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ИЗВЕСТИЯ

РОО «НАЦИОНАЛЬНОЙ
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NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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THERMOMECHANICAL PROCESSING OF MINERAL RAW MATERIALS TO PRODUCE $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ POWDER WITH PEROVSKITE STRUCTURE

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Abstract. The article is devoted to the detailed methods of processing mineral raw materials based on their thermomechanical activation. In this work, from the initial mineral raw materials by such processing a valuable product for various industries is obtained - perovskite. Aims and objectives. The problem of obtaining high-quality perovskites is the instability of quality and purity of the initial mineral raw materials. The aim of this work is to develop the technology of obtaining $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ powder with perovskite structure by solid-phase reaction. Methods - La_2O_3 , Mn_3O_4 , SrCO_3 powders were used as initial components for sintering. The optimum proportions of powders leading to the formation of perovskite structure were determined. Mixing of the powders was carried out in a Fritsch Pulverisette planetary mill. After mixing, microstructure, phase and chemical composition were investigated for the obtained powder. Research results - In this work, the synthesis modes of $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ compound with perovskite structure were determined by high-temperature solid-phase reaction method based on the interaction of carbonates with oxides. The parameters of thermomechanical processing of La_2O_3 , Mn_3O_4 ,

SrCO₃ powders in a planetary mill were determined in the course of research. The operating modes of this mill, providing obtaining of high-quality perovskite without impurities, were determined. Conclusions - The dispersed powder La_{1-x}Sr_xMn_zO₃ obtained in this work is suitable for the formation of high-emitting coatings in the infrared range by high-temperature gas-dynamic methods. XRD analysis showed the presence of La_{1-x}Sr_xMn_zO₃, the only compound after solid-phase synthesis. SEM analysis of the obtained La_{1-x}Sr_xMn_zO₃ powder confirmed the absence of initial components and any other phase formations except for the synthesized compound.

Keywords: Perovskites, high-temperature solid-phase reaction, lanthanum-strontium manganite.

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ПЕРОВСКИТ ҚҰРЫЛЫМЫ БАР LA_{1-x}SR_xMN_zO₃ ҰНТАҒЫН АЛУ ҮШІН МИНЕРАЛДЫ ШИКІЗАТТЫ ТЕРМОМЕХАНИКАЛЫҚ ӨНДЕУ

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Аннотация. Мақала минералды шикізатты олардың термомеханикалық активтенуіне негізделген өндеудің егжей-тегжейлі әдістеріне арналған. Бұл жұмыста бастапқы минералды шикізаттан осындай өндеу арқылы әр түрлі салалар үшін құнды өнім - перовскит алынады. *Мақсаттары мен міндеттері.* Жоғары сапалы перовскиттерді алу мәселесі бастапқы минералды шикізаттың сапасы мен тазалығының тұрақсыздығы болып табылады. Бұл жұмыстың мақсаты-қатты фазалық реакция арқылы перовскит

құрылымы бар $\text{La}_{1-x}\text{Sr}_x\text{Mn}_2\text{O}_3$ ұнтағын алу технологиясын жасау. Әдістер – агломерация үшін бастапқы компоненттер Ретінде La_2O_3 , Mn_3O_4 , SrCO_3 ұнтақтары қолданылды. Перовскит құрылымының пайда болуына әкелетін ұнтақтардың оңтайлы пропорциялары анықталды. Ұнтақтарды араластыру Fritsch Pulverisette планетарлық диірменінде жүргізілді. Араластырудан кейін алынған ұнтақтың микроқұрылымы, фазалық және химиялық құрамы зерттелді. *Зерттеу нәтижелері* – бұл жұмыста перовскит құрылымы Бар $\text{La}_{1-x}\text{Sr}_x\text{Mn}_2\text{O}_3$ қосылысының синтез режимдері карбонаттардың оксидтермен әрекеттесуіне негізделген жоғары температуралы қатты фазалық реакция әдісімен анықталды. Зерттеу барысында планетарлық диірмендегі La_2O_3 , Mn_3O_4 , SrCO_3 ұнтақтарын термомеханикалық өңдеу параметрлері анықталды. Бұл диірменнің қоспасыз жоғары сапалы перовскит алуды қамтамасыз ететін жұмыс режимдері анықталды. *Қорытындылар* – осы жұмыста Алынған дисперсті ұнтақ $\text{La}_{1-x}\text{Sr}_x\text{Mn}_2\text{O}_3$ жоғары температуралы газ-динамикалық әдістермен инфрақызыл диапазонда жоғары сәуле шығаратын жабындарды қалыптастыру үшін жарамды. XRD талдауы қатты фазалық синтезден кейінгі жалғыз қосылыс $\text{La}_{1-x}\text{Sr}_x\text{Mn}_2\text{O}_3$ бар екенін көрсетті. Алынған $\text{La}_{1-x}\text{Sr}_x\text{Mn}_2\text{O}_3$ ұнтағының SEM талдауы синтезделген қосылысты қоспағанда, бастапқы компоненттердің және кез келген басқа фазалық түзілімдердің жоқтығын растады.

Түйін сөздер: Перовскиттер, жоғары температуралы қатты фазалық реакция, лантан-стронций манганиті.

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ТЕРМОМЕХАНИЧЕСКАЯ ПЕРЕРАБОТКА МИНЕРАЛЬНОГО СЫРЬЯ ДЛЯ ПОЛУЧЕНИЯ ПОРОШКА $\text{La}_{1-x}\text{Sr}_x\text{Mn}_2\text{O}_3$ СО СТРУКТУРОЙ ПЕРОВСКИТА

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Аннотация. Статья посвящена детализации методов переработки минерального сырья основанных на термомеханической его активации. В данной работе из исходного минерального сырья путем такой переработки получается ценный продукт для различных отраслей промышленности – перовскит. *Цели и задачи.* Проблемой получения высококачественных перовскитов является нестабильность качества и чистоты исходного минерального сырья. Целью данной работы является отработка технологии получения порошка $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ со структурой перовскита методом твердофазной реакции. *Методы* – в качестве исходных компонентов для спекания использовали порошки La_2O_3 , Mn_2O_3 , SrCO_3 . Были определены оптимальные пропорции порошков, приводящие к образованию структуры перовскита. Смешивание порошков проводили в планетарной мельнице Fritsch Pulverisette. После смешивания для полученного порошка исследовали микроструктуру, фазовый и химический состав. *Результаты исследования* – в работе определены режимы синтеза соединения $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ со структурой перовскита методом высокотемпературной твердофазной реакции, основанной на взаимодействии карбонатов с оксидами. В ходе исследований были определены параметры термомеханической обработки порошков La_2O_3 , Mn_3O_4 , SrCO_3 в планетарной мельнице. Определены режимы работы данной мельницы, обеспечивающие получение качественного перовскита без примесей. *Выводы* – полученный в работе дисперсный порошок $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$, пригоден для формирования высокоизлучающих покрытий в инфракрасном диапазоне высокотемпературными газодинамическими методами. Рентгенофазовый анализ показал наличие $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$ – единственного соединения после твердофазного синтеза. РЭМ-анализ полученного порошка $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$ подтвердил отсутствие исходных компонентов и каких-либо иных фазовых образований кроме синтезированного соединения.

Ключевые слова: перовскиты, высокотемпературная твердофазная реакция, манганит лантана-стронция.

Introduction. Perovskite is a mineral containing rare earth metals such as yttrium, strontium and others. These metals are important for the production of various high-tech products such as plasma displays, gadolinium magnets, solar cells, etc. (Kondratiev, 2022; Yatsenko, et al., 2022) Therefore, perovskite mining plays a key role in the mining industry (Batukhtin, et al., 2019; Batukhtin, et al., 2020; Kondratiev, 2022). With the increasing demand for such products, perovskite mining is becoming more and more important to fulfil the needs of the industry (Kondratiev, 2023). Due to its rare earth metal content, perovskite has high value and high demand in the global market. In addition, perovskite mining contributes

to the development of the mining industry and the creation of new jobs. Perovskite mining is of great importance for the mining industry, as it provides the necessary materials for the production of modern technological products and contributes to the development of the economy (Galachieva, et al., 2023; Klyuev, 2024; Zharikov, et al., 2022; Zhukov, et al., 2022).

Perovskites are obtained from ore in several stages. First, the ore is crushed and pulverized (Kulikova, et al., 2024; Teplyakova, et al., 2022). The ore is crushed into small pieces and then ground to a fine dust to increase the surface area for further processing. After crushing, the ore undergoes a beneficiation process that removes impurities and unwanted elements. The result is an ore concentrate. The ore concentrate is treated with chemicals to extract the perovskite. This process may include crushing, flotation, sorting and other methods (Evdokimov, et al., 2024; Golik, et al., 2022; Golik, et al., 2023; Klyuev, et al., 2023). After chemical treatment, the perovskite is extracted from the mixture and purified to a high purity product. The resulting perovskite may be subjected to additional processing and recycling, such as for the production of solar cells or other technologies. Thus, obtaining perovskites from ore requires a complex beneficiation and chemical treatment process to extract and purify the desired product (Rikker, et al., 2022; Vidayev, et al., 2014; Zverev, et al., 2016).

At the final stage of perovskite production from mineral raw materials, the obtained powders containing metal oxides that are part of perovskite are mixed. The powders are mixed in a certain ratio and subjected to heat treatment. As a result of this process, a reaction between the powder components takes place, resulting in the formation of the crystalline structure of perovskite. After cooling, the perovskite powder is removed from the furnace and subjected to additional processing such as grinding or particle size sorting. This process produces perovskite powder with a high degree of purity and uniformity, making it ideal for use in a variety of applications such as solar cells, catalysts and other technologies (Pashkov, et al., 2014).

Perovskite powders are produced from their natural ores such as bastnesite, monazite, and others. The composition of natural ores may vary and the resulting powders may also contain varying amounts of impurities. As a result, after mixing these initial powders, the quality of the obtained perovskite can vary greatly. Therefore, it is extremely important to determine the modes of thermomechanical treatment of the prepared mineral raw materials, which will allow to obtain perovskite with the required properties.

The aim of the work is to obtain $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$ powder with perovskite structure by solid-phase reaction method. To achieve the goal, the following tasks were solved in the work: calculation of stoichiometry of initial components, mixing and firing of the obtained composition of materials, determination of microstructure, phase and particle size distribution of the obtained powder.

Methods. La_2O_3 (99.999% purity), Mn_2O_3 (99.99% purity), SrCO_3 (99.9%

purity) powders were used as initial components for sintering. Before mixing the components, the Mn₂O₃ powder was roasted in a corundum crucible in air at 1000 °C for 6 h to obtain the more active compound Mn₃O₄, where increasing the weight fraction of manganese in the compound will increase the reactivity in solid phase synthesis (Plotnikova, et al., 2016; Skeebe, et al., 2016). The following proportions of starting components were calculated to obtain the compound La_{0.9}Sr_{0.1}Mn₂O₃: La₂O₃, 64.17 weight %; SrCO₃, 3.491 weight % (in terms of oxide); Mn₃O₄, 33.38 weight %. Mixing of La₂O₃, Mn₃O₄, SrCO₃ powders was carried out in a Fritsch Pulverisette 6 planetary mill with an agate headset at a mass ratio of balls to mixture of 2:1 at a speed of 200 rpm for 5 min, after which the obtained mixture was pressed into a cylindrical sample with a diameter of 40 mm and a thickness of 10 mm. Firing of the obtained tablet was carried out in air atmosphere at 1350±50°C for 8 hours. The sample was then re-milled, re-pressed and fired under the same conditions at 1350±50°C for 8 hours, after which the resulting sintered agglomerate was milled to a powder size of less than 20 microns.

Standard modern methods of powder research were used to determine the microstructure, phase and chemical compositions. Information on the equipment used for the study is given in Table 1.

Table 1 – Equipment for studying the resulting powder

Study scope	Instrument name	Instrument brand
Phase composition	X-ray Diffractometer	ARL X'TRA
Microstructure	Scanning electron microscope	Teskan Mira 3
Particle size distribution	Laser particle size analyser	ANALYSETTE 22 NanoTec plus

Research results. In the course of experimental work, mixing of mineral raw materials in a planetary mill was carried out. When mixing La₂O₃, Mn₃O₄, SrCO₃ powders in a planetary mill, a number of different thermomechanical processes take place. The La₂O₃, Mn₃O₄, SrCO₃ powders will be mixed in the planetary mill under mechanical forces. This will allow uniform distribution of components and ensure homogeneity of the mixture composition. During the mechanical mixing of the powders in the planetary mill, the powders are broken down and the particle size is reduced. This can improve the reactivity of the components and promote more uniform distribution of the particles in the mixture. Mechanical agitation of powders in a planetary mill can help activate the surface of the particles, increasing their surface energy and enhancing reactivity. When mixing La₂O₃, Mn₃O₄, SrCO₃ powders in a planetary mill, initial formation of chemical bonds between the components may occur. This may promote more efficient reaction of the components during subsequent heat treatment. As a result of mechanical mixing and chemical reactions a new structure La_{0.9}Sr_{0.1}Mn₂O₃ is formed.

Comparison of the obtained diffraction spectrum with the PDF-40-1100 diffraction map data confirmed that the obtained compound has a perovskite structure. The determination of the space group P2/c (130) indicates the specific

symmetry of the perovskite crystal lattice, which allows a more accurate description of its structure. The absence of pronounced peaks corresponding to strontium (Sr) and its carbonate (SrCO_3) indicates that strontium atoms have been successfully incorporated into the perovskite crystal lattice. The absence of additional peaks, except those corresponding to $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$, indicates a high degree of homogeneity of the composition of the obtained powder.

The data obtained from the analysis of diffraction peaks confirm the successful synthesis of $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ compound with perovskite structure. The absence of peaks corresponding to the initial components indicates the completeness of the synthesis and successful introduction of strontium atoms into the crystal lattice. This is important for further studies, since the structure and composition of the material play a key role in its physical and chemical properties.

The diffractogram of the synthesised powder is shown in Figure 1.

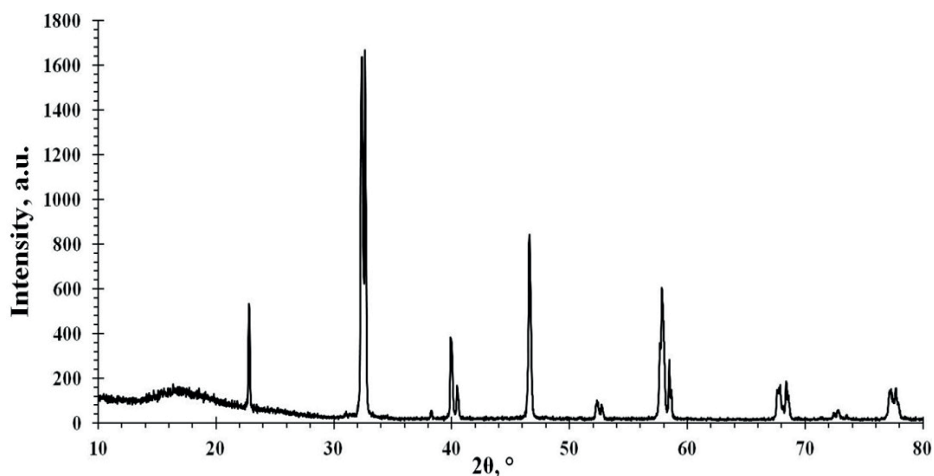


Fig. 1. X-ray diffraction pattern of the synthesized $\text{La}_{0.99}\text{Sr}_{0.01}\text{MnO}_3$ powder

The SEM images show a very high homogeneity of the structure of the synthesised material. There are no visible inhomogeneities such as pores, cracks or inclusions. The powder particles are uniformly distributed throughout the entire volume, indicating the high quality of the synthesis process and the absence of significant gradients. The presence of distinct facets on the particles confirms that the milling process was efficient and resulted in the splitting of large particles. The absence of agglomeration, i.e. sticking of particles, indicates that the grinding was sufficiently intensive to prevent the formation of large aggregates. The combination of efficient milling and lack of agglomeration indicates a high dispersibility of the powder, which is an important factor for many applications.

SEM analysis did not reveal the presence of starting components, confirming the completeness of the synthesis and the absence of residues of starting substances.

The absence of phase formations other than the main phase confirms the high purity of the obtained material. The results of scanning electron microscopy are shown in Figure 2.

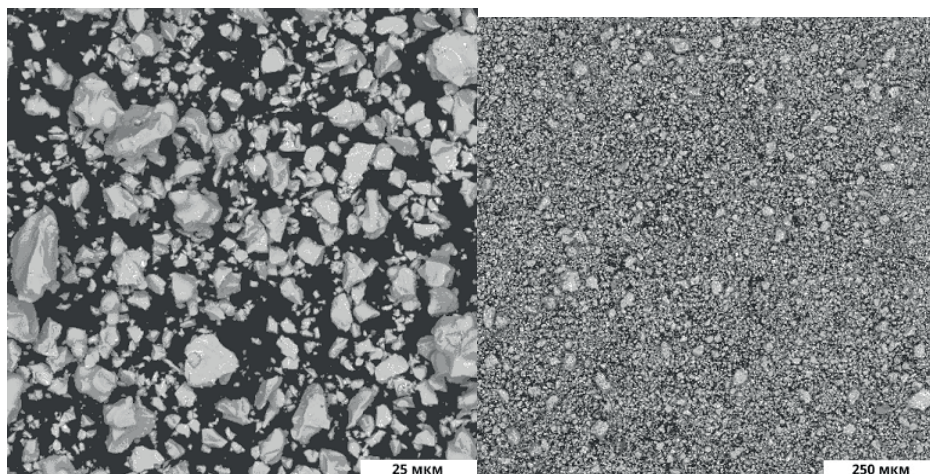


Fig. 2. Microstructure and particle size of $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$ powder.

The particle size distribution of the obtained perovskite powder describes the particle size distribution in it. The obtained data $d(10)=2.33 \mu\text{m}$, $d(50)=8.1 \mu\text{m}$, $d(90)=16.79 \mu\text{m}$ allow us to characterise the particle size distribution in the powder. In this case, 10% of particles are smaller than $2.33 \mu\text{m}$, 50% of particles are smaller than $8.1 \mu\text{m}$, and 90% of particles are smaller than $16.79 \mu\text{m}$. The difference between $d(10)$, $d(50)$ and $d(90)$ indicates that the particle size distribution in the powder is not uniform. The large difference between $d(10)$ and $d(90)$ ($16.79 \mu\text{m} - 2.33 \mu\text{m} = 14.46 \mu\text{m}$) indicates that the powder contains particles of a wide range of sizes, from very small ($2.33 \mu\text{m}$) to larger ($16.79 \mu\text{m}$). The result of the particle size distribution study is presented in Table 2.

Table 2 – Granulometric composition of the resulting powder $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$

Name, brand	Production method	Particle size distribution, μm		
		d(10)	d(50)	d(90)
Powder $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$	High-temperature solid-phase synthesis	2,33	8,1	16,79

Comparison of the obtained lanthanum-based perovskite powder with analogues showed a number of its similar parameters and a number of differences. The obtained lanthanum-based perovskite powder with the parameters $d(10)=2.33 \mu\text{m}$; $d(50)=8.1 \mu\text{m}$; $d(90)=16.79 \mu\text{m}$ differs from other analogues in a number of characteristics, but also has common features. The resulting powder is characterised by a wide

range of particle sizes, from 2.33 μm to 16.79 μm . Many analogues of lanthanum-based perovskites have a narrower range of particle sizes, e.g., $d(10) = 4 \mu\text{m}$, $d(50) = 6 \mu\text{m}$, $d(90) = 8 \mu\text{m}$. This difference in particle size distribution can affect material properties such as density, strength and reactivity. The obtained powder is characterised by a high homogeneity of structure, which is a significant advantage. Some lanthanum-based perovskite analogues may have a heterogeneous structure, for example with the presence of agglomerates or pores. The high homogeneity of the obtained powder improves its properties and increases its efficiency in various applications.

The resulting powder is characterised by high purity, with no chemical impurities of other elements. Some analogues of lanthanum-based perovskites may contain impurities of other elements, which can negatively affect the properties of the material. The high purity of the resulting powder ensures the predictability of its properties and improves its efficiency. All lanthanum-based perovskites have the same chemical composition, which makes them similar in their basic properties. The only differences in properties are the presence of impurities and different particle size distribution. A wide range of particle sizes in the resulting powder can improve its reactivity and increase its efficiency in catalytic reactions (Galachieva, et al., 2024; Sharipzyanova, et al., 2023). However, it can also deteriorate its properties in ceramic materials, reducing their strength. The high homogeneity of the obtained powder makes it more predictable in use and improves its properties in various applications. The high purity of the obtained powder makes its properties predictable and improves its performance in various applications.

The obtained lanthanum-based perovskite powder differs from its analogues in its wide range of particle sizes, high homogeneity and purity. These differences can influence the properties of the material and make it more suitable for applications where increased reactivity (catalytic reactions) is required.

The resulting lanthanum-based perovskite has a structure similar to the mineral perovskite (CaTiO_3). It has unique properties that make it useful for a wide range of applications. One of the main applications of the resulting lanthanum-based perovskite is in electronics and photonics. This material has semiconducting properties that allow it to be used in solar cells, LEDs, lasers and other electronic devices. The obtained lanthanum-based perovskite can be used as a catalyst for various chemical reactions. The obtained lanthanum-based perovskite can be used as a material for energy storage and conversion. For example, it can be used in supercapacitors, fuel cells and other devices for energy-efficient energy storage and processing.

However, the above data showed that the best application of the obtained perovskite is possible as a catalyst. Lanthanum-based perovskite (LaVO_4) is an effective catalyst for a wide range of chemical reactions due to its unique physical and chemical properties.

Oxidation of alkenes and alkanes: $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$, is capable of catalysing

oxidative reactions using oxygen or hydrogen peroxide as an oxidant. For example, $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$, can be used to oxidise cyclohexene to adipic acid, which is an important intermediate in the production of nylon.

Hydrogenation: $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$, can be used to catalyse the hydrogenation of double bonds in organic compounds, yielding valuable products such as alkanes and cycloalkanes.

Dehydrogenation: $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$, can be used to catalyse the dehydrogenation of alcohols and aldehydes, for example, converting ethylbenzene into styrene, which can be further used in the production of plastics.

Hydrogenation: $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$, can catalyse the hydrogenation of organic compounds, for example by converting nitroxyls into amine.

In addition to the reactions listed above, lanthanum-based perovskite can be applied to a wide range of other chemical processes due to its unique structure and activity.

Conclusion. The paper presents the results of synthesis of $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ compound with perovskite structure by high-temperature solid-phase reaction method based on the interaction of carbonates with oxides. The parameters of thermomechanical processing of La_2O_3 , Mn_3O_4 , SrCO_3 powders in a planetary mill have been determined. The operating modes of this mill, providing obtaining of high-quality perovskite without impurities, have been determined.

The obtained dispersed powder $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$, is suitable for the formation of high-emitting coatings in the infrared range by high-temperature gas-dynamic methods. XRD analysis showed the presence of $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$, the only compound after solid-phase synthesis. SEM analysis of the obtained $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ powder confirmed the absence of initial components and any other phase formations besides the synthesised compound.

The obtained $\text{La}_{1-x}\text{Sr}_x\text{Mn}_z\text{O}_3$ material with perovskite structure will be used for further production of high-emitting coatings in the infrared range by high-temperature gas-dynamic methods.

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