential for practical application.

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LiNbO_3 ҚОСПАСЫМЕН ЛЕГИРЛЕНГЕН Li_2TiO_3 НЕГІЗІНДЕГІ КЕРАМИКАНЫҢ ҚҰРЫЛЫМДЫҚ ӨЗГЕРІСТЕРІ МЕН БЕРІКТЕНУ МЕХАНИЗМДЕРІН ЗЕРТТЕУ.

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Литий ниобатымен (LiNbO_3) 0.05–0.25 М концентрациялар диапазонында модификацияланған литий метатитанаты (Li_2TiO_3) негізіндегі керамикалық материалдар зерттелді. Синтез 150–600 айн/мин аралығындағы ұнтақтау жылдамдығында, 50 айн/мин қадаммен планетарлық диірменде механикалық активтендіру әдісімен жүргізіліп, кейін 700 °С температурада 5 сағат бойы термиялық тұрақтандыру жүзеге асырылды. Жұмыстың мақсаты модификациялаушы қоспаның концентрациясы, механикалық өңдеу энергиясы және алынған керамиканың құрылымдық-механикалық қасиеттері арасындағы тәуелділікті анықтау болды.

Фазалық құрамы мен құрылымдық параметрлері рентгендік дифракция әдісімен, ал керамиканың морфологиялық ерекшеліктері сканерлеуші электрондық микроскопия әдісімен зерттелді. Моноклиндік Li_2TiO_3 фазасы мен ромбоэдрлік LiNbO_3 фазасынан тұратын екі фазалы жүйенің қалыптасуы модификациялаушы қоспа мөлшерінің өзгеруіне байланысты кристалдық тор параметрлерінің өзгеруімен және когерентті шашырау аймақтары өлшемдерінің азаюымен қатар жүретіні анықталды. Сканерлеуші электрондық микроскопия (СЭМ) нәтижелері LiNbO_3 қосылған кезде керамика морфологиясының қоспа концентрациясына байланысты айқын өзгеретінін көрсетті. Механикалық активтендіру жылдамдығын 600 айн/мин-ге дейін және модификациялаушы қоспа концентрациясын 0.25 М-ге дейін арттыру ұсақ түйіршікті композиттік микроқұрылымның қалыптасуына ықпал етіп, модификацияланбаған материалмен салыстырғанда беріктік сипаттамаларының жоғарылауына әкеледі.

Алынған нәтижелер LiNbO_3 мөлшері мен механикалық активтендіру параметрлерін өзгерту литийқұрамды керамикалық материалдардың фазалық-құрылымдық күйі мен механикалық қасиеттерін басқарудың тиімді тәсілдерінің бірі болып табылатынын көрсетті. Бұл тәсіл жоғары температуралы энергетикалық қолдану салалары үшін литийқұрамды керамикалардың қасиеттерін одан әрі оңтайландыруға мүмкіндік береді.

Түйін сөздер: *литийқұрамды керамика, литий метатитанаты, литий ниобаты, рентгендік фазалық талдау, кристалдық құрылым.*

ИССЛЕДОВАНИЕ СТРУКТУРНОЙ ЭВОЛЮЦИИ И УПРОЧНЕНИЯ КЕРАМИК НА ОСНОВЕ
 Li_2TiO_3 , ДОПИРОВАННЫХ LiNbO_3 .

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Были изучены керамические материалы на основе метатитаната лития (Li_2TiO_3), модифицированные ниобатом лития (LiNbO_3) в диапазоне концентраций 0,05–0,25 М. Синтез проводился методом механической активации в планетарной мельнице при скоростях измельчения 150–600 об/мин с шагом 50 об/мин, с последующей термической стабилизацией при 700 °С в течение 5 часов. Целью работы было определение зависимости между концентрацией легирующей примеси, энергией механической обработки и структурно-механическими свойствами полученной керамики.

Фазовый состав и структурные параметры изучались методом рентгеновской дифракции, а морфологические особенности керамики – методом сканирующей электронной микроскопии. Как видно, образование двухфазной системы, включающей моноклинную фазу Li_2TiO_3 и ромбоэдрическую фазу LiNbO_3 , сопровождается увеличением вариации содержания легирующей примеси, одновременно с изменением параметров кристаллической решетки и уменьшением размера областей когерентного рассеяния. Анализ методом сканирующей электронной микроскопии (СЭМ) показал выраженные изменения морфологии керамики при добавлении LiNbO_3 в зависимости от концентрации допанта. Увеличение скорости механической активации до 600 об/мин и увеличение концентрации легирующей добавки до 0,25 М приводит к созданию мелкозернистой композитной микроструктуры, где наблюдается улучшение прочностных характеристик по сравнению с нелегированным материалом.

Полученные данные показывают, что сочетание изменения содержания LiNbO_3 и параметров механической активации является одним из наиболее эффективных инструментов контроля фаз и прочности литийсодержащих керамических материалов, что открывает огромные возможности для оптимизации в высокотемпературных энергетических приложениях.

Ключевые слова: литийсодержащая керамика; метатитанат лития; ниобат лития; рентгенофазовый анализ; кристаллическая структура.