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Peculiarities of layered perovskite-related $GdSrFeO_4$ compound solid state synthesis

E.A. Tugova[a,b,*], V.V. Gusarov[b,a]

[a] *Laboratory of New Inorganic Materials, Ioffe Physical-Technical Institute of RAS, Saint-Petersburg 194021, Russia*
[b] *Department of Physical Chemistry, Saint-Petersburg State Institute of Technology (Technical University), Saint-Petersburg 190013, Russia*

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ABSTRACT

It is shown that $Sr_{1-x}Gd_xFeO_{3-\alpha}$ solid solution formation on initial stage of $GdSrFeO_4$ oxide solid state synthesis leads to the occurrence of stable secondary products, mainly, $Gd_2SrFe_2O_7$. The data investigation presents some ways of intermediate compounds formation avoidance by revising of solid state chemical reaction passing scheme.

1. Introduction

Obtaining of complex oxides with perovskite-related structure, including layered perovskite-related compounds, is conjugated with number of synthetic problems [1–10]. Mentioned previously difficulties are connected with thermal instability and multi-staged formation character of many from considered oxides [11–17]. Survey of literature on the data subject has shown the importance of mechanism and kinetics formation investigation to develop the optimal synthesis conditions of such complex compounds [18–20]. Besides, it should be taken into account, that formation of layered oxides with perovskite-related structure of Ruddlesden–Popper (RP)-type often comes through the formation of intermediate compounds, also including RP phases of different composition, which can be in some cases stable enough to prevent the yield of required products [21–25]. So, for instance, the occurrence of one-layered $GdSrFeO_4$ RP phase, which has not almost been investigated, under solid phase synthesis of two-layered RP-phase $Gd_2SrFe_2O_7$ [26] encouraged our work in the field of formation processes research with particular attention on mechanism formation investigation. Besides, the article [27] devoted to study of complex oxide $GdSrFeO_4$ kinetics formation at 1300 °C has not in full manner answered questions we were interested in. So, present work is aimed at the research of $GdSrFeO_4$ compound formation under solid state synthesis.

2. Experimental details

2.1. Synthesis technique

$GdSrFeO_4$ compound with RP type's structure was synthesized by solid state chemical reactions method under 1100–1500 °C temperature region in air. As starting materials strontium carbonate $SrCO_3$ (special purity grade 7-2 according to *TU* (specifications) 6-09-01-659-91) (the correction for the decarbonization of the $SrCO_3$ oxide was calculated on the thermogravimetric data, performed by MOM Q-1500C Paulik–Paulik–Erdey derivatograph (Hungary)), gadolinium oxide Gd_2O_3 (special purity grade with content of the main component, equal to 99.99%), preliminarily calcined at 1000 °C for 2 h and Fe_2O_3 oxide (analytical grade), dried at 300 °C for 2 h were used.

Reagent mixtures corresponding to the stoichiometry of $GdSrFeO_4$ compound were prepared by mechanical stirring at aqueous medium in a corundum mortar with a corundum pestle. The samples were pressed at 500 MPa into cylinders of 15 mm in diameter and 3–4 mm in thickness. Crucible with observed samples was entered into furnace of room temperature and then was heated up to the required temperature of given exposure, after which the quenching of samples at rapid pace to room temperature was followed.

2.2. Characterizations

The phase composition of the specimens was checked by X-ray powder diffraction (XRD) using the DRON-3 X-ray diffractometer with the Cu Kα-radiation ($\lambda = 154.178$ pm).

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[*] Corresponding author at: Laboratory of New Inorganic Materials, Ioffe Physical-Technical Institute of RAS, Saint-Petersburg 194021, Russia. Tel.: +7 812 2927902; fax: +7 812 2971017.
E-mail address: katugova@inbox.ru (E.A. Tugova).

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Table 1
Electron probe microanalysis data for the regions indicated in Fig. 2a–d, d'.

Sample (Fig.)[a]	Sintering temperature (°C)	Examined region	Components content (mol%)			Phases
			$FeO_{1.5}$	SrO	$GdO_{1.5}$	
a (2,3)	1100	P1	7.13	2.36	90.51	c–Gd_2O_3
		P2	9.82	9.71	80.48	c–Gd_2O_3
		P3	25.45	19.48	55.06	c–Gd_2O_3, $Sr_{1-x}Gd_xFeO_{3-\delta}$
		P4	34.63	33.53	31.84	$GdSrFeO_4$
		P5	51.42	39.34	9.25	$Sr_{1-x}Gd_xFeO_{3-\delta}$
		P6	50.18	41.99	7.83	
		P7	51.07	47.09	1.84	
b (2,3)	1200	P1	7.97	4.78	87.26	c–Gd_2O_3
		P2	12.65	3.61	83.74	c–Gd_2O_3
		P3	10.29	10.56	79.14	c–Gd_2O_3, $SrFeO_{3-\delta}$
		P4	21.83	14.32	63.86	c–Gd_2O_3, $Sr_{1-x}Gd_xFeO_{3-\delta}$, $Sr_4Fe_6O_{13}$
		P5	24.85	14.13	61.03	
		P6	21.61	19.28	59.11	c–Gd_2O_3, $Sr_{1-x}Gd_xFeO_{3-\delta}$
		P7	24.40	22.91	52.68	c–Gd_2O_3, $SrFeO_{3-\delta}$
		P8	36.38	22.54	41.09	Phase related to $Gd_2SrFe_2O_7$
		P9	37.50	30.05	32.45	$GdSrFeO_4$
		P10	34.91	32.40	32.68	
		P11	33.35	36.98	29.67	
		P12	51.32	39.17	9.51	$Sr_{1-x}Gd_xFeO_3$
c (2,3)	1300	P1	2.93	0.00	97.07	c–Gd_2O_3
		P2	2.32	1.36	96.33	c–Gd_2O_3
		P3	1.20	0.00	98.80	c–Gd_2O_3
		P4	8.57	9.35	82.08	c–Gd_2O_3, $SrFeO_{3-\delta}$
		P5	18.99	13.70	67.30	c–Gd_2O_3
		P6	34.62	25.76	39.62	$Sr_{1-x}Gd_xFeO_{3-\delta}$
		P7	34.57	35.62	29.81	$GdSrFeO_4$
		P8	31.43	32.62	35.95	
		P9	34.25	34.73	31.02	
		P10	45.60	41.91	12.48	c–Gd_2O_3, $SrFeO_{3-\delta}$
		P11	46.87	44.43	8.70	$Sr_{1-x}Gd_xFeO_3$
d (2,3)	1400	P1	2.63	1.78	95.59	c–Gd_2O_3
		P2	2.20	1.97	95.83	c–Gd_2O_3
		P3	10.58	6.28	83.15	c–Gd_2O_3, $Sr_{1-x}Gd_xFeO_{3-\delta}$ $Sr_4Fe_6O_{13}$
		P4	41.70	22.14	36.16	Phase related to $Gd_2SrFe_2O_7$
		P5	41.87	20.86	37.27	
		P6	36.00	31.29	32.71	$GdSrFeO_4$
		P7	36.56	30.51	32.92	
		P8	52.00	33.93	14.07	$Sr_{1-x}Gd_xFeO_{3-\delta}$
d' (3)		P1	43.96	20.94	35.10	Phase related to $Gd_2SrFe_2O_7$
		P2	43.14	22.96	33.90	
		P3	44.19	21.74	34.07	
		P4	44.00	21.40	34.60	
		P5	43.73	22.39	33.88	
		P6	37.02	32.09	30.89	$GdSrFeO_4$
		P7	37.65	31.55	30.80	
		P8	38.33	31.54	30.12	
		P9	37.70	31.80	30.51	
		P10	38.05	31.48	30.47	

[a] Number of sample corresponds to the number of figure.

The microstructure of the specimen, elemental composition and the composition of separate phases were analyzed by means of scanning electron microscopy (SEM) and energy-dispersive X-ray spectrometry (EDX) using the CamScan MV2300 coupled with the Oxford Link microprobe analyzer. The error in determining the elements content by this method varies with the atomic number and equals to ±0.3 mass% on average.

3. Results and discussion

X-ray and SEM/EDX data (Figs. 1 and 2a; Table 1) show the chemical solid state reaction passing in the initial mixture influenced by thermal treatment under 1100 °C during 5 h. At the same time $GdSrFeO_4$ oxide formation as well as fixing of source and intermediate compounds such as α-Fe_2O_3, solid solutions both based on cubic Gd_2O_3 and perovskite-related phases $GdFeO_3$, $SrFeO_{3-\delta}$, $Sr_3Fe_2O_6$, $Sr_4Fe_6O_{13}$ are observed. The compositions of formed under shown conditions phases are drifted on triangle and demonstrated in Fig. 3a. The existence at reactive medium mixture of phases based on $SrFeO_{3-\delta}$, $Sr_3Fe_2O_6$, $Sr_4Fe_6O_{13}$, belonging to individual binary system SrO–Fe_2O_3 are evidently related to the comparable formation rates of named above oxides under 1100 °C thermal treatment. This fact is confirmed by results, demonstrated at [28,29]. So, for example, $SrFeO_{3-\delta}$ oxide as is pointed out at [30] was synthesized by calcination of initial oxides under 1000 °C in air. Subject to [29] $Sr_4Fe_6O_{13}$ compound was the member of layered perovskite-related compounds and was formed and stable at 775–1225 °C temperature region. Sr_2FeO_4, $Sr_3Fe_2O_6$, $Sr_3Fe_2O_{7-\alpha}$ compounds, belonging to Ruddlesden–Popper phase's family were obtained by sintering under 900–1100 °C temperature according data, presented at [28,29,31]. It is worth mentioning in conformity with [28] there was the opportunity for forming the narrow limit region of solid solutions between $Sr_3Fe_2O_6$, $SrFeO_{3-\delta}$, $Sr_4Fe_6O_{13}$ and $SrFe_{12}O_{19}$ compounds.

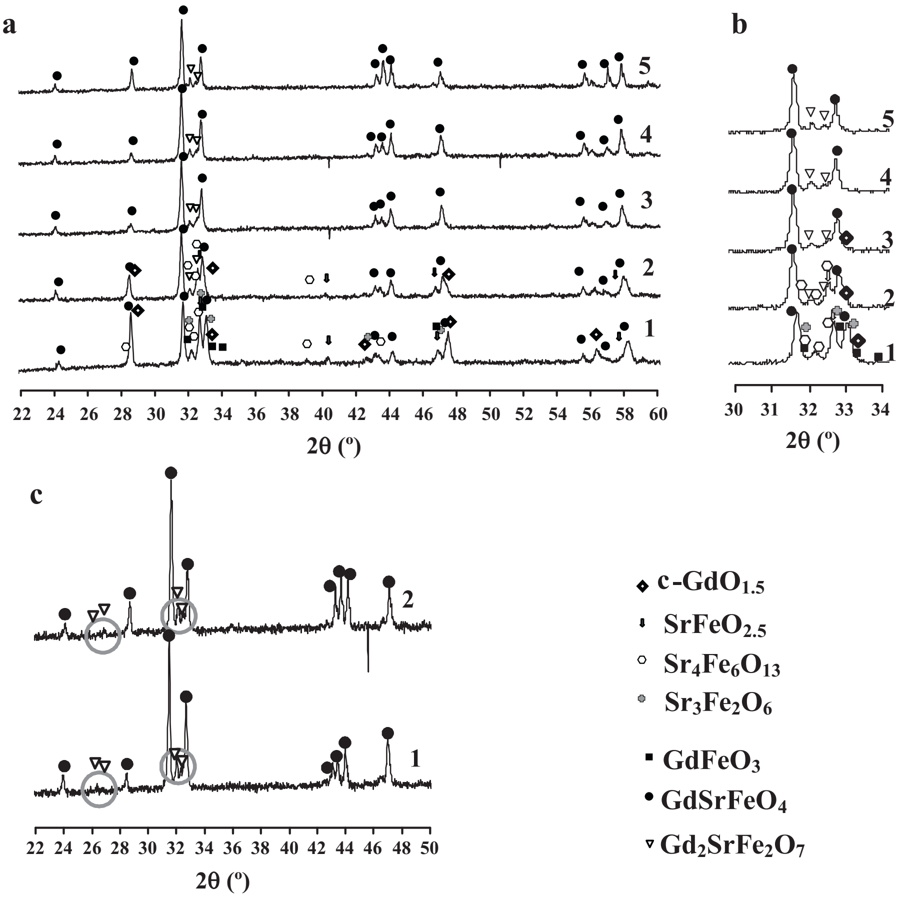

Fig. 1. X-ray diffraction patterns of initial mixtures corresponding to stoichiometry of $GdSrFeO_4$ compound after sintering in air at °C. (a, b) 1 – 1100, 2 – 1200, 3 – 1300, 4 – 1400, 5 – 1500 °C for 5 h; (c) 1 – 1300, 2 – 1400 °C for 24 h.

Thus, the high and comparable formation value rates of strontium–ferrite oxides lead to the set of phases, consisting of the targeted synthesis product as well as the mixture of strontium–ferrite oxides and initial reagents after 1100 °C thermal treatment (Fig. 3a).

Based on X-ray and SEM/EDX data (Figs. 1, 2b, and 3b; Table 1) samples heat treated under 1200 °C temperature contain the required synthesis product, amount of which is increased in comparison with the sample under 1100 °C heat treatment and the following fixed phases such as the solid solutions on cubic Gd_2O_3 and $Sr_4Fe_6O_{13}$, $SrFeO_{3-\delta}$ and also small amount of phase, closed on structure to $Gd_2SrFe_2O_7$. According to the literature data [26] the formation of $Gd_2SrFe_2O_7$ complex oxide was proceeded through the formation of perovskite-like phases of simplest structure types, like as $GdFeO_3$, $SrFeO_{3-\delta}$ oxides with perovskite structure and one-layered $GdSrFeO_4$ compound. Absence of $GdFeO_3$ oxide at the considered reactive medium in compliance with X-Ray and SEM/EDX data appears to be connected with the solid solution $Sr_{1-x}Gd_xFeO_{3-\delta}$ formation as the result of $GdFeO_3$ and $SrFeO_{3-\delta}$ interaction (Fig. 3a–d). Hence, the occurrence of compound, structurally related to $Gd_2SrFe_2O_7$ phase can be explained by coupling of previously formed $Sr_{1-x}Gd_xFeO_{3-\delta}$ or/and $Sr_4Fe_6O_{13}$ with phase based on cubic gadolinium oxide (Figs. 2 and 3b; Table 1). Thus, under 1200 °C heat treatment, some simultaneous and step-by-step solid state chemical reactions are proceeded, thereby forming the one-layered $GdSrFeO_4$ Ruddlesden–Popper phase and phase structurally related to $Gd_2SrFe_2O_7$ (Fig. 3b; Table 1). It should be noted, that absence of $Sr_3Fe_2O_6$ compound at the reaction medium can be connected with the reasons of comparable value rates of $SrFeO_{3-\delta}$, $Sr_3Fe_2O_6$, $Sr_4Fe_6O_{13}$, $SrFe_{12}O_{19}$ oxide's formation under 1000–1100 °C temperature region and the dominant process of $SrFeO_{3-\delta}$ formation under 1200 °C temperature.

Under 1300–1400 °C of samples heat treatment, as shown in Fig. 1 and Table 1, the existence of phases related on chemical composition to $GdSrFeO_4$ and $Gd_2SrFe_2O_7$ compounds, belonging to Ruddlesden–Popper series are observed. It worth mentioning, that the content of impurity product – phase of $Gd_2SrFe_2O_7$ type is increased with the raise of heat treatment exposure from 5 to 24 h (Fig. 1c). The got data is in the contradiction with the results, performed at [27], in which homogeneous $GdSrFeO_4$ compound has been obtained by method of solid state chemical reactions under 1300 °C calcination. But it should be noted, that both the view of shown X-ray powder diffraction patterns of samples, heat treated under 1300 °C during 3–34 h exposures and results, produced from Messbauer spectroscopy investigation, demonstrated the changes of compound's composition based on $SrFeO_{3-\delta}$ oxide during $GdSrFeO_4$ obtaining, point out the more complicated $GdSrFeO_4$ mechanism formation, than that of being reported at [27].

Results of scanning electron microscopy with the element phase analysis (Figs. 2c, d and 3c, d'; Table 1) also indicate the multi-staged $GdSrFeO_4$ compound formation process, consisting of the intermediate phase formation of variable composition in the SrO:Gd_2O_3:Fe_2O_3 = 1:0.5:0.5 mixture. The calcination's temperature rising till 1400–1500 °C increases the content of not aimed product – phase of $Gd_2SrFe_2O_7$ type (Fig. 1a and c). The formed as the result of solid state chemical interaction intermediate $Sr_{1-x}Gd_xFeO_{3-\delta}$ or/and $Sr_4Fe_6O_{13}$ phases under the reaction with phase based on Gd_2O_3 are, evidently, able to form the phase with the composition related to $Gd_2SrFe_2O_7$ (Fig. 3d; Table 1).

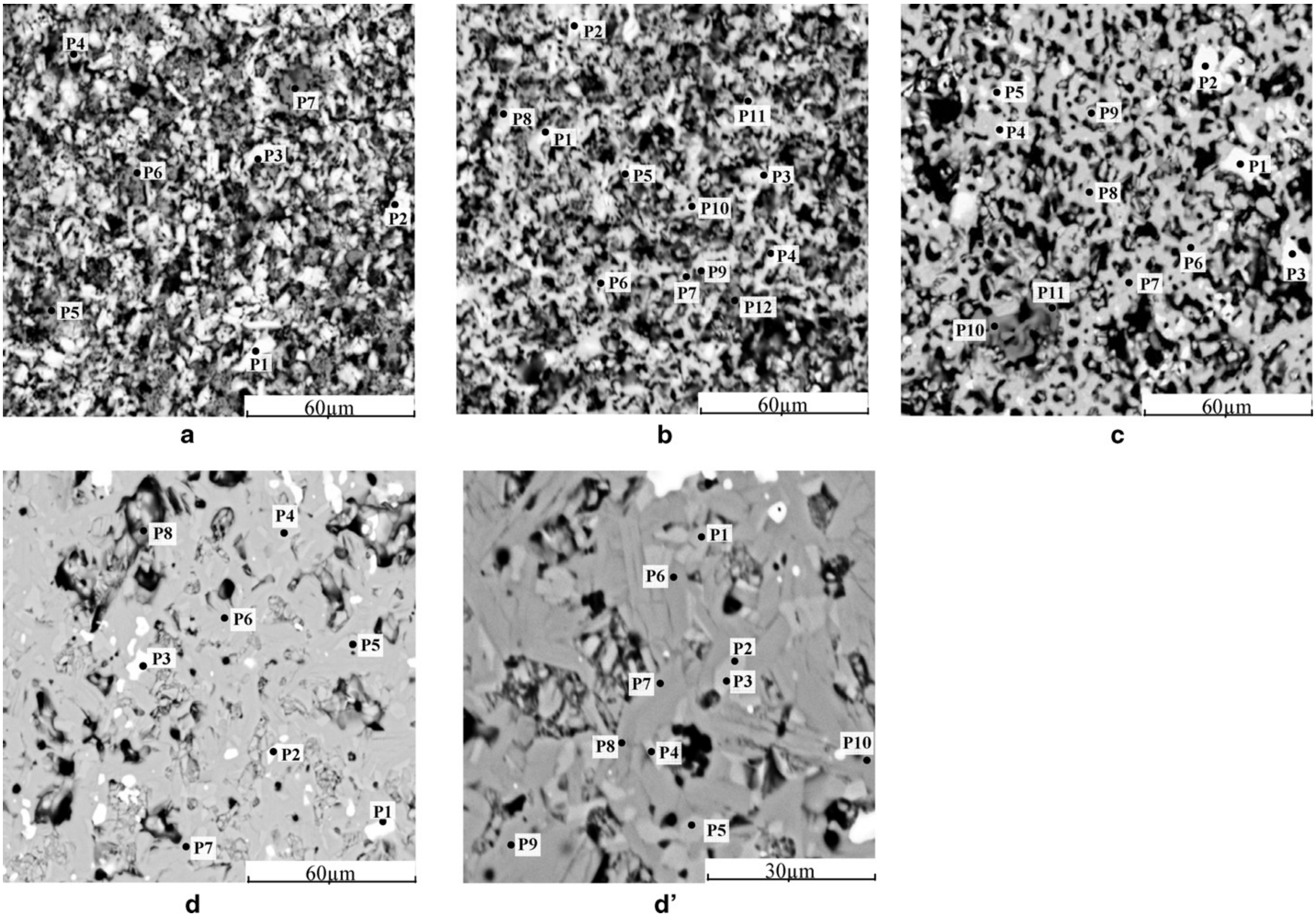

Fig. 2. SEM photographs of initial mixtures corresponding to stoichiometry of $GdSrFeO_4$ compound after sintering in air for 5 h at °C. (a) 1100, (b) 1200, (c) 1300, (d, d') 1400.

At the same time, it should be noted, that reaction of formed $Gd_2SrFe_2O_7$ compound with other system components would bring to $GdSrFeO_4$ obtaining but for the low diffusion rate of mentioned above compounds through the separating layer of $GdSrFeO_4$ (Fig. 3d–d').

The obtained presented in diagram form data (Fig. 3a–d) on solid state interaction character at $GdO_{1.5}$–SrO–$FeO_{1.5}$ system, allows to assume the following phase relation mechanism. On the prime stages under low synthesis temperature the reaction between initial components with the more reactive at considered system, iron oxide is observed. The mentioned above interaction leads to the intermediate compounds formation, presented by the following equations.

$$SrCO_3 = SrO + CO_2 \quad (1)$$

$$SrO + (0.25\text{–}0.5\delta)O_2 + FeO_{1.5} = SrFeO_{3-\delta} \quad (2)$$

$$3SrO + 0.5\delta O_2 + 2FeO_{1.5} = Sr_3Fe_2O_{6+\delta} \quad (3)$$

$$4SrO + 6FeO_{1.5} = Sr_4Fe_6O_{13} \quad (4)$$

$$GdO_{1.5} + FeO_{1.5} = GdFeO_3 \quad (5)$$

The next step in $GdSrFeO_4$ solid state synthesis is the reaction between the formed under prime stages intermediate compounds (Eqs. (1)–(5)) with both initial components and/or each other.

$$(1-x)SrFeO_{3-\delta} + xGdFeO_3 = Sr_{1-x}Gd_xFeO_{3-\alpha}, \quad \alpha = \delta(1-x) \quad (6)$$

$$Sr_{1-x}Gd_xFeO_{3-\alpha}. + (1-x)GdO_{1.5} = (1-2x)GdSrFeO_4 + xGd_2SrFe_2O_7 + 0.5(0.5-\alpha)O_2 \quad (7)$$

$$GdO_{1.5} + SrFeO_{3-\delta} = GdSrFeO_4 + (0.5-\delta)/2O_2 \quad (8)$$

$$6GdO_{1.5} + Sr_4Fe_6O_{13} = 2GdSrFeO_4 + 2Gd_2SrFe_2O_7 \quad (9)$$

All mentioned above stages (Eqs. (1)–(9)) are presented in Fig. 3a–d in the form of lines, linking points, corresponded to phase composition defined by methods of scanning electron microscopy with the element phase analysis and X-ray powder diffraction analysis. Thus the performed lines can be considered as trajectories of phase relation processes, which illustrate the target $GdSrFeO_4$ oxide mechanism formation. The main phase-formed process is the reaction between perovskite-related oxides $SrFeO_{3-\delta}$ and $GdFeO_3$, obtained by 2 and 5 equations, resulting in $Sr_{1-x}Gd_xFeO_{3-\alpha}$ occurrence (Eq. (6), line 1, Fig. 3a–d). It should be noted, that formation of variable composition phase $Sr_{1-x}Gd_xFeO_{3-\alpha}$ is fixed under all investigated temperatures (see line 1, Fig. 3a–d). The literature survey devoted to study of phase relations at $LnMO_3$–$SrMO_{3-\delta}$ (Ln = La, Gd; M = Fe, Mn) systems allows to make a conclusion on structural similarity of $Sr_{1-x}Ln_xFeO_{3-\alpha}$ phase to RP-family thereby having the opportunity to be the nucleus-formed precursors of latter phases [32–34]. Lines II, III and III' (Fig. 3a–d) demonstrate the interaction between initial reagent Gd_2O_3 with formed under previous stages $SrFeO_{3-\delta}$, $Sr_{1-x}Gd_xFeO_{3-\alpha}$, $Sr_4Fe_6O_{13}$ oxides, respectively. Thus, $GdSrFeO_4$ compound is obtained by the way of Gd_2O_3 with $SrFeO_{3-\delta}$ reaction as shown in Fig. 3a–d and expressed by Eq. (8). Lines III and III' illustrate the interaction between Gd_2O_3 oxide with solid solution $Sr_{1-x}Gd_xFeO_{3-\alpha}$ and perovskite-related $Sr_4Fe_6O_{13}$

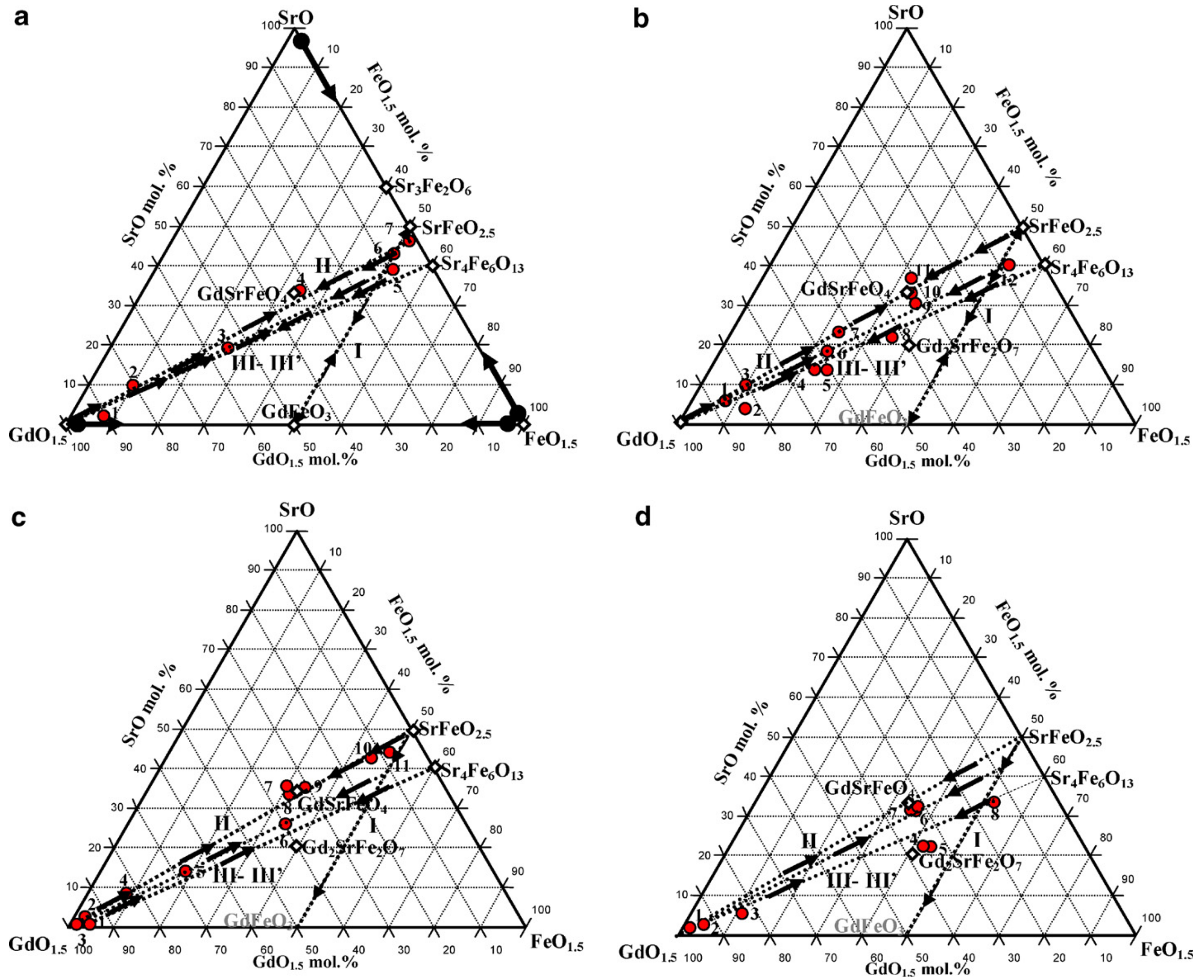

Fig. 3. Phase formation schemes, describing processes which are taken place under heat treatment of initial mixture of SrO:Gd_2O_3:Fe_2O_3 = 1:0.5:0.5 composition at◦C. (a) 1100, (b) 1200, (c) 1300, (d) 1400. *Note*: ◇, phases, fixed by X-ray analysis; ●, phases, fixed by SEM/EDX method.

oxide, respectively, which leads to occurrence of phases occupying the intermediate place on composition between one-layered $GdSrFeO_4$ and two-layered $Gd_2SrFe_2O_7$ and having the structural type of RP-phases (Table 1; Figs. 1 and 3). The formed under reactions (7) and (9) RP-phases should have structure with alternated one and two-layered perovskite-like blocks. Evidently, such compounds are unstable and are fallen to pieces on $GdSrFeO_4$ and $Gd_2SrFe_2O_7$, the increased content of those compounds can serve the demonstration of suggested mechanism (Figs. 1 and 3). At the same time the way of simultaneous formation of $GdSrFeO_4$ and $Gd_2SrFe_2O_7$ compounds under reactions (8) and (9) is possible too.

4. Conclusion

In conclusion, it should be noted, that $Sr_{1-x}Gd_xFeO_{3-\alpha}$ solid solution formation on initial stages of $GdSrFeO_4$ compound solid state synthesis is the main reason of occurrence of phase on composition related to $Gd_2SrFe_2O_7$. The stable existence of foreign substance can be explained both by the complication of components mass carry through the layer of formed aimed product and relatively high stability of RP-phase based on $Gd_2SrFe_2O_7$. $GdSrFeO_4$ oxide occurrence under $Gd_2SrFe_2O_7$ compound solid state synthesis [26] seems like similar to mentioned above situation.According to the features of solid state interaction between Fe_2O_3, $SrCO_3$ and Gd_2O_3 oxides mentioned above to avoid the secondary stable phase formation under $GdSrFeO_4$ solid state synthesis the reaction passing scheme should be revised. This purpose can be reached for instance, by varying the different precursors, by changing the dispersibility rate and homogeneity level of all reactive composition and also by entering of small excess of this or that reagent, etc., i.e. by modifying the order and state of forming under solid state reaction intermediate products.

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