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Research Article

Magnetic properties of $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ depending on the phase composition of the reaction mixture

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Abstract

This paper presents investigations of phase transformations during the crystallization of $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ by the solid-phase technique from a stoichiometric mixture of oxides MoO₃, La_2O_3 and Fe_2O_3 and $SrCO_3$ and precursors $Sr_{0.5}La_{0.5}FeO_3$ and $SrMoO_4$. Using XRD and thermogravimetric analyses, the influence of synthesis modes on the chemical processes occurring during the formation of double perovskite was studied. It has been established that the synthesis of lanthanum-strontium ferromolybdate in a mixture of oxides proceeds through a number of series-parallel stages. At the initial stage of interaction, the resulting lanthanum-strontium ferromolybdate is enriched with iron and its composition changes during the reaction towards an increase in the molybdenum content. As the temperature increases, the content of double perovskite increases, and the concentration of the secondary phase $SrMoO_4$ does not disappear to zero until the synthesis temperature, which indicates that solid-phase reactions with the formation of a solid solution $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ from oxides are difficult to occur. It was determined that to minimize the influence of studying the temperature dependences of the degree of phase transformations during the crystallization of double perovskite, combined heating modes were optimized. The use of optimized synthesis modes made it possible to obtain single-phase $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ powder with the superstructural ordering (82%), the Curie temperature of 450 K and a magnetization value of $40.9 \text{ A} \cdot \text{m}^2 \cdot \text{kg}^{-1}$ at T = 77 K in a magnetic field with induction $B \ge 0.86$ T.

Keywords

double perovskite, lanthanum strontium ferromolybdate, ferrimagnetic, superstructural ordering of Fe/Mo cations, sequence of phase transformations, thermogravimetric analysis, X-ray phase analysis, magnetization

1. Introduction

One of the promising materials for use in spintronic devices that have the necessary combination of magnetic and magnetoresistive characteristics is the ferrimagnet $Sr_2FeMoO_{6-\delta}$ with a double perovskite structure. This material has high Curie temperatures ($T_C \sim 400-430$ K), large values of negative magnetoresistance (up to 40%)

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at 4.2 K) and almost 100% degree of spin polarization [1–5]. In addition, it has high electrical conductivity and satisfactory thermal stability at elevated temperatures, over a wide range of oxygen partial pressures.

Above the Curie temperature, strontium ferromolybdates is in a paramagnetic state with a cubic structure (Fm3m), below which a ferrimagnetic ordering in a tetragonal structure (I4/m) is formed, characterized by the presence of Fe³⁺-O²⁻-Mo⁵⁺ chains with superstructural ordering cations [6, 7]. The presence of point (anti-structural) defects ([FeMo] and [MoFe]) destroys superstructural ordering due to the fact that iron and molybdenum ions can be in different valence states (Fe²⁺, Fe³⁺ and Mo⁵⁺, Mo⁶⁺), having a significant effect on the magnetic structure magnetic [8–11]. Depending on the concentration of such defects, ferrimagnetic, antiferromagnetic or mixed magnetic states can be realized [12–14]. These research objects have unique and extremely important for practical application magnetic and magnetotransport properties, which can be used as basic elements of spintronic devices, magnetic field sensors, non-volatile dynamic memory devices (MRAM), in spin-polarized electron injectors, as electrodes for solid fuel elements, spin transistors, etc. [1, 2, 4, 15–19].

Low resistivity and high Curie temperature are the important conditions for the use of a certain magnetic material in microelectronic devices are. Recent studies [20–22] have shown that the Curie temperature can be increased by partially replacing Sr^{2+} with La^{3+} in $Sr_2FeMoO_{6-\delta}$. An increase in the content of lanthanum cations significantly changes the bond lengths and bond angles for Fe-O-Mo and Mo-Mo, and hence the magnetic interaction between Fe and Mo atoms, which, in turn, affects the electronic and magnetic properties of the solid solution $Sr_{2-r}La_rFeMoO_{6-\delta}$. It was found that $Sr_{1-5}La_{0-5}FeMoO_{6-\delta}$ has the lowest resistivity [23]. It should be noted that an additional increase in the La content in the composition of the material leads to a deterioration in its galvanomagnetic properties, which is due to the metastability of the magnetic state [24, 25]. In connection with the above, in this work we have focused on the investigation of the $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ solid solution.

With the development of new technology and with increasing technical needs for high performance and special physical properties, special requirements are placed on the physico-chemical characteristics of spintronic devices. An important task in this area remains the improvement of the technology for obtaining high-quality samples of double perovskites with reproducible magnetic and galvanomagnetic properties. When analyzing the accumulated data obtained by a number of authors, the multi-stage crystallization process of $Sr_{2-x}La_xFeMoO_{6-\delta}$ was established, which is due to the complexity of phase transformations, low phase formation kinetics and low mobility of Fe³⁺ and Mo⁵⁺ cations [26–30]. Publications contain information about the preparation of $Sr_{2-x}La_xFeMoO_{6-\delta}$ by the mechano-chemical method followed by the use of high-temperature synthesis in a reducing gas environment [26–30]. At the same time, in the investigations performed, there are practically no strict correlations linking the functional characteristics of materials with their production conditions. In this case, to form a single-phase $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ powder with reproducible physico-chemical properties, control over defect formation processes is required.

Therefore, modern research focuses on the deeper and more detailed approaches to the synthesis of double perovskites related to the study of the sequence of phase transformations during their crystallization. In this regard, research aimed at studying high-temperature phase transformations and determining the composition of intermediate crystalline phases during the synthesis of Sr_{1.5}La_{0.5}FeMoO_{6- δ} is of a particular importance. In this article, a correlation will be established between the rate of phase transformations and the degree of phase transformation of lanthanum-strontium ferromolybdate obtained by different methods, which will allow for a targeted change in the phase composition of the synthesized ceramics with reproducible physico-chemical properties.

2. Experimental

To investigate the sequence of phase transformations in $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$, La_2O_3 , Fe_2O_3 and MoO_3 oxides were used, as well as strontium carbonate $SrCO_3$. Grinding and mixing of the stoichiometric mixture of the starting reagents was carried out in a planetary ball mill of the PM 100 type manufactured by Retsch GmbH (Germany) in a liquid medium (alcohol) for 3 h. The resulting mixtures were dried at a temperature of 350 K and pressed into pellets. The mixture has been annealed in a polythermal mode at temperatures in the range of 300–1370 K in a 5% H₂/Ar flow, followed by the quenching at room temperature.

The phase composition of solid-phase synthesis products was determined on a PANalytical Empyrean diffractometer in CuK_{α} radiation using the ICSD-PDF2 database (Release 2022). The X-ray diffraction (XRD) patterns have been recorded at room temperature in the angle range $2\theta = 10-90^{\circ}$ with step $\Delta 2\theta = 0.026^{\circ}$. The argon-hydrogen atmosphere was created by a constant flow of argon-hydrogen mixture through an AntonPaar HTK 1200N high-temperature chamber. The experiments have been carried out in the temperature range 290–1370 K with a heating rate of 10 deg/min. In this case, each point was measured sequentially 4 times upon reaching the specified temperature (exposure time 2.5 min). The quantitative-phase composition of solid-phase synthesis products and the degree of superstructural ordering were determined based on the XRD data using the POWDERCELL software [31] and the FullProf [32] software by the Rietveld method.

The thermal behavior of the samples was investigated by thermogravimetric analysis (TGA) on the Netzsch 209 F1 Libra gravimenter in an argon flow at a heating rate of 10 deg/min.

The Curie temperature was determined by analyzing the temperature dependences of the magnetization of the $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ sample by the ponderomotive method in the temperature range of 77–800 K in an applied magnetic field of 0.86 T, using a universal PPMS set up manufactured by the Cryogenic Ltd.

3. Results and discussion

Based on the study of the sequence of phase transformations during the crystallization of double perovskite $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$, it was established that the synthesis of lanthanum-strontium ferromolybdate in a mixture of oxides proceeds through a number of serial-parallel stages (Fig. 1). In particular, as a result of consideration of the phase transformations during the crystallization of double perovskite with a heating rate of 1.5 deg/min in the temperature range of 300-1300 K from a mixture of reagents $MoO_3 + 0.25La_2O_3 + 0.5Fe_2O_3 + 1.5SrCO_3$ of the stoichiometric composition, according to the X-ray phase analysis (XRD) data, the following sequence of phase transformations has been established: {Fe₂O₃, La₂O₃, MoO_3 , $SrCO_3$ (300 K) \rightarrow { $SrMoO_4$, $SrCO_3$, $La_2O_2(CO_3)$, $Fe_{3}O_{4}, Fe_{1-x}O, MoO_{2}\} (973 \text{ K}) \rightarrow \{SrMoO_{4}, SrCO_{3},$ MoO_2 , La_2SrO_x , $Sr_{1-x}La_xFeO_3$ } (1073 K) \rightarrow {SrMoO₄, SrCO₃, Sr_{1-x}La_xFeO₃, (Sr,La)₂FeMoO₆} (1173 K) \rightarrow {SrMoO₄, SrCO₃, (Sr,La)₂FeMoO₆} (1273 K) \rightarrow {SrMoO₄, (Sr,La)₂FeMoO₆} (1373 K) (Fig. 1).

Initially, at $T \approx 970$ K, the SrMoO₄ compound is formed, and then at $T \approx 1070$ K, the appearance of X-ray reflections of the Sr_{1-x}La_xFeO₃ phase is detected. With increasing temperature in the range of 1191–1244 K, the amplitude values of the diffraction peaks of Sr_{1-x}La_xFeO₃ decrease much faster than those of the SrMoO₄ compound, while the diffraction peaks of the Sr_{2-x}La_xFeMoO_{6- δ} compound appear and grow (see Fig. 1). Upon reaching the maximum synthesis temperature of 1370 K, in addition to the main phase, the impurity SrMoO₄ remains.

For samples synthesized from stoichiometric mixtures $MoO_3 + 0.25La_2O_3 + 0.5Fe_2O_3 + 1.5SrCO_3$ it was found that at the initial stage of interaction the resulting lanthanum-strontium ferromolybdate is enriched with iron and its composition changes during the reaction towards an increase in molybdenum content. Molybdenum oxide in a ternary mixture reacts with strontium carbonate somewhat faster than iron oxide. Iron, which is part of the complex oxides $Sr_{1-x}La_xFeO_3$ in the stoichiometric mixture of the initial reagents, is more reactive and stimulates the formation of lanthanum-strontium ferromolybdate. The active centers for the appearance of nuclei of the $Sr_{2-x}La_xFeMoO_{6-\delta}$ phase are the surface of strontium lanthanum ferrite, on which the dissociation process occurs, accompanied by the disappearance of Sr_{1-x}La_xFeO₃ during the growth of double perovskites. Starting from the nuclei of the new phase, the interface between the solid phases moves deeper into the crystal lattice of the parent phase and the decrease in the growth rate of $Sr_{2-x}La_xFeMoO_{6-\delta}$ is facilitated by an increase in the thickness of the reaction product layer. If the resulting product layer has a low mobility of cations and anions, then the heterogeneous reaction from adsorption-chemical goes



Figure 1. XRD patterns of samples synthesized in a continuous flow of 5% H_2/Ar from a stoichiometric mixture of initial reagents $MoO_3 + 0.25La_2O_3 + 0.5Fe_2O_3 + 1.5SrCO_3$ at a heating rate of 1.5 deg/min in the temperature range 300–1240 K with their subsequent quenching at room temperature



Figure 2. Temperature dependence of the change in the normalized mass of a mixture of powders $MoO_3 + 0.25La_2O_3 + 0.5Fe_2O_3 + 1.5SrCO_3$ and its derivative, during annealing in a continuous flow of Ar at a heating rate of 10 deg/min in the temperature range 300–1370 K

into the diffusion mode, which in turn leads to a slowdown in the growth rate of the double perovskite and the dissociation of side phases.

Using the TGA method, it was established that when a sample consisting of initial reagents in the stoichiometric ratio MoO₃ + 0.25La₂O₃ + 0.5Fe₂O₃ + 1.5SrCO₃ is heated from 300 K to 1000 K, practically no changes in mass occur. At temperatures close to 1000 K, mass loss begins ($\Delta m/m_0 < 0.4\%$) (Fig. 2), which can be explained by the release of carbon dioxide during the decomposition of strontium carbonate and the intermediate phase La₂O₂(CO₃) (see Fig. 1).

When heated to higher temperatures, chemical processes intensify and in the temperature range of 1000– 1370 K two main pronounced effects of mass loss are observed (see Fig. 2). The first effect, accompanied by a decrease in the mass of the sample to $\Delta m/m_0 \approx 96\%$, begins at a temperature of 1070 K and reaches an extreme at T = 1191 K (see Fig. 2). According to the XRD data, the appearance and growth of the Sr_{1-x}La_xFeO₃ phase was detected in this temperature range (see Fig. 1). The small peak at 1230 K (see Fig. 2) is due to the large amount of absorbed oxygen.

With a further increase in temperature to 1244 K, a sharp increase in the rate of decrease in the mass of the sample was noted, reaching an extremum at $\Delta m/m_0 \approx 94\%$ (see Fig. 2). According to the XRD data, heating the mixture to a temperature of 1244 K leads to a decrease in the number of side phases SrMoO₄ and Sr_{1-x}La_xFeO₃ with the simultaneous appearance and growth of the Sr_{2-x}La_xFeMoO_{6- δ} compound (see Fig. 1). As the temperature increases, the content of double perovskite increases, and the concentration of the SrMoO₄ phase decreases , but does not disappear to zero up to the synthesis temperature T = 1370 K, which indicates the difficulty of solid-phase reactions with the formation of the Sr_{1.5}La_{0.5}FeMoO_{6- δ} solid solution.

The TGA data cannot be directly related to the XRD results, since the synthesis of $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ proceeds in a reducing atmosphere of argon-hydrogen, while the TGA was carried out in a flow of pure argon. However, an important result of thermal analysis can be considered the fact that the decomposition of carbonates, after which the formation of the desired complex oxides becomes



Figure 3. XRD patterns of $Sr_{1.5}La_{0.5}FeMoO_{6-d}$ samples: (curve *I*) obtained from a mixture of oxides, (curve *2*) obtained from precursors $Sr_{0.5}La_{0.5}FeO_3$ and $SrMoO_4$ at T = 1370 K in a flow of 5%H₂/Ar for 40 h; (curve *3*) obtained from precursors $Sr_{0.5}La_{0.5}FeO_3$ and $SrMoO_4$ using combined heating modes



Figure 4. Temperature dependences of the magnetization of $Sr_{1.5}La_{0.5}FeMoO_{6-d}$ samples: (curve *I*) obtained from precursors $Sr_{0.5}La_{0.5}FeO_3$ and $SrMoO_4$ at T = 1370 K in a flow of 5%H₂/Ar for 40 h; (curve 2) obtained from precursors $Sr_{0.5}La_{0.5}FeO_3$ and $SrMoO_4$ using combined heating modes

possible, begins at a temperature of about 1000 K, at which, according to the XRD results, in a hydrogen-containing atmosphere, iron and molybdenum are present in the Fe₃O₄, Fe_{1-x}O and MoO₂ reduced phases. In the Sr_{1.5}La_{0.5}FeMoO₆ phase, these elements (iron and molybdenum) are in a more oxidized state. Thus, obtaining a single-phase sample of a given composition turns out to be impossible in an argon-hydrogen atmosphere from the MoO₃ + $0.25La_2O_3 + 0.5Fe_2O_3 + 1.5SrCO_3$ mixture (Fig. 3, curve *I*).

Based on the fact that complex oxides $SrMoO_4$, $Sr_{0.5}La_{0.5}FeO_3$ are formed in the initial charge during its annealing, which are accompanying in almost the entire temperature range of the synthesis of strontium lanthanum ferromolybdate and are poorly soluble, to accelerate the dissolution of intermediate products of chemical processes, they were used as starting reagents. In these phases, all elements are in high oxidation states and in the form of oxides. The reducing atmosphere turned out to be favorable for the formation of the desired phase. As a result, single-phase lanthanum-strontium ferromolybdate without superstructural ordering (Fig. 3, curve 2) was synthesized at T = 1370 K in a flow of 5%H₂/Ar for 40 h, according to the following chemical reaction:

 $SrMoO_4 + Sr_{0.5}La_{0.5}FeO_3 + H_2 = Sr_{1.5}La_{0.5}FeMoO_6 + H_2O\uparrow.$

In this case, the magnetization value of the resulting sample with a Curie temperature of 443 K is $M = 21.8 \text{ A} \cdot \text{m}^2 \cdot \text{kg}^{-1}$ at T = 77 K in the magnetic field with induction $B \ge 0.86 \text{ T}$ (Fig. 4, curve 1).

To optimize the conditions for obtaining a single-phase $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ sample with superstructural ordering of Fe/Mo cations from precursors, the temperature dependences of the degree of phase transformations during the crystallization of double perovskite and their derivatives were plotted and analyzed (Figs 5 *a* and *b*).

It was found that with an increase in heating temperature above 770 K, a decrease in the amplitude values of α_{max} is observed for both phases SrMoO₄ and Sr_{0.5}La_{0.5}FeO₃ (Fig. 5 a). The presence of more significant kinetic difficulties in the dissolution of SrMoO₄ is confirmed by data on temperatures at which the amplitude values of the derivative of the degree of conversion $(d\alpha/dT)_{min}$, indicating the maximum dissolution rate, are 100 K higher than those for the Sr_{0.5}La_{0.5}FeO₃ compound. When considering the decomposition rates of oxides, it was noted that the highest minimum values $(d\alpha/dT)_{min} = -0.23$ are observed for $Sr_{0.5}La_{0.5}FeO_3$ at T = 1049 K, and for $SrMoO_4$ $(d\alpha/dT)_{min} = 0.19$ at T = 1150 K (Fig. 5 b). This indicates a higher rate of chemical processes with the dissolution of $Sr_{0.5}La_{0.5}FeO_3$ compared to the situation with strontium molybdate (Fig. 5 b). Based on the above data, it follows that in order to reduce the processes of phase formation and increase the rate of decomposition of the intermediate reaction products SrMoO₄ and Sr_{0.5}La_{0.5}FeO₃ during crystallization of the Sr_{1.5}La_{0.5}FeMoO_{6-δ} solid solution,



Figure 5. Temperature dependences of the degree of conversion (*a*) and their derivatives (*b*) of the compounds $SrMoO_4$, $Sr_{0.5}La_{0.5}FeO_3$ and $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$

the dynamics of phase transformations should be taken into account and combined heating conditions should be used. Thus, in the low-temperature region, where the formation and growth of double oxides occurs, the temperature rise rate should be maximum, and in the high-temperature region, where the dissolution of the formed side compounds is observed, it should be low (Figs 1 and 5).

According to the analysis data $\alpha = f(T)$, it was found that with increasing temperature, the degree of conversion of Sr_{1.5}La_{0.5}FeMoO_{6- δ} increases, and at T = 1370 K reaches maximum values $\alpha = 100\%$ (Fig. 5 *a*). At the same time, in the temperature range $T \cong 1060-1140$ K, a slowdown in the growth rate of double perovskite is observed with the presence of min $|(d\alpha/dT)|$ at 1100 K, most likely due to a decrease in the coefficients of chemical diffusion of reagents into the reaction zone (Fig. 5 *b*). The discovered gradual change in the growth rate of Sr_{1.5}La_{0.5}FeMoO_{6- δ} with the presence of two maxima (max $|d\alpha/dT|$) of the rate of change in the degree of transformation of a function of the form $d\alpha/dt = f(T)$ at $T \cong 1040$ K and 1160 K coincides with temperatures of maximum dissolution of Sr_{0.5}La_{0.5}FeO₃ and SrMoO₄ established above.

In this case, the revealed fact of a faster reaction of $Sr_0 {}_5La_0 {}_5FeO_3$ in the temperature range where the first maximum exists $\max_1 |d\alpha/dT|$, the growth rate of $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ is most likely associated with the implementation of a crystallization mechanism in which kinetic difficulties are minimized due to the intensive dissolution of Sr_{0.5}La_{0.5}FeO₃, which leads to an increase in the double perovskite growth rate. The same chemical process during the crystallization of a magnet is realized in the temperature range where the second maximum $\max_2 |d\alpha/dT|$ exists for the growth rate of $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ only now due to the intensive dissolution of SrMoO₄. In this case, the rate of the entire transformation is determined by the rate of interaction of the reagents at the interface with the $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ grains.

Based on the above data, combined heating modes were optimized to obtain a single-phase solid solution:

- at the first stage, preliminary synthesis has been carried out at T = 1050 K for 20 h. This temperature was chosen due to the fact that at its value min $|(d\alpha/dT)|$ for the compound Sr_{0.5}La_{0.5}FeO₃, as well as max $|(d\alpha/dT)|$ for the Sr_{1.5}La_{0.5}FeMoO_{6- δ} solid solution;

– at the second stage, to increase the reactivity of the mixture and the diffusion mobility of the reagents, the resulting layer of the reaction product was crushed, homogenized and a high dispersion of the mixture was achieved by fine vibration grinding in alcohol for 2 h;

– at the third stage, in order to maximize the rapid decomposition of the intermediate phases $SrMoO_4$ and

Sr_{0.5}La_{0.5}FeO₃ and achieve the degree of conversion $\alpha = 100\%$ for Sr_{1.5}La_{0.5}FeMoO_{6- δ} the synthesis has been carried out at T = 1050 K for 5 h followed by heating to T = 1150 K, since under such conditions the maximum rates of change in the degree of conversion of the double perovskite were achieved.

As a result of using combined synthesis modes, it was possible to obtain a single-phase compound $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ with the presence of the superstructural ordering of iron and molybdenum cations, as indicated by the XRD reflections (101) and (103) (see Fig. 3, curve 3). Under these synthesis conditions, the magnetization value is 40.9 A · m² · kg⁻¹ at T = 77 K in the magnetic field of 0.86 T, and the Curie temperature is 450 K (see Fig. 4, curve 2).

4. Conclusions

Based on the investigation of the sequence of phase transformations during crystallization of the $Sr_{1.5}La_{0.5}FeMoO_{6-\delta}$ solid solution and the establishment of correlations between synthesis modes and the degree of phase transformations of the synthesized compounds, using a stoichiometric mixture of oxides MoO₃ + $0.25La_2O_3 + 0.5Fe_2O_3 + 1.5SrCO_3$, a multi-stage system was determined of the double perovskite crystallization process. This is due to the complexity of phase transformations due to the occurrence of serial-parallel chemical reactions and low phase formation kinetics. It has been established that to reduce the influence of intermediate reaction products, it is necessary to use the Sr_{0.5}La_{0.5}FeO₃ and SrMoO₄ precursors. Based on the investigation of the temperature dependences of the degree of phase transformations and their derivatives, combined heating modes were optimized. As a result of using combined synthesis modes, it was possible to obtain a single-phase compound Sr_{1.5}La_{0.5}FeMoO_{6-d} from the precursors Sr_{0.5}La_{0.5}FeO₃ and SrMoO₄ with a Curie temperature of 450 K, a magnetization value of 40.9 A \cdot m² \cdot kg⁻¹ at T = 77 K in a magnetic field 0.86 T and with the presence of superstructural ordering of iron and molybdenum cations (82%).

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