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Thermal Behaviour of $\text{Sm}_{0.5}\text{R}_{0.5}\text{FeO}_3$ ($R = \text{Pr}, \text{Nd}$) Probed by High-Resolution X-ray Synchrotron Powder Diffraction

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Abstract

Mixed ferrites $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ with orthorhombic perovskite structure isotypic with GdFeO_3 were synthesized by solid-state reaction technique in air at 1473 K. Structural parameters obtained at room temperature prove a formation of continuous solid solutions in the $\text{SmFeO}_3\text{--PrFeO}_3$ and $\text{SmFeO}_3\text{--NdFeO}_3$ pseudo-binary systems. $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ show strongly anisotropic nonlinear thermal expansion: thermal expansion in the b direction is twice lower than in the a and c directions. The average linear thermal expansion coefficients of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ in the temperature range of 298–1173 K are in the limits of $(9.0\text{--}11.1) \times 10^{-6} \text{ K}^{-1}$, which is close to the values reported for the parent $R\text{FeO}_3$ compounds. Subtle anomalies in the lattice expansion of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ detected at 650–750 K reflect magnetoelastic coupling at the magnetic ordering temperature T_N .

Keywords: Mixed rare earth ferrites, Perovskites, Crystal structure, Thermal expansion, Magnetoelastic coupling

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Background

Complex oxides with perovskite structure $R\text{FeO}_3$, where R is the rare earth (RE), represent an important class of functional materials. The $R\text{FeO}_3$ -based materials are used as electrodes in solid oxide fuel cells, as catalysts, gas sensory materials and semiconductor ceramics [1–6]. Complementary, the interest in the rare earth ferrites is stimulated by their interesting fundamental physical properties, such as spin-reorientation transitions at 80–480 K and the para- to antiferromagnetic transitions at 620–750 K [7–10]. Just recently, the interest to RE ferrite perovskites was renewed due to reported multiferroic properties of NdFeO_3 , SmFeO_3 and other $R\text{FeO}_3$ compounds [11–13]. At room temperature (RT), all RE orthoferrites adopt orthorhombic perovskite structure isotypic with GdFeO_3 [14, 15]. No structural phase transitions were reported in the literature for $R\text{FeO}_3$ compounds, with an exception of LaFeO_3 , which undergoes a high-temperature (HT) transition to rhombohedral structure at 1220–1280 K [16, 17]. Orthorhombic $R\text{FeO}_3$ perovskites show strongly anisotropic

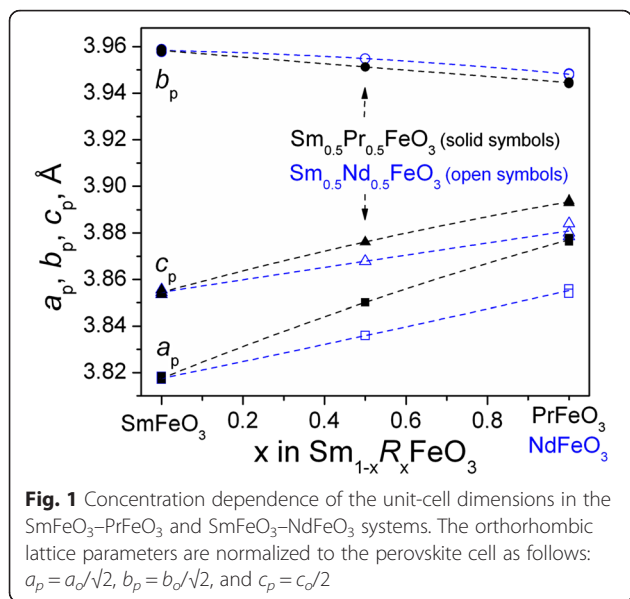
thermal expansion: the expansivity in the b direction in the $Pbnm$ setting is ca. two times lower than in the a and c directions. Subtle anomalies in the lattice expansion of PrFeO_3 and SmFeO_3 are observed in the b direction at 600–800 K, which is indicative for magnetoelastic coupling at the magnetic ordering temperature T_N [18, 19]. In ref. [9], it was shown that the spin-reorientation transition in NdFeO_3 between 100 and 200 K is associated with changes of the b -lattice parameter, which has a broad local minimum in the spin-reorientation region near 160 K. However, no lattice anomalies in NdFeO_3 were found around the Néel temperature of 687 K in [10].

The aim of the present work is the detail study of the thermal behaviour of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ in order to reveal the possible magnetoelastic coupling in these mixed perovskite ferrites.

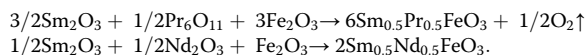
Methods

Polycrystalline samples with nominal compositions $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ have been prepared from stoichiometric amounts of constituent oxides Sm_2O_3 , Pr_6O_{11} , Nd_2O_3 and Fe_2O_3 by solid-state

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reaction technique according to the following reaction schemes:



Precursor oxides were ball-milled in ethanol for 5 h, dried, pressed into pellets and annealed in air at 1473 K for 20 h. The as-obtained product was repeatedly re-ground and annealed at 1473 K for 20 h and, after that, slowly cooled to RT for 20 h.

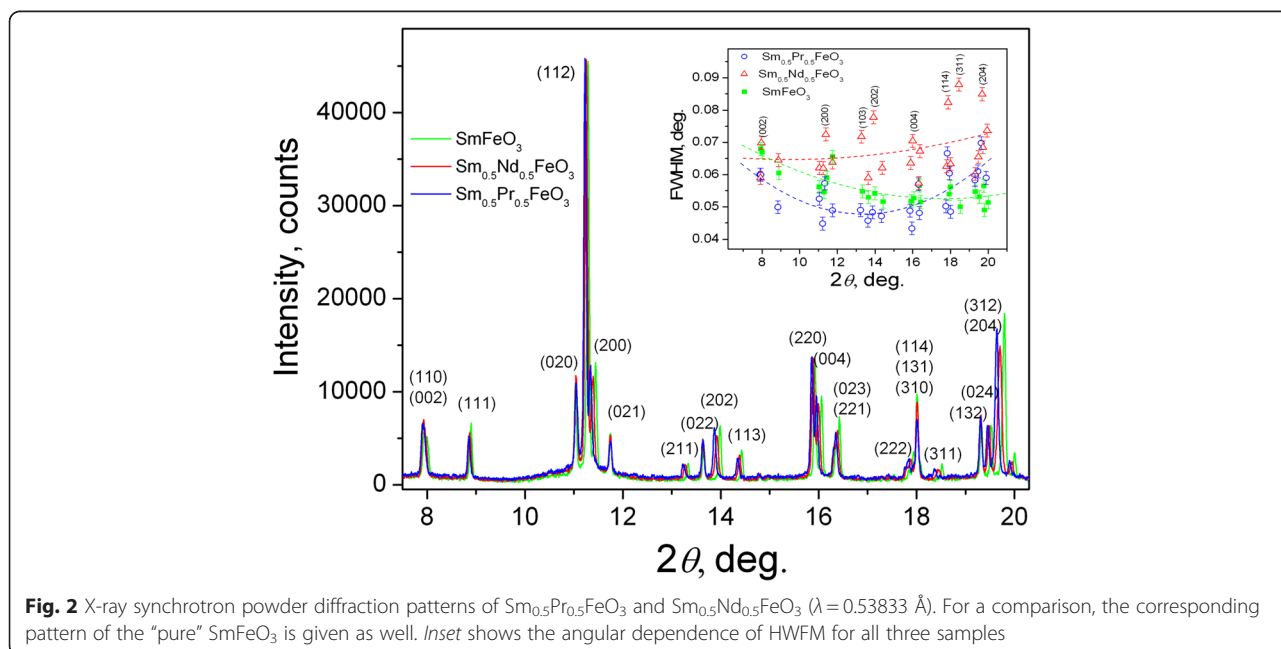
X-ray phase and structural characterization of the samples was performed at room temperature by using

imaging plate Guinier camera G670 ($\text{Cu K}\alpha_1$ radiation, $\lambda = 1.54056 \text{ \AA}$). Thermal behaviour of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ structures has been studied in situ in the temperature range of 298–1173 K by means of high-resolution X-ray synchrotron powder diffraction technique. The corresponding experimental powder diffraction patterns were collected with the temperature steps of 30 K at beamline B2 of synchrotron laboratory HASYLAB/DESY (Hamburg, Germany). Structural parameters of the samples were derived from the experimental diffractograms by using full-profile Rietveld refinement technique applying WinCSD program package [20].

Results and Discussion

X-ray powder diffraction examination revealed that both samples synthesized possess orthorhombic perovskite structure isotypic with GdFeO_3 . No extra crystalline phases were found. The unit-cell dimensions of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ at room temperature are in good agreement with the structural data of the parent SmFeO_3 , PrFeO_3 and NdFeO_3 [14, 15] compounds, (Fig. 1), thus proving possible formation of continuous solid solutions $\text{Sm}_{1-x}\text{Pr}_x\text{FeO}_3$ and $\text{Sm}_{1-x}\text{Nd}_x\text{FeO}_3$ in the $\text{SmFeO}_3\text{-PrFeO}_3$ and $\text{SmFeO}_3\text{-NdFeO}_3$ systems.

Precise high-resolution X-ray synchrotron powder diffraction examination confirms phase purity of the $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ samples (Fig. 2). The values of full width at half maximum (FWHM) of the mixed samarium-praseodymium and samarium-neodymium ferrites are in the limits of $0.043^\circ\text{-}0.089^\circ$, which is comparable with those of the “pure” SmFeO_3 ferrite (Fig. 2, inset). Angular dependence of FWHM of



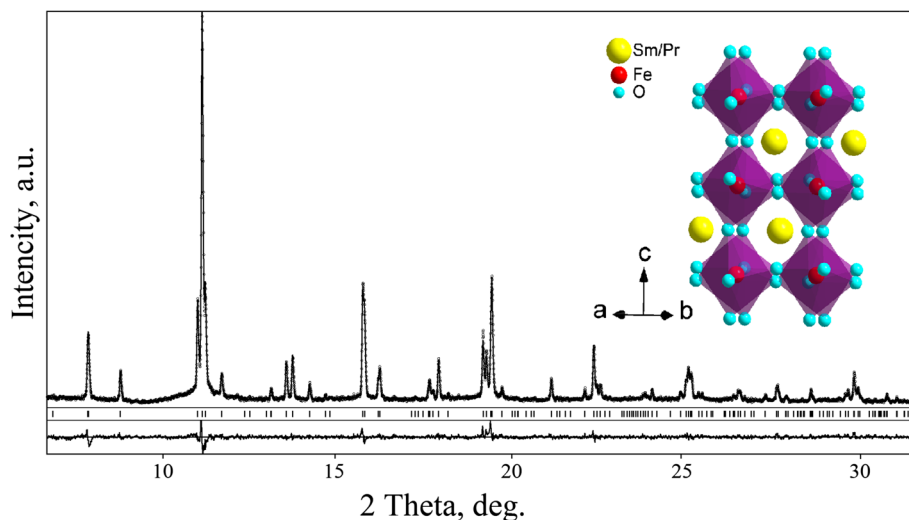
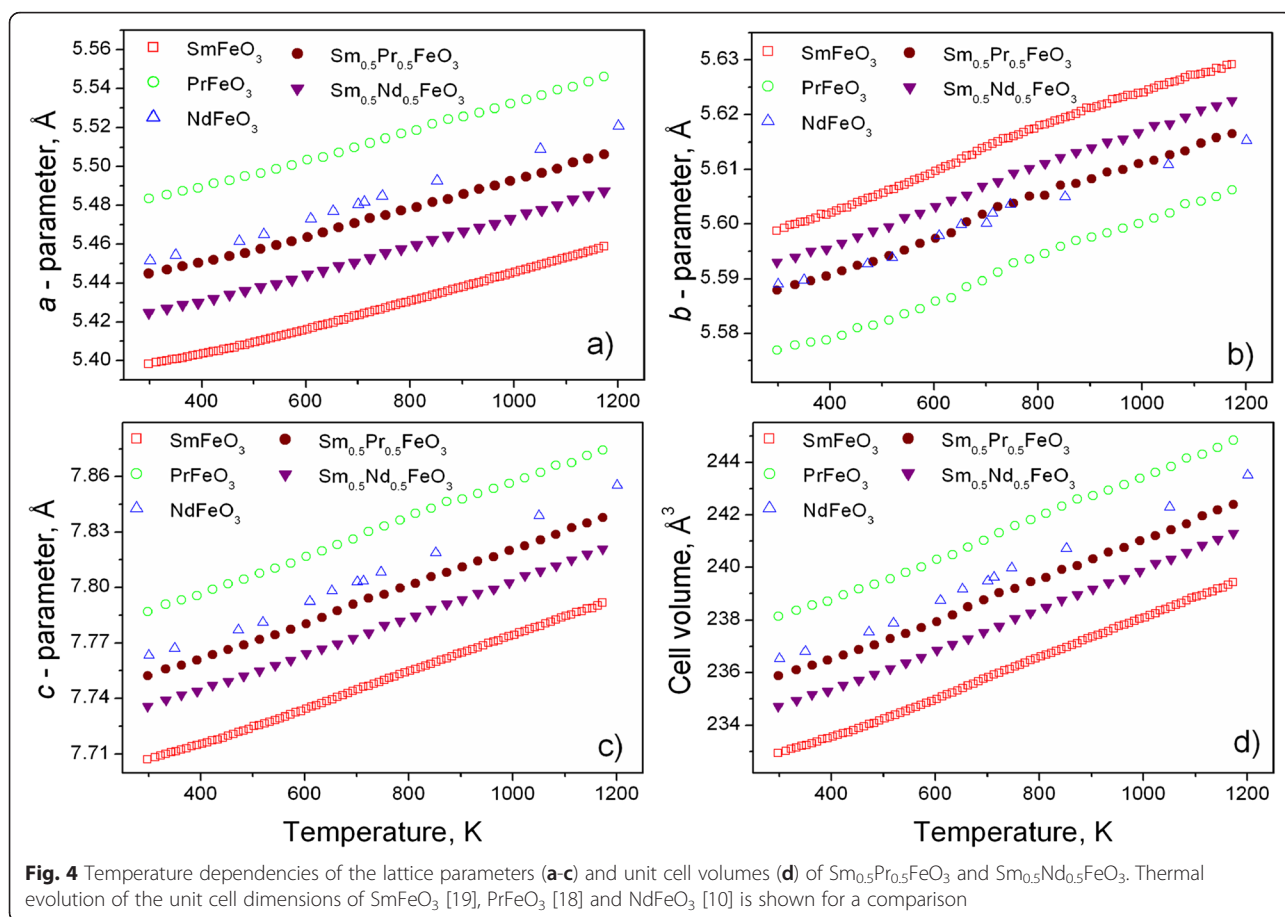


Fig. 3 Graphical results of Rietveld refinement of the $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ structure at 1173 K. Experimental X-ray synchrotron powder diffraction pattern ($\lambda = 0.53833 \text{ \AA}$) collected at 1173 K (dots) is shown in comparison with the calculated pattern (lines). The difference between measured and calculated profiles is shown as a curve below the diagrams. Short vertical bars indicate the positions of diffraction maxima in the space group $Pbnm$. Inset shows the view of the structure as corner-shared FeO_6 octahedra with Sm/Pr species located between them

Table 1 Lattice parameters, coordinates and displacement parameters of atoms in the $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ structures at RT, 753 and 1173 K

Atoms, sites	Parameters, residuals	$\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$			$\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$		
		$T = 298 \text{ K}$	$T = 753 \text{ K}$	$T = 1173 \text{ K}$	$T = 298 \text{ K}$	$T = 753 \text{ K}$	$T = 1173 \text{ K}$
$a, \text{ \AA}$		5.4449(1)	5.4749(1)	5.5064(2)	5.4248(1)	5.4554(2)	5.4872(2)
$c, \text{ \AA}$		5.5879(1)	5.6038(1)	5.6165(2)	5.5930(1)	5.6093(2)	5.6228(2)
$b, \text{ \AA}$		7.7523(2)	7.7962(2)	7.8378(2)	7.7357(2)	7.7795(2)	7.8210(3)
$V, \text{ \AA}^3$		235.87(2)	239.19(2)	242.39(2)	234.70(2)	238.06(2)	241.31(3)
Sm/Pr(Nd), 4c	x	-0.0099(3)	-0.0086(4)	-0.0081(4)	-0.0095(3)	-0.0089(4)	-0.0072(5)
	y	0.0495(2)	0.0466(2)	0.0433(2)	0.0517(2)	0.0497(2)	0.0451(2)
	z	1/4	1/4	1/4	1/4	1/4	1/4
	$B_{\text{isor}}, \text{ \AA}^2$	0.98(1)	1.12(2)	1.59(2)	0.942(9)	1.13(2)	1.58(2)
Fe, 4b	x	0	0	0	0	0	0
	y	1/2	1/2	1/2	1/2	1/2	1/2
	z	0	0	0	0	0	0
	$B_{\text{isor}}, \text{ \AA}^2$	0.76(3)	0.75(4)	1.08(5)	0.83(3)	0.70(4)	1.16(5)
O1, 4c	x	0.092(2)	0.091(2)	0.094(2)	0.090(2)	0.084(2)	0.085(2)
	y	0.481(2)	0.484(2)	0.481(2)	0.4821(14)	0.487(2)	0.482(2)
	z	1/4	1/4	1/4	1/4	1/4	1/4
	$B_{\text{isor}}, \text{ \AA}^2$	0.7(2)	0.9(3)	1.5(3)	0.5(2)	0.6(3)	1.7(3)
O2, 8d	x	-0.2934(13)	-0.2955(15)	-0.296(2)	-0.2961(13)	-0.299(2)	-0.289(2)
	y	0.2939(13)	0.2960(14)	0.301(2)	0.2965(12)	0.297(2)	0.294(2)
	z	0.0433(10)	0.0403(11)	0.0362(13)	0.0465(9)	0.0454(11)	0.0462(13)
	$B_{\text{isor}}, \text{ \AA}^2$	0.39(12)	0.4(2)	0.6(2)	0.44(12)	0.9(2)	0.6(2)
	R_1	0.104	0.102	0.109	0.102	0.104	0.104
R_p	0.168	0.170	0.185	0.166	0.187	0.196	



$\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ substantially resembles the behaviour of the parent SmFeO_3 compound, whereas a rather scattered behaviour is observed for the $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ sample (Fig. 2, inset). To some extent, *hkl*-dependent anisotropic broadening of Bragg peaks points on the possible compositional, thermal and elastic microstrains presented in the $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ sample [21].

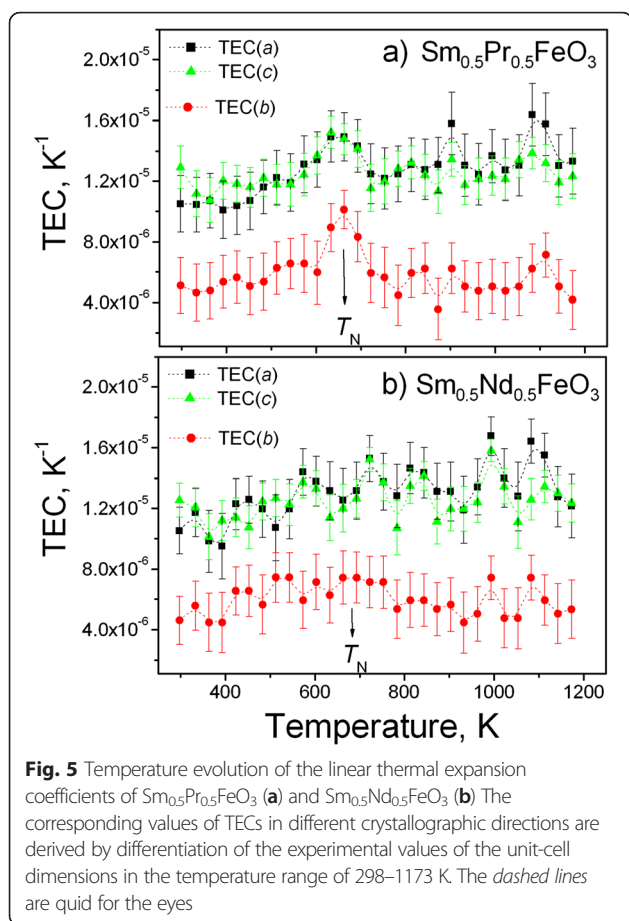
In situ high-temperature X-ray synchrotron powder diffraction investigations prove that $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ remain orthorhombic at least up to 1173 K. No structural phase transitions were detected in the whole temperature range investigated. Based on the experimental X-ray synchrotron powder diffraction data, the unit-cell dimensions and positional and displacement parameters of atoms in the $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ structures between RT and 1173 K were derived by full-profile Rietveld refinement technique. As an example, Fig. 3 represents the graphical results of Rietveld refinement of the $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ structure at 1173 K. Refined structural parameters of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ at the selected temperatures are presented in Table 1.

Temperature dependencies of the unit-cell dimensions of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ in comparison

with the literature data for the “pure” ferrite perovskites SmFeO_3 [19], PrFeO_3 [18] and NdFeO_3 [10] are presented in Fig. 4.

Temperature evolution of the lattice parameters of mixed Sm-Pr and Sm-Nd ferrites resemble for the most part the thermal behaviour of the parent compounds. In both cases, clear deviations from the “normal” trend are observed in the *b* direction at 650–750 K, whereas much less visible anomalies in the lattice expansion are observed in the *a* and *c* directions (Fig. 4a–c). It is evident that similar to SmFeO_3 and PrFeO_3 , a kink in the *b*-lattice expansion of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ is associated with the para- to antiferromagnetic transitions that occurred in these specimens at the Néel temperatures. Earlier, nonlinear lattice expansion across the antiferromagnetic to paramagnetic transitions was also observed in LaFeO_3 at $T_N = 735$ K [17].

Similar to the “pure” *RFeO*₃ perovskites, thermal expansion of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ shows a clear anisotropic behaviour. Calculated thermal expansion coefficients (TECs) in the *b* direction are in the limits of $(5.3\text{--}6.2) \times 10^{-6} \text{ K}^{-1}$ which is twice lower than the values of $(11.1\text{--}13.6) \times 10^{-6} \text{ K}^{-1}$ in the *a* and *c* directions (Fig. 5). Such anisotropic thermal expansion



is rather typical for the majority of perovskite oxides with a GdFeO_3 type of structure and is inherent for the families of rare earth aluminates, gallates [22–25] and other perovskites. The average linear thermal expansion coefficients of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ in the temperature range of 298–1173 K are in the limits of $(9.0\text{--}11.1) \times 10^{-6} \text{ K}^{-1}$. It is close to the TEC value of $(10.8\text{--}11.8) \times 10^{-6} \text{ K}^{-1}$ reported for LaFeO_3 [26, 27] and other rare earth ferrites confirming the suggestion that the nature of rare earth ions does not influence the thermal expansion in $R\text{FeO}_3$ [15].

Subtle maxima at the TEC curves of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ around 670 K (Fig. 5a) reflect the observed lattice anomalies at the Néel temperature. In spite of no obvious maxima observed on the TEC curves of $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$, a change of the slope of the TEC(b) values occurs around 650–700 K (Fig. 5b). A similar step at the thermal expansion coefficient at $723 \pm 50 \text{ K}$, corresponding with the Néel temperature, has been revealed in LaFeO_3 by dilatometric measurements [16].

The lattice expansion of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ could be also affected by a possible change of the oxygen defect structure during the heating of the samples, as it was detected in PrFeO_3 and

SmFeO_3 by thermogravimetric measurements [15]. As it was shown, detectable weight loss due to the fast oxygen desorption begins in these ferrites above 573 K. As a consequence, thermal expansion behaviour of SmFeO_3 shows a change of the slope at around 593 K close to the temperature of sharp weight loss detected by TGA [15].

Conclusions

Crystal structure parameters of the mixed samarium-praseodymium and samarium-neodymium ferrites $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ synthesized by solid-state reaction technique in air at 1473 K have been studied in a wide temperature range of 298–1173 K by means of high-resolution X-ray synchrotron powder diffraction technique. Close analysis of the temperature dependence of the unit-cell dimensions in comparison with the literature data for the parent $R\text{FeO}_3$ compounds revealed strongly anisotropic lattice expansion and subtle anomalies associated with the para- to antiferromagnetic transitions at 650–750 K. The average linear thermal expansion coefficients of $\text{Sm}_{0.5}\text{Pr}_{0.5}\text{FeO}_3$ and $\text{Sm}_{0.5}\text{Nd}_{0.5}\text{FeO}_3$ derived from the experimental values of the unit-cell dimensions in the temperature range of 298–1173 K are in the limits of $(9.0\text{--}11.1) \times 10^{-6} \text{ K}^{-1}$, which is close to the corresponding values reported for the parent $R\text{FeO}_3$ compounds.

Competing Interests

The authors declare that they have no competing interests.

Authors' Contributions

OP synthesized the samples, contributed to the data evaluation and wrote the manuscript. LV performed the laboratory X-ray and HT synchrotron powder diffraction measurements, made the structural characterization of the samples and contributed to the manuscript writing. OB contributed to the discussion of the results and manuscript writing. All authors read and approved the final manuscript.

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