Crystallographic, electrical, and magnetic properties of the system $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$

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On the basis of x-ray structural data and studies of the electrical, magnetic, and magnetoresistive properties and the ferromagnet resonance spectra of ceramic samples of $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ ($0 \leqslant x \leqslant 0.10$) it is established that a competition between structural changes influencing the ferromagnetism leads to a monotonic weakening of the ferromagnetism (a lowering of T_C by around 5 K/%Fe). The increase of the magnetoresistance in the low-temperature region and the large values of the low-field contribution to the magnetoresistance are indicative of an inhomogeneous character of the electrical conductivity and magnetization of the samples at $T < T_C$, which may be due to the coexistence of the ferro- and paramagnetic phases over a wide temperature region. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2171513]

The observation of a strong influence of the magnetic field on the electrical resistance of the mixed manganites $La_{1-x}A_xMnO_3$ (A=Ca, Sr, Ba...) has stimulated interest in further research on such systems.[1–3] The significant lowering of the resistivity ρ under the influence of magnetic field H, which has been given the name colossal magnetoresistance, may find practical application in magnetic sensors, read heads for ultrahigh-density magnetic storage media, etc.

The mixed manganites $La_{1-x}A_xMnO_3$, which belong to the perovskite structural type ABO_3 with mixed valence of the manganese ions (Mn^{3+}/Mn^{4+}), are characterized by an intimate connection between the magnetic and electrical properties, the crystallographic parameters, and the chemical composition.[1,2] The end compounds $La^{3+}Mn^{3+}O_3$ and $A^{2+}Mn^{4+}O_3$ are antiferromagnetic insulators. However, in the $La_{1-x}A_xMnO_3$ system in the concentration range $0.2 \leqslant x \leqslant 0.5$, with decreasing temperature a transition occurs from a paramagnetic state with an activated character of the conductivity to a ferromagnetic metallic state, resulting in the appearance of a maximum on the temperature dependence of the resistivity $\rho(T)$.[1–3] The temperature of the resistance maximum is close to the temperature T_C of the transition from the ferro- to the paramagnetic state (the Curie point). As a rule, the curve of the magnetoresistance versus temperature, MR(T), also has such a maximum near T_C. In the paramagnetic region the magnetoresistance goes to zero.

It is clear that practical application of the MR effect requires materials with both a high sensitivity to magnetic field and a Curie temperature close to 300 K. The key parameters influencing the temperature of the MR maximum are: the mean radius $\langle R_A \rangle$ of the ions of the A sublattice of the ABO_3 perovskite structure, the deviations in the size of the cations occupying the A sublattice, and the valence state of the manganese, which is determined by the ratio of the numbers of triply and quadruply ionized manganese ions, Mn^{3+}/Mn^{4+} (Refs. 3–6). The theoretical explanation of the colossal MR effect is based on the concept of double exchange—the continual transfer of an electron from Mn^{3+} to Mn^{4+} manganese cations via in equivalent positions via the $2p$ orbitals of the O^{2-} anion.[7] An important role in the MR mechanism is played by the strong electron-phonon interaction, which is caused by the Jahn-Teller local distortion of the crystal lattice by the Mn^{3+} ions.[8,9]

Besides the Jahn–Teller ions Mn^{3+}, local distortions of the crystal lattice in $La_{1-x}A_xMnO_3$ compounds can be caused by substitution of the manganese by other $3d$ metal ions. Based on the close values of the ionic radii R_{Mn3+} (0.65 Å) and R_{Mn4+} (0.645 Å),[10] the existence of $La_{1-x}A_xMn_{1-y}Fe_yO_3$ solid solutions is possible. Here the double exchange can be influenced not only by a change of the number of exchange positions in the manganese sublattice but also by the formation of discontinuities in the $Mn^{3+}-Mn^{4+}$ pairs when Fe^{3+} is built into the $Mn^{3+}-O^{2-}-Mn^{4+}$ chains.

At present there are data[11–16] on the influence of iron on the properties of lanthanum-calcium manganites $La_{1-x}Ca_xMnO_3$. For $La_{0.75}Ca_{0.25}Mn_{1-y}Fe_yO_3$ the authors of Refs. 11 and 12 observed the existence of size-distributed clusters, which become delocalized at a certain critical value of y. In the solid solution $La_{0.67}Ca_{0.33}Mn_{0.90}Fe_{0.10}O_3$ a competition between ferromagnetic and antiferromagnetic character of the interactions between clusters was revealed, and a transition to a spin-glass state was observed.[14] For the same composition $La_{0.7}Ca_{0.3}Mn_{0.95}Fe_{0.05}O_3$ the reported values of the lowering of T_C in comparison with the undoped sample range from 10 K/%Fe in Ref. 15 to 13 K/%Fe in Ref. 16; the value of the MR itself changes insignificantly upon iron doping of $La_{1-x}Ca_xMnO_3$.

Undoubtedly there is significant scientific and practical interest in the study of the influence of iron doping on the properties of lanthanum-strontium manganites $La_{1-x}Sr_xMnO_3$ having $T_C \geqslant 300$ K. The majority of papers reporting the results of studies of the substitution of manganese ions by iron confirm that the Fe^{3+} ions do not take part in the double exchange, but the authors of those papers mention a significant change of the magnetic, electrical, and magnetoresistive properties as Fe substitutes for Mn.[17–24] For example, it was observed in Ref. 17 that the system $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ becomes insulating in the entire temperature range for $x > 0.2$. The value of the lowering of T_C upon iron doping of lanthanum-strontium manganites fluctuates from 7 K/%Fe in Ref. 18 to 15 K/%Fe in Ref. 21. In the majority of cases, however, those studies did not include an analysis of the influence of the change of the crystallographic parameters on the magnetic and electrical properties, and the data on the value of the MR are spotty.

The goal of the present study is to conduct a comprehensive investigation of the structural, magnetic, and electrical properties of $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ solid solutions in the range $0 \leqslant x \leqslant 0.10$.

EXPERIMENTAL PART

The samples were prepared by solid-phase synthesis from dried La_2O_3, Mn_2O_3, Fe_2O_3 (extra-pure grade), and $SrCO_3$ (reagent grade) mixed and homogenized with distilled water on a vibrating mill for 6 hours. After the first heat treatment at 1190 K for 4 hours a repeated homogenizing grinding was done. The second heat treatment was at a temperature of 1120 K for 4 h. A binder was introduced into the annealed homogenized $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ powder, and tablets 10 mm in diameter and 3–4 mm thick were pressed and then sintered for 2 hours at temperatures of 1620–1670 K.

The structural parameters were determined by the method of Rietveld full-profile analysis. X-ray studies were done on a DRON-4-07 diffractometer in CuK_α radiation [external standards: SiO_2 (2θ standard) and NIST SRM1979—Al_2O_3 (certified intensity standard)]. The diffractograms were taken in the angle interval $2\theta = 10-150°$ in the discrete mode with a step of $\Delta 2\theta = 0.02°$ and a 10-second exposure at each point.

The resistivity of the sintered ceramics was measured by the four-probe method in the temperature interval 77–370 K. The samples for study were cut out in the form of rectangular slabs with dimensions of $2 \times 3 \times 10$ mm. Contacts were deposited by the brazing-in of a silver-containing paste. The magnetoresistance MR was measured in magnetic fields up to 1.2 MA/m and calculated using the relation $MR = [(\rho_H - \rho_0)/\rho_0] \cdot 100\%$, where ρ_0 is the resistivity at zero field $H = 0$ and ρ_H is the resistivity at field H.

The ferromagnetic resonance (FMR) spectra were recorded on a RADIOPAN spectrometer on samples in the form of parallelepipeds with dimensions of $1 \times 1 \times 5$ mm in a magnetic field directed parallel to the long axis of the sample.

RESULTS AND DISCUSSION

We studied $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ samples with $x = 0$, 0.02, 0.04, 0.06, 0.08, and 0.10. Figure 1 shows the x-ray diffractograms of the $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ system. All of the samples synthesized were single-phase and could be indexed in the rhombohedral structure with space group $R\bar{3}c$.

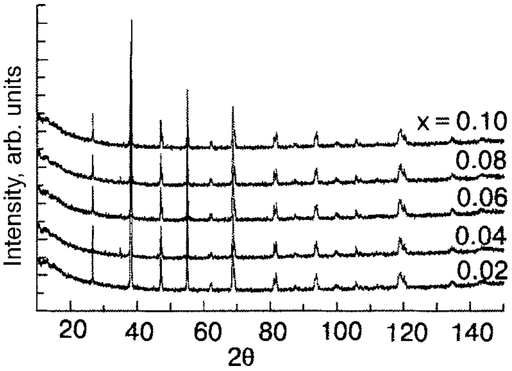

FIG. 1. X-ray diffractograms of polycrystalline samples of the system $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$; synthesis temperature 1650 K, 2 hours.

The results of calculations of the crystallographic parameters of $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ by the Rietveld method in the hexagonal system $R\bar{3}c$ are presented in Table I, from which it follows that the unit cell volume and length of the Mn–O bond in $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ solid solutions decrease with increasing iron content. This fact is explained by the smaller ionic radius of Fe^{3+} (0.645 Å) as compared to that of Mn^{3+} (0.65 Å) and serves as one piece of evidence confirming that charge compensation $Mn^{3+} \rightarrow Fe^{3+}$ is realized; this agrees with the data presented in Ref. 23. According to Mössbauer studies, it is the high stability of the $t_{2g}^3 e_g^2$ configuration of iron that impedes the participation of Fe^{3+} in the formation of the e_g bond in manganites.[24]

In analyzing the character of the change of the crystallographic parameters with increasing iron concentration one should note the somewhat unexpected result of a calculation of the isotropic microstresses characterizing the degree of stability of the structure:[25] as the manganese positions are substituted by ions of another element—iron—the stability of the structure increases. The immediate cause of this may be the increase of the Mn–O–Mn bond angle, which is equal to 180° in the most stable state.

The temperature of the ferromagnetic transition, according to Refs. 26 and 27, should increase as the Mn–O bond length decreases and the Mn–O–Mn angle increases. Therefore, according to the data presented in Table I, the character of the change of the crystallographic parameters upon the substitution $Mn^{3+} \rightarrow Fe^{3+}$ should lead to enhancement of the ferromagnetic interaction. On the other hand, since the introduction of Fe^{3+} ions decreases the number of exchange positions for electron transport and causes breaks in the $Mn^{3+}-O^{2-}-Mn^{4+}$ chains, the substitution of manganese by iron will weaken the ferromagnetism in the $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ system, and that should have a substantial effect on the magnetic and electrical properties.

The temperature dependence of the resistivity $\rho(T)$ of polycrystalline samples of $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ that had been sintered at 1650 K is presented in Fig. 2. For all the doping levels studied, the transition from a metallic character of the conduction ($d\rho/dT > 0$, ferromagnetic phase) to an activated character ($d\rho/dT < 0$, paramagnetic phase) is indistinct and occurs over a broad temperature interval. In such a case the position of the maximum on the $\rho(T)$ curve need not correspond to the Curie temperature,[28,29] and in order to ob-

TABLE I. Crystallographic parameters of the system $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ (synthesis temperature 1650 K; 2 hours).

x	0	0.02	0.04	0.06	0.08	0.10
			$R\bar{3}c$ cell parameters			
a, Å	5.5082(4)	5.5074(5)	5.5066(5)	5.5060(4)	5.5053(4)	5.5048(4)
c, Å	13.3718(6)	13.3671(7)	13.3651(7)	13.3644(6)	13.3641(7)	13.3640(7)
V, Å^3	351.35(4)	351.12(5)	350.97(5)	350.88(4)	350.78(4)	350.71(4)
			coordinates of O_1 ions			
x/a	0.456(3)	0.457(3)	0.458(2)	0.459(3)	0.460(3)	0.462(5)
			Isotropic microstrains, %			
	15.87	15.13	14.32	8.43	8.02	6.82
			Interatomic distances and bond angles			
Mn–O, Å	1.957(2)	1.956(2)	1.955(2)	1.954(2)	1.953(2)	1.952(3)
Mn–O–Mn, deg	165.8(6)	166.1(6)	166.4(4)	166.7(6)	167.1(6)	167.7(9)
			Reliability factors			
R_B, %	5.59	4.86	6.29	6.51	6.95	6.70
R_f, %	5.43	5.14	6.17	6.12	7.69	6.53

Note: The position and coordinates of the ions in the $R\bar{3}c$ structure are: La 6*a* (0 0 1/4); Mn 6*b* (0 0 0); O 18*e* (x 0 1/4).

tain information about the magnetic properties it is necessary to make additional measurements—magnetic or magnetoresistive.

The character of the MR(H) curves at room temperature (Fig. 3a) and also the nonzero values of the MR attest to the fact that at 300 K all of the samples are found in the ferromagnetic state. Thus for all the samples with $x \leqslant 10$ the Curie temperature T_C is above 300 K, and the positions of the individual broad temperature maxima of the resistivity with $T < 300$ K in Fig. 2 do not reflect the actual temperatures of the ferromagnetic transitions.

The results of a study of the influence of magnetic field on the MR of samples of the system $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ at liquid nitrogen temperature are presented in Fig. 3b. The absolute values of the MR at 77 K are substantially higher than at 300 K, ranging from 23% ($x=0.02$) to 20% ($x=0.10$) at a magnetic field strength of 1.2 MA/m. The sharp change in the trend of the $MR_{77}(H)$ curves near $H=0.16$–0.17 MA/m may indicate the existence of at least two mechanisms by which the magnetic field influences the resistivity of substituted manganites, one of which is predominant at low fields ($H \leqslant 0.17$ MA/m) and the other at high fields ($H \geqslant 0.17$ MA/m). The first of these is due to the spin-dependent transport of charge through the grain boundaries or poorly conducting regions, and the second is the result of the effect of magnetic field on the electronic subsystem of the core of the grains.[1–3,30]

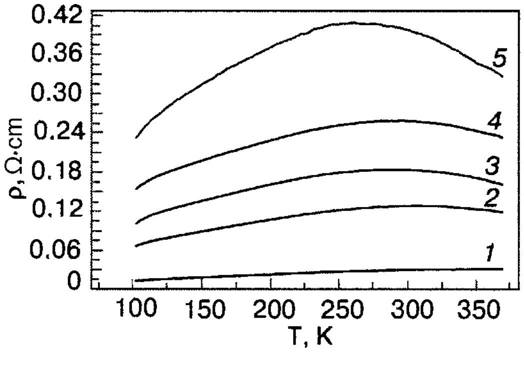

FIG. 2. Temperature dependence of the resistivity of polycrystalline samples of $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$: $x=0.02$ (curve *1*), 0.04 (*2*), 0.06 (*3*), 0.08 (*4*), 0.10 (*5*).

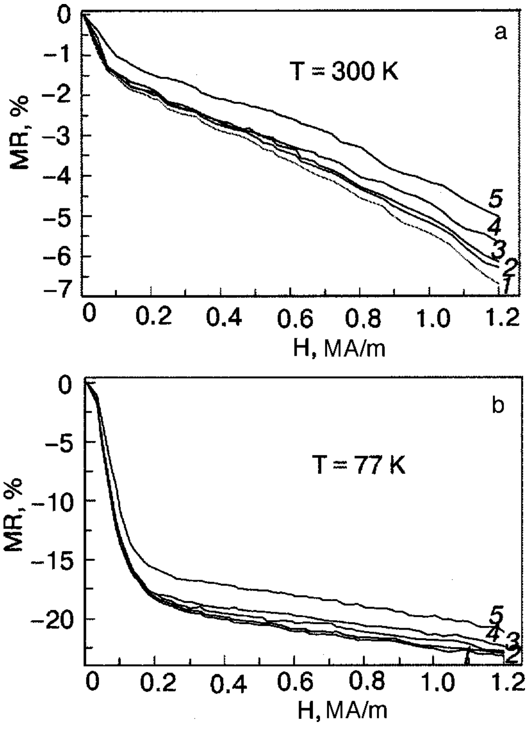

FIG. 3. Influence of magnetic field on the magnetoresistance at room temperature (a) and at liquid nitrogen temperature (b) for polycrystalline samples of $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$: $x=0.02$ (curve *1*), 0.04 (*2*), 0.06 (*3*), 0.08 (*4*), 0.10 (*5*).

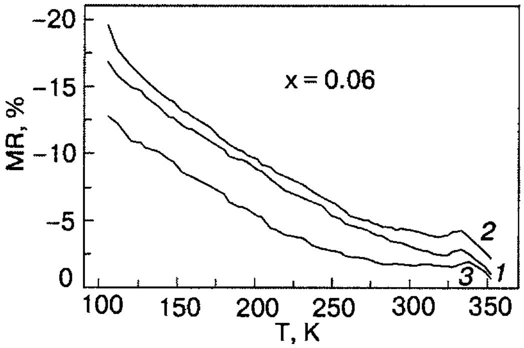

FIG. 4. Temperature dependence of the magnetoresistance at a field of 1.2 MA/m for a polycrystalline sample of $La_{0.7}Sr_{0.3}Mn_{0.94}Fe_{0.06}O_3$, sintered at temperatures T [K]: 1620 (*1*), 1650 (*2*), 1670 (*3*).

The temperature dependence of the MR at a field $H = 1.2$ MA/m for polycrystalline samples of $La_{0.7}Sr_{0.3}Mn_{0.94}Fe_{0.06}O_3$ that had been sintered at different temperatures is presented in Fig. 4. The weakly expressed peaks of the MR at 332–336 K are apparently due to a ferromagnet-paramagnet transition.[3] In the present study we determined T_C as the temperature of the peaks on the MR(T) curves. For the samples investigated in this study the lowering of T_C upon iron doping in the $La_{0.7}Sr_{0.3}MnO_3$ system is around 5 K/%Fe.

The shifts of the position of the maximum of the MR for the same composition, as can be seen in Fig. 4, indicate that the synthesis temperature has a decisive influence on the Curie point of substituted manganites. The highest values of the MR are seen in the ceramics sintered at temperatures close to 1650 K. The influence of the synthesis temperature on the MR may be due to a change of the oxygen content in the structure and, accordingly, to a change of the Mn^{4+}/Mn^{3+} ratio.

Noticeable growth of the negative magnetoresistance below 300 K on the MR(T) curves is usually attributed to spin-dependent charge transport through magnetically disordered regions.[1–3,31] Coexistence of the para- and ferromagnetic phases is observed quite often in substituted manganites[31,32] and is due to the strong dependence of the magnetic properties on chemical inhomogeneities, local deformations caused by the introduction of foreign ions into the manganese sublattice, features of the microstructure, etc.

Figure 5 shows the FMR spectra measured at 77 K for $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ samples. Such spectra are characteristic for the ferromagnetic phase of substituted manganites.[28,29,32] As is seen in Fig. 5, as the iron content is increased to 0.10 the shape of the spectra does not undergo significant changes. At the same time, there is a monotonic increase of the difference between the position of the maxima and minima (ΔB) of the FMR spectra from 170–175 mT ($x=0$) to 245–250 mT ($x=0.10$). This may indicate an increase in the inhomogeneity and the resultant lowering of the ferromagnetic phase fraction in lanthanum-strontium manganites as the concentration of Fe^{3+} ions increases.

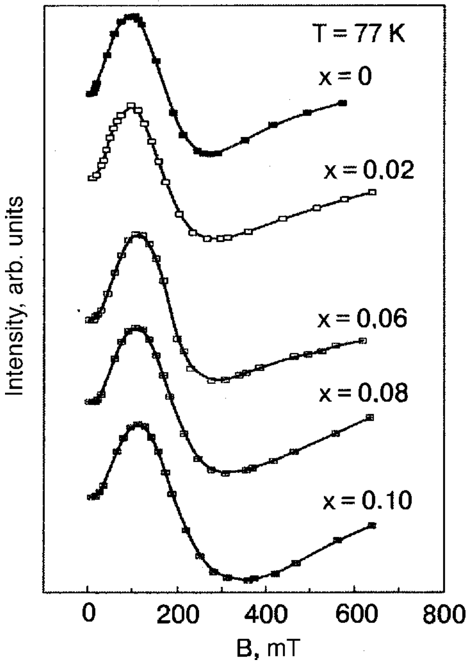

FIG. 5. Ferromagnetic resonance spectra measured at liquid nitrogen temperature for polycrystalline samples of $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$.

CONCLUSIONS

The realization of the charge compensation mechanism $Mn^{3+} \rightarrow Fe^{3+}$ in the system $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ ($0 \leqslant x \leqslant 0.10$) leads to a combination of two competing tendencies, one of which (the decrease of the unit cell volume, the shortening of the Mn–O bond length, and the increase of the Mn–O–Mn bond angle) promotes ferromagnetism, while the other (breaks in the Mn–O–Mn chains, decreasing of the number of exchange positions) weakens the ferromagnetism.

According to our data, for all samples of the system $La_{0.7}Sr_{0.3}Mn_{1-x}Fe_xO_3$ with $0 \leqslant x \leqslant 0.10$ the values of the Curie temperature T_C are above 300 K, although with increasing x a monotonic weakening of the ferromagnetism occurs (a lowering of T_C by around 5 K/%Fe). The synthesis temperature of the solid solutions of manganites also has a definite influence on T_C. The monotonic growth of the magnetoresistance in the low-temperature region and the large values of the low-field contribution to the magnetoresistance are indicative of an inhomogeneous character of the electrical conductivity and magnetization of the samples at $T < T_C$, possibly due to coexistence of the ferro- and paramagnetic phases over a wide temperature region.

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