



Original Article

Phase Transformations and Magnetic Properties of Non-Stoichiometric Strontium Ferromolybdate

Nikolay Kalanda¹, Alexander Petrov¹, Sholpan Yespenbetova^{2,*}, Dmitry Kiselev³, Andrey Yudenkov⁴, Deleg Sangaa⁵, Munkhtsetseg Sambuu⁶, Sergei Lazarouk⁷, Esenzhol Nazarov^{2,*}, Aleksandr Doroshkevich^{8,9}, Nurbol Appazov², Altyn Altynbassova⁸

¹SSPA "Scientific-Practical Materials Research Centre of the NAS of Belarus", 220072 Minsk, Belarus

²Korkyt Ata Kyzylorda University, 120000, Kyzylorda, Kazakhstan

³National University of Science and Technology "MISIS", 119049 Moscow, Russia

⁴State Center "Belmicroanalysis" of the Affiliate & Design Center "Belmicrosystems" of JSC "INTEGRAL", 220108 Minsk, Belarus

⁵Institute of Physics and Technology, Mongolian Academy of Sciences, 13330 Ulaanbaatar, Mongolia

⁶Natural Sciences Division, National University of Mongolia, 14201 Ulaanbaatar, Mongolia

⁷Belarusian State University of Informatics and Radioelectronics, 220013 Minsk, Belarus

⁸Joint Institute for Nuclear Research, 141980 Dubna, Russia

⁹Dubna State University, Dubna 141980, Russia

ARTICLE INFO

Article history

Submitted: 2025-10-01

Revised: 2025-11-17

Accepted: 2025-12-28

ID: CHEMM-2512-2053

DOI: [10.48309/chemm.2026.563461.2053](https://doi.org/10.48309/chemm.2026.563461.2053)

KEYWORDS

Magnetic metal-oxide compound

X-ray phase analysis

Crystallization kinetics

Phase transformation extent

Magnetization

ABSTRACT

This study investigates the phase transformation dynamics during the synthesis of non-stoichiometric strontium ferromolybdate ($\text{Sr}_2\text{Fe}_{1.2}\text{Mo}_{0.8}\text{O}_{6-\delta}$, SFMO) via solid-state reaction from SrCO_3 , Fe_2O_3 , and MoO_3 . Intermediate phases SrFeO_3 and SrMoO_4 form sequentially between 500–850 K and hinder complete SFMO crystallization due to kinetic limitations at high temperatures (≥ 1270 K). To overcome this, combined synthesis modes involving controlled heating rates and intermediate grinding were developed, enabling the production of single-phase SFMO with 89% Fe/Mo superstructural ordering. Magnetic characterization revealed that reduced cation ordering increases antiferromagnetic clustering, suppressing long-range ferrimagnetic order and lowering magnetization in field-cooling measurements. Zero-field-cooling data confirmed superparamagnetic behavior, indicating magnetic inhomogeneity with coexisting superparamagnetic nanoparticles and ferrimagnetic grains. The results demonstrate that precise control of synthesis conditions is essential to minimize kinetic barriers, suppress defect formation, and achieve reproducible magnetic properties in SFMO, a promising candidate for spintronic applications.

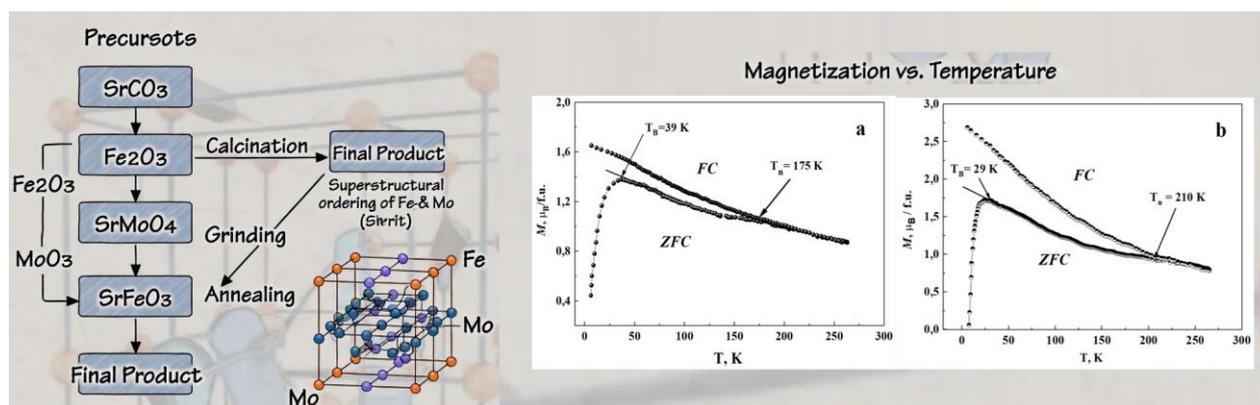
* Corresponding author: Sholpan Yespenbetova, Esenzhol Nazarov

E-mail: yespenbetova@korkyt.kz; nazarov_e@korkyt.kz

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GRAPHICAL ABSTRACT



Introduction

Recently, the non-stoichiometric SFMO solid solution having a double perovskite structure has attracted the attention of researchers in the field of promising spintronic materials [1-4]. This solid solution exhibits chemical stability in a reducing atmosphere, high Curie temperatures (~ 400 - 420 K), and a high degree of spin polarization of conduction electrons, reaching approximately 100% at sufficiently low magnetic fields of < 0.5 T, provided superstructural ordering of iron and molybdenum cations occurs in the material [5-9]. Due to its practically important and, in some respects, unique magnetic and magneto transport properties, this compound is superior in several parameters to the known systems based on manganites, nickelates and cobaltites [10].

High-temperature annealing at $T \sim 1,370 - 1,470$ K in reducing gas media ($5\% \text{H}_2/\text{Ar}$, $1\% \text{H}_2/\text{Ar}$, and Ar) has been used for the synthesis of SFMO with the solid-state reaction method [8,9,11,12]. The initial components for the synthesis of SFMO were simple metal oxides Fe_2O_3 and MoO_3 , as well as strontium carbonate SrCO_3 [11,12]. Before the compound synthesis, they were mixed in a vibrating mill for 2 h with ethanol added to obtain a homogeneous mixture of the initial reagents in a stoichiometric ratio. The powder was dried in air at 343 K and subsequently annealed at 1,050–1,100 K in air for 24 h, followed by pressing into tablets and annealing at 1,450–1,500 K in a $5\% \text{H}_2/\text{Ar}$ gas mixture for 12-15 h [11-14]. Strontium ferrimolybdate was synthesized in a reducing medium to avoid double perovskite

phase decomposition. The authors of some earlier works [11,12,14] pointed out that the average grain size of the powders was on the order of several microns. This is because this synthesis method involves high-temperature annealing, leading to an increase in the size of $\text{Sr}_2\text{Fe}_{1+x}\text{Mo}_{1-x}\text{O}_{6-\delta}$ grains in the synthesized powder.

Obtaining single-phase SFMO with the required anionic and cationic zero-dimensional defect content is a difficult task due to uncontrolled defect formation processes in the double perovskite structure [15-17]. Analysis of previous data on SFMO indicates the non-repeatability and even inconsistency of the results of magnetic and electrophysical investigations [1-3,16,17]. As a result, the physicochemical properties of double perovskite obtained by various researchers differ [1,12,17-20]. This inconsistency can be attributed to disregarding the complexity of the SFMO compound synthesis process caused by multistage phase transformations, slow phase formation kinetics and low mobility of Fe^{3+} and Mo^{5+} cations [1,9,11-16]. These investigations revealed, in fact, no strict relationship between the physical characteristics of the material and its synthesis conditions.

Based on the above, it becomes obvious that obtaining single-phase SFMO with reproducible physico-chemical properties is impossible without detailed analysis of high-temperature phase transformations at every stage of its synthesis and mandatory studies of phase transformation kinetics and degree during crystallization. Fulfilling the above requirements will allow for a controlled change in the phase composition of synthesized ceramics with optimal

magnetic properties, and that is the aim of this work.

Experimental

SrCO₃, Fe₂O₃, and MoO₃ reagents (99.5% purity, purchased from the Sigma-Aldrich company) were used as raw materials for studying the sequence of phase transformations in variable-composition SFMO compounds. A stoichiometric mixture of the raw reagents was mixed and ground in a PM 100 ball mill from Retsch GmbH in ethanol for 3 h. The resulting powder has been pressed into 10 mm diameter, 4-5 mm thick tablets. The material was annealed at 300-1,470 K in polythermal mode in a continuous 5% H₂/Ar gas mixture flow at various heating rates followed by room temperature quenching.

X-ray diffraction analysis (XRD) was performed on a Bruker AXS D8 Focus diffractometer with a LynxEye detector using Cu K α radiation. The measurements were recorded at a standard rate of 1.5 2 θ /min. The phase composition of solid-phase synthesis products was quantitatively characterized based on XRD data using the PowderCell and FullProf software with the Rietveld method [21,22].

Magnetization of the samples was measured as a function of temperature in a 0.01 T magnetic field in the 4.2 K to 320 K range using a Liquid Helium-Free High-Field Measurement System Vibrating Sample Magnetometer (Cryogenic Ltd., UK) in various modes: "zero magnetic field cooling" (ZFC) and "cooling in a magnetic field" (FC).

Results and Discussion

Phase composition analysis of the charge, *i.e.*, a mixture of raw reagents having the SrCO₃ + 0.6Fe₂O₃ + 0.8MoO₃ stoichiometric composition showed that with an increase in temperature, the SrMoO₄ phase forms initially in the 500–600 K range, and then the SrFeO₃ and SFMO complex compounds form almost simultaneously in the 800 – 850 K range (Figure 1a). The amount of double perovskite increases with an increase in temperature, while the concentration of strontium molybdate in the mixture decreases more slowly than the concentration of strontium ferrite (Figure

1a). It was found that in the high-temperature 1,270 – 1,470 K range, the growth rate of SFMO decreases at transformation degrees $\alpha \geq 94\%$. The changes in the contents of the SrFeO₃ and SgMoO₄ phases are but slight, with the $\alpha(T)$ curve showing a plateau. At T = 1,370 K, the main reflection of the SrMoO₄ phase attenuates, and the SrMoO₄ content in the sample is as low as within 5% (Figure 1b). With a further increase in temperature to 1,470 K, the composition of the material changes toward higher magnet concentrations, *i.e.*, SFMO- $\alpha(T)$ = 97%, and lower strontium molybdate, SrMoO₄ = 3%. This indicates a hindrance of the solid-state reactions that produce the strontium ferromolybdate solid solution. One can therefore assume that the forming product layer has low mobility of cations and anions, thus creating kinetic impedance to chemical reactions during the synthesis of the single-phase strontium ferromolybdate compound. In this case, the mutual diffusion of the chemical elements increases the thickness of the solid-state interface, contributing to greater diffusion paths of the reagents to the reaction zone. As a result, the heterogeneous reaction transitions from chemical adsorption mode to diffusion mode.

Analysis of the phase transformation dynamics showed that the main concomitant compounds during the crystallization of the SFMO double perovskite solid solution are SrMoO₄ and SrFeO₃. This indicates that the SrMoO₄ and SrFeO₃ compounds are structure-forming ones for the strontium ferromolybdate solid solution. Thus, since the SrMoO₄ and SrFeO₃ complex oxides are concomitant in the initial charge over almost the entire annealing temperature range during the compound synthesis, it is important to develop such single-phase SFMO synthesis modes for which the most rapid dissolution of the above compounds occurs. In the light of the above, the effect of heating rates was considered on the quantitative composition of the phases.

It was noted that with an increase in the heating rate (v), the amplitude of $\alpha_{\max}=f(T)_{\vartheta=\text{const}}$ decreases for both the SrFeO₃ and SrMoO₄ compounds. Furthermore, the values of $\alpha_{\max}=f(T)_{\vartheta=\text{const}}$ shift towards higher temperatures with increasing heating rate, which also indicates the presence of

kinetic impedance during their crystallization (Figure 2).

Analysis of the decomposition rates of the double oxides showed that the values of the minima $(d\alpha/dt)_{\min} = -0.17$ for SrMoO_4 and $(d\alpha/dt)_{\min} = -$

0.18 for SrFeO_3 are almost equal at $T = 1,223$ K and $T = 1,213$ K, respectively, for a 1 °C/min heating rate (Figures 3 and 4).

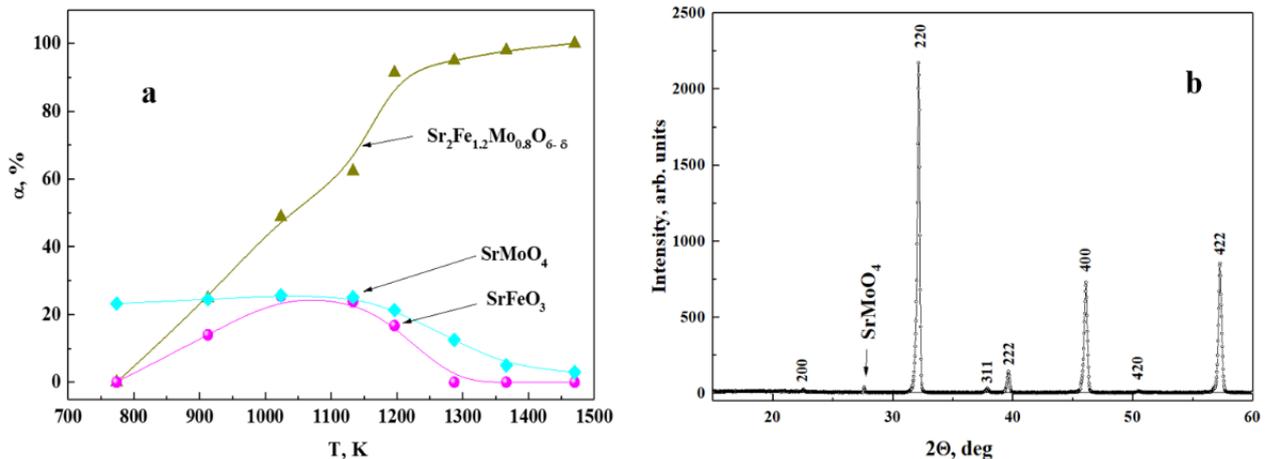


Figure 1: (a) Transformation degree of SrMoO_4 , SrFeO_3 , and SFMO phases as a function of temperature for samples synthesized from MoO_3 , Fe_2O_3 , and SrCO_3 reagent mixture; (b) XRD pattern of sample synthesized from SrCO_3 , Fe_2O_3 , and MoO_3 reagent mixture in a continuous 5% H_2/Ar flow at 5 °C/min heating rate to $1,470$ K

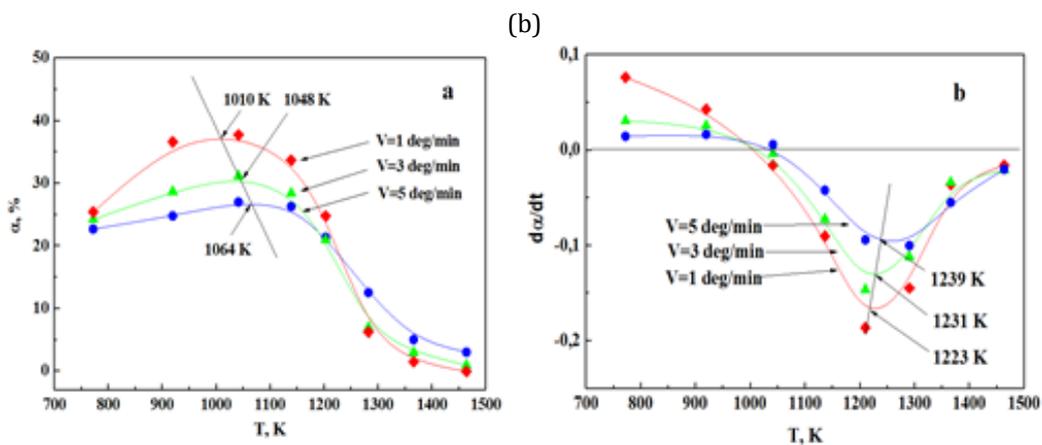


Figure 2: (a) Transformation degree $\alpha=f(T)_{9=\text{const}}$ and (b) growth rate $d\alpha/dt=f(T)_{9=\text{const}}$ of SrMoO_4 compound as a function of temperature for samples synthesized at different heating rates

Analysis of $\alpha=f(T)_{9=\text{const}}$ showed that with an increase in the heating rate, the increase in the SFMO phase transformation degree slows down, reaching a plateau at above $T=1,208$ K. However, the maximum strontium ferromolybdate growth rate was observed at $T=985$ K for samples synthesized in polythermal mode at $v = 1$ °C/min. At other heating rates, the maximum strontium ferromolybdate growth rate shifts to higher temperatures and reaches a 100% maximum at $T=1,470$ K (Figure 4). Considering the series-parallel reactions occurring simultaneously with

the formation of various products, primarily, SrFeO_3 and SrMoO_4 , the SFMO crystallization rate was determined by the slower stage.

One can assume that the limiting stage of the double perovskite growth is the bulk diffusion of reagents through the reaction product layer, leading to an increase in the diffusion path of the reagents. Therefore, to increase the growth rate of SFMO, it is necessary to shorten the diffusion path of the initial reagents into the reaction zone by avoiding the formation of intermediate reaction

products during the crystallization of nonstoichiometric strontium ferromolybdate. The above data suggest that to reduce the phase formation processes and increase the decomposition rate of the intermediate products of the SrFeO_3 and SrMoO_4 reaction during the crystallization of the strontium ferromolybdate solid solution, one should consider the dynamics

of the phase transformations and use combined heating conditions. For example, in the low-temperature region, where the formation and growth of the double oxides occur, the heating rate should be set the highest, and in the high-temperature region, where the formed by-products dissolve, the heating rate should be lowered.

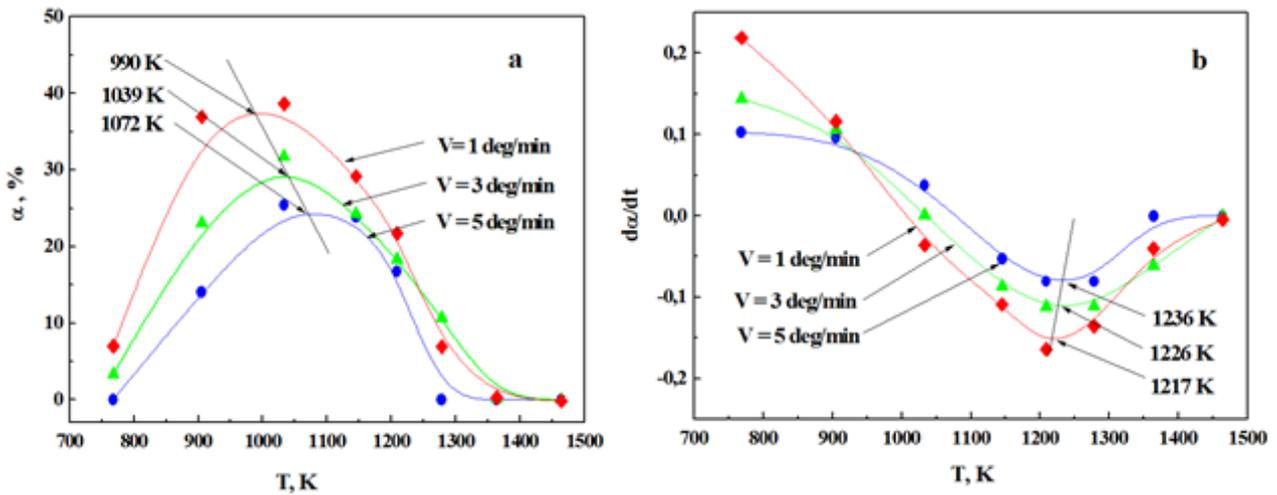


Figure 3: (a) Phase transformation degree $\alpha=f(T)_{9=\text{const}}$ and (b) growth rate $d\alpha/dt=f(T)_{9=\text{const}}$ of SrFeO_3 compound as a function of temperature for samples synthesized from stoichiometric $\text{SrCO}_3 + 0.6\text{Fe}_2\text{O}_3 + 0.8\text{MoO}_3$ mixture in a continuous 5% H_2/Ar gas mixture flow at various heating rates to 1,470 K

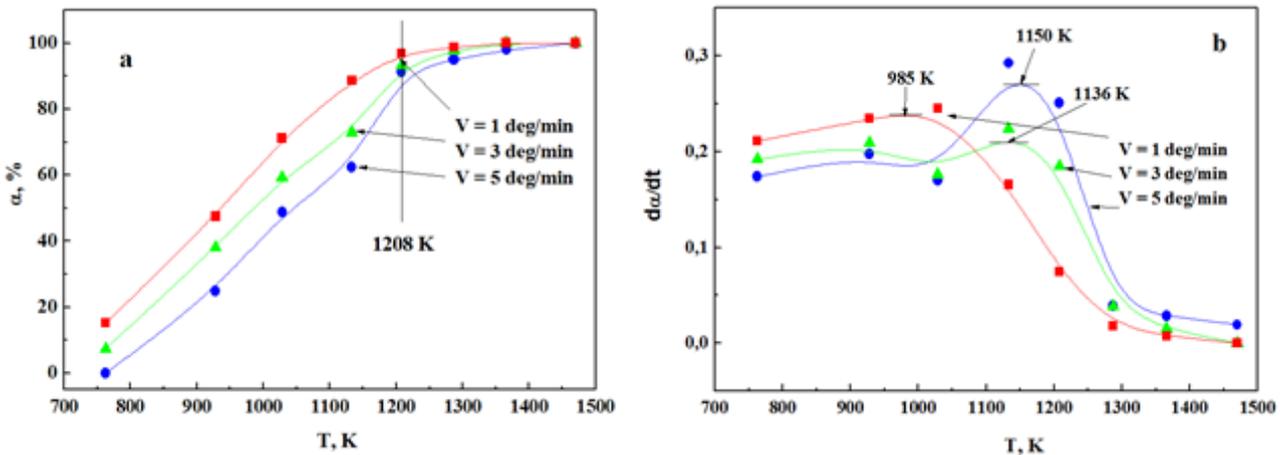


Figure 4: (a) Phase transformation degree $\alpha=f(T)_{9=\text{const}}$ and (b) growth rate $d\alpha/dt=f(T)_{9=\text{const}}$ of SFMO compound as a function of temperature for samples synthesized from stoichiometric $\text{SrCO}_3 + 0.6\text{Fe}_2\text{O}_3 + 0.8\text{MoO}_3$ mixture in a continuous 5% H_2/Ar gas mixture flow at various heating rates to 1,470 K

In this case, the observed almost simultaneous involvement of SrMoO_4 and SrFeO_3 in the reaction with the formation and growth of SFMO is most likely associated with the implementation of a magnet crystallization mechanism for which kinetic impedance is minimized and, accordingly,

the growth rate of strontium ferromolybdate increases. In this case, the rate of the overall transformation is determined by the interaction rate of the reagents at the interface with the SFMO grains, which is controlled by the thickness of the intermediate reaction product layer.

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

At the first stage, preliminary polythermal synthesis was performed at a heating rate of 5 °C/min to T=1,208 K in a continuous 5%H₂/Ar gas mixture flow, followed by a 10 h exposure. This temperature was chosen since it is higher than the maximum growth temperatures of the SrFeO₃ and SrMoO₄ compounds, but lower than the temperature at which the growth rate of the SFMO solid solution is the lowest.

At the second stage, to increase the reactivity of the mixture and the diffusion mobility of the reagents, the former layer of the reaction product was crushed and homogenized, and a high dispersion of the charge was achieved by fine vibration grinding in ethanol for 2 h.

At the third stage, in order to achieve the most rapid decomposition of the SrFeO₃ and SrMoO₄

intermediate phases and the α=100% transformation degree for SFMO, polythermal synthesis was carried out in the T=1,210-1,470 K range in a continuous 5%H₂/Ar gas mixture flow at a heating rate of 1 °C/min, in which the values of (dα/dt)_{min} for the SrMoO₄ and SrFeO₃ phases are included, and reaching the maximum α=100% for strontium ferromolybdate.

The synthesis of SFMO from a SrCO₃ + 0,6Fe₂O₃ + 0.8MoO₃ reagents mixture in a continuous 5%H₂/Ar gas mixture flow at T=1,470 K for 20 h proved to produce a single-phase magnet (sample No. 1) without superstructural ordering of Fe/Mo cations (Figure 5a). For combined synthesis modes, single-phase strontium ferromolybdate (sample No. 2) was obtained, with superstructural ordering of Fe/Mo cations (P = 89%) (Figure 5b).

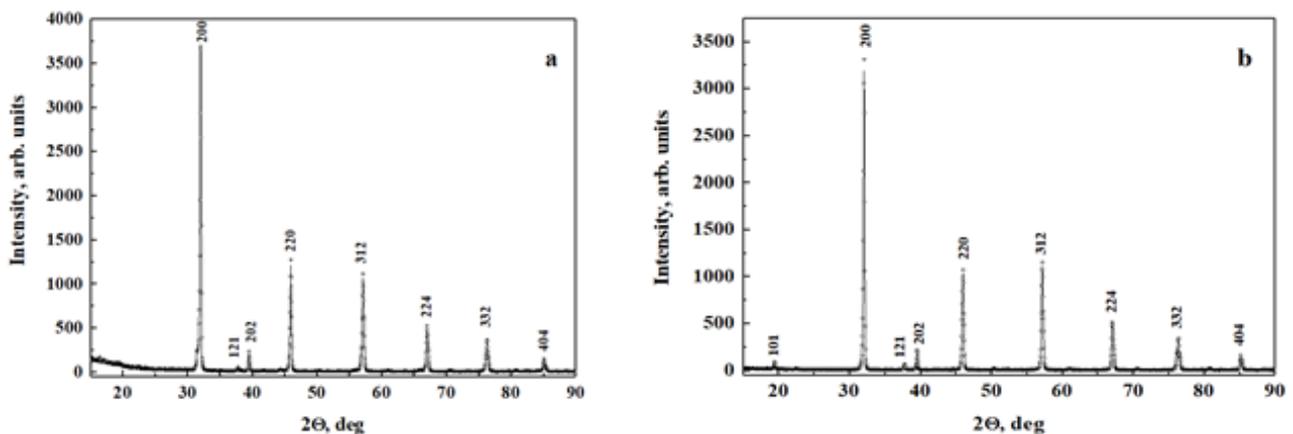
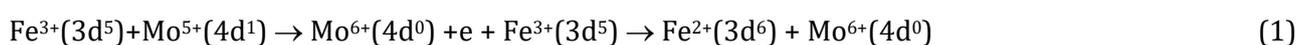


Figure 5: (a) XRD patterns of SFMO, samples synthesized in a continuous 5%H₂/Ar gas mixture flow at T=1,470 K for 20 h (sample No. 1) (b) and in combined synthesis modes (sample No. 2)

Since the magnetic moment of the SFMO double perovskite is determined by the ferrimagnetic ordering of the magnetic moments (*M*) of high-spin Fe³⁺(3d⁵,t_{2g}³↑↑↑e_g²↑↑, S=5/2) and Mo⁵⁺(4d¹,t_{2g}¹↓, S=1/2) cations, the lower M(No.1)_{4.2K} values are caused by the transition of some Fe cations to the intermediate spin t_{2g}⁵e_g¹ or low spin t_{2g}⁶e_g⁰ states (Figure 6). Moreover, in this case the material

contains a mixture of high- and low-spin cations, due to the presence of various types of point defects, primarily, antistructural [Fe_{Mo}] and [Mo_{Fe}] ones [2,5,7]. At the same time, the presence of antistructural defects stimulates the redistribution of electron density with a change in the electronic configuration of some iron and molybdenum ions following the Equation 1:



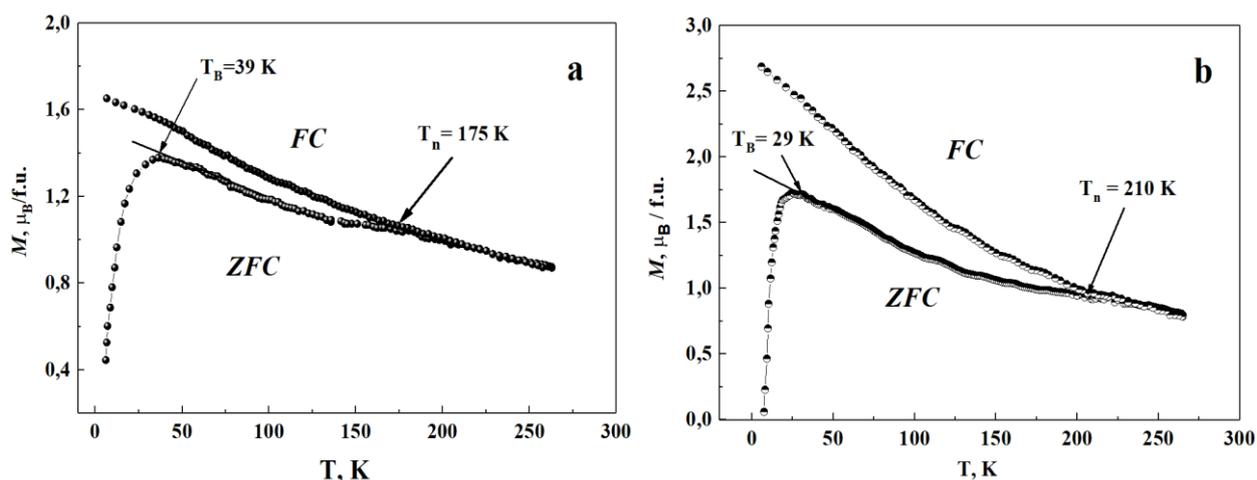


Figure 6: Temperature dependences of the magnetization of SFMO samples synthesized: (a) in a continuous 5% H_2 /Ar gas mixture flow at $T=1,470$ K for 20 h (sample No. 1) (b) and in combined modes (sample No. 2)

Since the diamagnetic $Mo^{6+}(4d^0)$ cations do not participate in the exchange interactions, and only negative exchange interactions are possible between the $Fe^{2+}(3d^6)$ ions, which have a lower magnetic moment than the $Fe^{3+}(3d^5)$ ones, antiferromagnetic ordering of magnetic moments between the $Fe^{2+}(3d^6)$ ions occurs, leading to a decrease in the total magnetic moment. The presence of antiferromagnetic clusters in the compound reduces the likelihood of the formation of long-range ferrimagnetic ordering, which leads to splitting large ferrimagnetic domains into smaller ones due to the tendency of the system to minimize the free energy consisting of several components, *e.g.*, magnetostatic, magnetoelastic, exchange interaction and magnetic anisotropy. This statement is well confirmed by the experimental data on the magnetization as a function of temperature in ZFC and FC modes.

It should be noted that with decreasing P values of the samples, the $M_{ZFC}=f(T)_{B=const}$ and $M_{FC}=f(T)_{B=const}$ curves diverge at a lower temperature for the sample No. 1 ($T_n = 175$ K) than that for the sample No. 2 ($T_n = 210$ K), where T_n is the defreezing temperature of magnetic moments in grains of the maximum size. The obtained results indicate that the high superstructural ordering of Fe/Mo cations ($P = 89\%$) stabilizes the long-range ferrimagnetic order. The strong coupling of magnetic moments throughout the grain creates a rigid magnetic framework, the disordering of which requires significant thermal energy. This

"resistance" is manifested in the preservation of the divergence between the ZFC and FC curves up to 210 K, which indicates the ability of the system to "remember" the magnetic history and its "rigid" magnetic behavior.

1. At the low superstructural ordering, defects and antiferromagnetic (AFM) clusters predominate within the ferrimagnetic (FM) matrix. Uncompensated spins arise at their boundaries, which, under ZFC conditions, are locked in random, energetically unfavorable configurations, creating local anisotropies. Since these clusters are isolated, overcoming only the local energy barrier is sufficient to unlock them, rather than disrupting the long-range order. As a result, the system "forgets" its magnetic history (with that ZFC and FC converging) already at 175 K, since the sources of "memory" (locked spins) are easily unlocked by thermal fluctuations.

2. Thus, the observed effect confirms that as cationic ordering decreases, the role of magnetic inhomogeneity, local anisotropy, and clusters with competing magnetic interactions increases. "Unfreezing" such an inhomogeneous system requires less energy than destroying long-range order in an ordered sample.

An analysis of the temperature dependences of magnetization measured in the ZFC mode for the samples No. 1 and No. 2 revealed a sharp increase in magnetization in the temperature range of 4.2 K – T_B upon heating in a weak magnetic field of 0.01 T (Figure 6). The temperature T_B corresponds to

the blocking temperature at which the magnetic moments of particles in the superparamagnetic state are unfrozen. The sharp rise in the ZFC curve reflects the activation of the magnetic moments of the smallest particle fraction, indicating low coercivity ($H_c \rightarrow 0$) and a characteristic feature of superparamagnetic compounds [23-25]. This observed effect confirms the presence of a fraction of ultrafine particles in the samples, the sizes of which are below the critical value required for a stable ferrimagnetic state, leading to their transition to the superparamagnetic state. The obtained ZFC results also indicate the magnetic inhomogeneity of the material, which is a composite system in which superparamagnetic nanoparticles coexist with larger ferrimagnetic grains.

Conclusion

The dynamics of phase transformations in the SrFeO_3 , SrMoO_4 and SFMO compounds have been investigated. It was concluded that combined synthesis modes should be used to reduce the effect of intermediate reaction products, to minimize kinetic impedances and, hence, to increase the growth rate of single-phase strontium ferromolybdate with superstructural ordering of Fe/Mo cations. It was shown that with a decrease in the superstructural ordering degree of Fe/Mo cations, the magnetization measured in the FC mode decreases, and the number of antiferromagnetic clusters reduces the probability of long-range ferrimagnetic ordering increases. Analysis of the magnetization vs temperature curves measured in ZFC mode indicated the presence of superparamagnetism in the SFMO compound.

Conflict of Interest

No conflict of interest was reported by the authors of this study.

Authors' Contributions

All authors contributed to data analysis, drafting, and revising the article and agreed to be responsible for all aspects of this work.

Funding

The present work has been financially supported by the Belarusian Republican Foundation for Fundamental Research (Project No. F24MN-009); the Russian Scientific Foundation (Grant No. 24-19-00729); Kazakhstan – JINR Cooperation Project No. 351 2025, item 15.

ORCID

Nikolay Kalanda
<https://orcid.org/0000-0001-7679-4968>
 Alexander Petrov
<https://orcid.org/0000-0003-1208-5913>
 Sholpan Yespenbetova
<https://orcid.org/0000-0002-6488-6846>
 Dmitry Kiselev
<https://orcid.org/0000-0003-1069-4715>
 Andrey Yudenkov
<https://orcid.org/0009-0005-2259-0368>
 Deleg Sangaa
<https://orcid.org/0000-0003-3611-5531>
 Munkhtsetseg Sambuu
<https://orcid.org/0009-0005-6169-5293>
 Sergei Lazarouk
<https://orcid.org/0000-0002-6317-2112>
 Esenzhol Nazarov
<https://orcid.org/0000-0003-2368-6466>
 Aleksandr Doroshkevich
<https://orcid.org/0000-0002-1353-1965>
 Nurbol Appazov
<https://orcid.org/0000-0001-8765-3386>
 Altyn Altynbassova
<https://orcid.org/0009-0005-1212-7871>

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HOW TO CITE THIS ARTICLE

N. Kalanda, A. Petrov, S. Yespenbetova, D. Kiselev, A. Yudenkov, D. Sangaa, M. Sambuu, S. Lazarouk, E. Nazarov, A. Doroshkevich, N. Appazov, A. Altynbassova Phase Transformations and Magnetic Properties of Non-Stoichiometric Strontium Ferrumolybdate. *Chem. Methodol.*, 2026, 10(4) 428-437

DOI: <https://doi.org/10.48309/chemm.2026.563461.2053>

URL: https://www.chemmethod.com/article_238579.html