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Oxygen diffusion in different thicknesses $La_{0.8}Sr_{0.2}MnO_{3-\delta}$ films on $NdGaO_3$ substrates

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The results of investigation by the indirect method of step-by-step varying the oxygen content in the series of epitaxial $La_{0.8}Sr_{0.2}MnO_{3-\delta}$ films on single-crystal $NdGaO_3$ substrates are presented. Using numerical simulation we have revealed that the oxygen diffusion coefficient significantly decreases along the film thickness in the direction from the outer surface to the film-substrate interface under conditions of „compressive" mechanical stresses caused by the mismatch of the in-plane crystalline parameters of the film and substrate materials. In films of $d \approx 12-75$ nm thickness, the effect is manifested in the fact that the value of the diffusion coefficient in the vicinity of the outer surface of the films also decreases significantly as the thickness of the films decreases. The questions of the applicability of the indirect method for evaluating the oxygen content in thin epitaxial films, as well as other manifestations of effects caused by mechanical stresses are discussed.

Keywords: doped manganites, oxygen index, heat treatment, mechanical stress, the oxygen diffusion coefficient.

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Introduction

The technology of obtaining high-quality films on the basis of doped manganites with oxygen content (OC) close to stoichiometric composition usually includes thermal treatment of film structures in oxygen-containing gas medium. It is quite difficult [1–3] to provide a stoichiometric composition of oxygen directly during film growth (*in situ*). The heat treatment procedure of the finished film allows to change the OC of the film material and thus to adjust the electrical and magnetic characteristics of the object, for example, to provide the maximum value of the temperature coefficient of resistance in a given temperature range. This is important for the operation of IR radiation sensors, devices for monitoring the value of thermal kinetic coefficients in film structures by a non-stationary method [4–6]. The fundamental interest in OC variation in films is to obtain the most perfect objects for studying the properties of strongly correlated systems.

The main problem in the research of oxygen variation processes in doped manganite films is the lack of direct non-destructive methods for controlling OC. Since a thin oxide film is in contact with a relatively thick oxide substrate, estimation of small OC deviations in the film against the background of large oxygen content in the microscopic layer of the substrate using energy dispersive spectrometers is almost impossible due to low accuracy. Alternative possibilities for quantitative assessment of the OC are associated with the use of indirect methods.

It should be noted at once, that the physical properties of thin films and monocrystalline samples can differ significantly for a number of reasons. The crystalline parameters of the epitaxial films and the substrates with closest structure are characterized by some mismatch. As a result, thin epitaxial films are mechanically stressed. The influence of mechanical stresses on the galvanomagnetic characteristics of the films, including the effect of colossal magnetic resistance, has been widely studied in films based on various doped manganites, with different levels of doping on several types of single-crystal substrates [7–18].

The authors characterize the mechanical stresses of epitaxial films based on X-ray diffraction (XRD) analysis of the film structures. The method is good at recording changes in crystal lattice parameters in films of different thicknesses, less available are data on the distribution of these changes over the thickness of the relatively thick film. The issue of quantitative assessment of OC in the film material on the basis of estimates of crystal parameters is not considered. The authors obtain useful information about the OC of the film material in the form of the ratio of $Mn^{+3}-Mn^{+4}$ ion concentrations from X-ray photoelectron spectroscopy (XPS) data. The limitations of this method are usually due to the fact that the information obtained relates only to a thin near-surface layer of the film.

Let us note another of the previously discussed approaches for controlling the oxygen index, which is based on theoretical calculations of the equilibrium OC in the film material at several actual temperatures depending on the partial pressure of oxygen in the gas medium. It was used to investigate the effect of excess oxygen on the characteristics of epitaxial $La_{1-x}Sr_xMnO_{3-\delta}$ (LSMO) films [19]. Clearly, this straightforward approach is useful in planning experiments, but is limited because, for example, it does not take into account the effects caused by mechanical stresses on the film material. More productive were indirect methods based on the use of the galvanomagnetic characteristics of films [20,21].

The most promising method for epitaxial LSMO films is based on the relation between the oxygen index (δ) and the double phase transition temperature (T_C), in particular the metal-dielectric transition. The method was originally proposed for single-crystal [22] and later adapted for LSMO ($x = 0.3$) film with thickness ~ 100 nm [21]. The application of the method demonstrated a sufficiently high resolution of the OC changes of the film during the multistep heat treatment and the possibility to calibrate the corresponding diagram $T_C - \delta$, which allowed a detailed quantification of the δ values during the step-by-step variation of the OC in the film structure. Work [23] considers the peculiarities of the application of the indirect method to estimate the OC in films of different thicknesses. In the present work, we present the results of studies of oxygen diffusion processes in a series of films with thicknesses from ~ 12 to ~ 75 nm using this method. The main focus of the research is to reveal the effects of non-uniform oxygen diffusion in films of different thicknesses in the presence of „compressive" planar mechanical stresses of the epitaxial film material from the $NdGaO_3$-substrate. Previously, the so-called strongly non-uniform diffusion effect was found in LSMO films on $SrTiO_3$-substrate, causing „tensile " mechanical stress [21].

1. Experiment details

The films were produced by DC-magnetron sputtering of a ceramic target onto single-crystal $NdGaO_3$ (110) substrates heated to ~ 873 K in an atmosphere of a gas mixture —80% argon and 20% oxygen. Note that in our experiments we used high-quality single-crystal substrates from Mateck (Germany). The time of film growth ranged from 5 to 30 min. The film thickness was monitored using several methods, including the method developed by the authors of thickness estimation based on data from the INCA Energy-350 energy dispersive spectrometer included in the JSM-6490-LV [24] electron microscope.

The films obtained by the method described above are characterized by the deficiency of OC [25]. The sequential saturation of the film material with oxygen was carried out by a step-by-step thermal treatment of the film structures in air at several fixed temperatures in the range of $773-1173$ K. The mode of heating the film structure to the operating temperature and cooling to room temperature was carried out for half an hour, at a rate of approximately $20-30$ K/min. After each heat treatment step with durations from 0.5 to 2 h, electrical contacts were formed on the film using silver paste, and temperature dependences of resistance were measured at the temperature range $77-360$ K in a fixed magnitude magnetic field of $H = 0, 5, 10$ and 15 kOe. Next, the temperature of the metal-dielectric phase transition was fixed at $H = 0$ and the value of the oxygen index was determined according to the method described in [21]. As a result, we obtained a set of dependences of the oxygen index in films of different thicknesses on the heat treatment time at several fixed temperatures (Fig. 1). The temporal changes of the oxygen index in films from 75.4 to 12.6 nm thickness are shown in Fig. 1 with open circles.

The values of the oxygen index shown in Fig. 1 are derived from the experimental values of T_C according to the diagram $T_C - z$ from the work [21]. Parameter z is defined as $z = x - 2\delta$. The $T_C - z$ diagram was constructed for LSMO-films on $SrTiO_3$ substrate by making a correction lowering the maximum achievable value of T_C in monocrystalline samples by 4% (from 375 to 360 K). For our LSMO-films on $NdGaO_3$-substrates, due to the relatively low alloying level ($x = 0.2$), the maximum achievable value of T_C $(x, \delta \approx 0)$ has not been experimentally established. Therefore, the correction value to the maximum achievable value of T_C needed to calibrate the diagram is unknown in this case.

Theoretically, the effect of mechanical distortions of crystal parameters on the value of T_C of the film is usually described using the phenomenological expression [8,26–28]:

$$T_C(\varepsilon) = T_C(0)(1 - \alpha\varepsilon_b - \beta\varepsilon^{*2}/2). \tag{1}$$

Here, the parameter ε_b characterizes the change in the volume of the lattice cell, and ε^* — the biaxial in-plane, or so called Jan-Teller distortions of the lattice. $T_C(0)$ corresponds to its value in the absence of mechanical distortions. It follows from relation (1) that a change in the volume of the lattice cell can lead to an increase or decrease of T_C, and planar distortions always lead to a decrease of T_C regardless of the sign of distortion. The quantitative changes of T_C depend on the coefficients α and β, which have been calculated from experimental data by the authors of several papers [8,27], but the results of the estimates are not yet conclusive. Indeed, the authors of [8,29], in accordance with relation (1), estimated the values of the coefficients α and β using the fact of reduced values of T_C in films on $SrTiO_3$ and $NdGaO_3$ substrates by 3 and 1 K. But the point is that the authors used $T_C(0)$ as the value $T_C(0) = 334$ K (or 336 K) for the LSMO-film on LSAT substrate, for which mechanical distortion was considered negligible. Nevertheless, these values are much lower than in LSMO monocrystals T_C $(x = 0.34, \delta = 0) \approx 375$ K [30]. Thus, the authors of these works suggest the presence of an additional factor that significantly lowers the value of T_C in films in addition to mechanical stresses. This issue is related to the material quality of the films and substrates studied and, above all, the OC control.

Given the small temperature difference T_C in films on $SrTiO_3$ and $NdGaO_3$ substrates according to the authors [8,29], the $T_C - z$ diagram has not been specified in this paper.

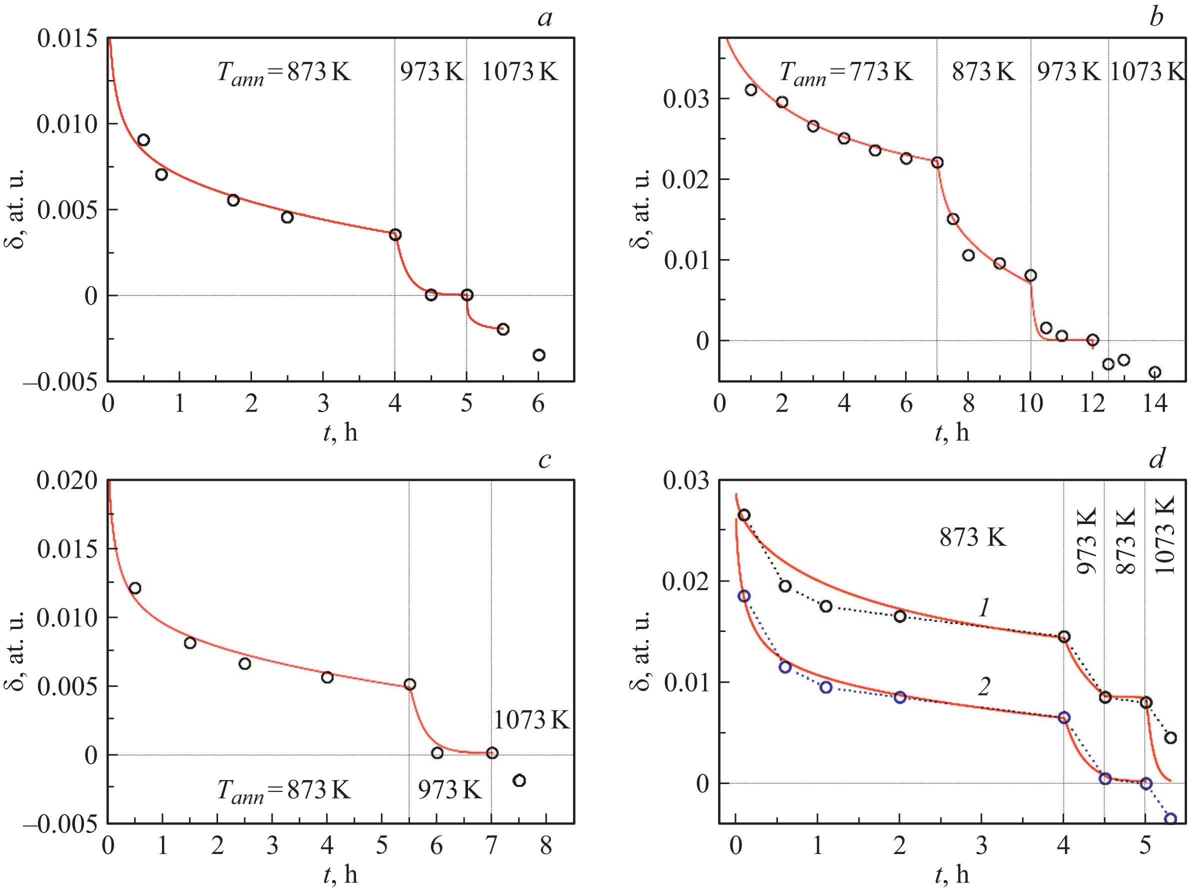

Figure 1. Time dependences of oxygen index variation during heat treatment at $T = 873{-}1073$ K in air $La_{0.8}Sr_{0.2}MnO_{3-\delta}$-thick films: *a* — 75.4, *b* — 45.2, *c* — 25.1, *d* — 12.6 nm (*1*).

2. Numerical simulation of the oxygen diffusion process

To describe the process of oxygen diffusion in the films, we used a physical model that takes into account the possible dependence of the diffusion coefficient on the thickness of the oxygen-deficient film. Note that in [21], it was found that the type of time dependences of the oxygen index during the heat treatment of the oxygen-deficient film indicates large (by two orders of magnitude) changes in the value of the atomic oxygen diffusion coefficient along the film thickness. The physical nature of this dependence was tentatively associated with mechanical stresses in the film material. Thus, the results of the present work are the additional verification of the physical interpretation of the observed effect.

In the framework of the mathematical model, the temperature changes of the diffusion coefficient are traditionally represented as an Arrhenius relation:

$$D = D_0 \exp[-(U_1 - U_2(y))/kT]. \qquad (2)$$

Here, $D_0 = \text{const}$, U_1 — activation energy, y — coordinate along the film thickness, counted from the external interface, $U_2(y)$ determines the possible spatial dependence of the diffusion coefficient along the film thickness in the form of:

$$U_2(y) = \Delta U\big(1 - \exp[-(y/y_0)^4]\big). \qquad (3)$$

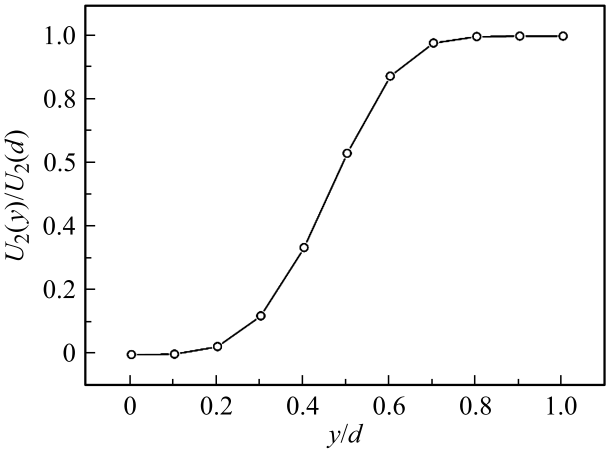

Figure 2. Spatial dependence of the parameter $U_2(y)$ on the film thickness at $y_0 = 0.5$.

Fig. 2 shows the coordinate dependence of $U_2(y)$. At the outer interface ($y = 0$), according to expression (2), the diffusion coefficient is given by the multiplier D_0 and the activation energy U_1, and the parameter ΔU determines the decreasing amplitude D along the film thickness. In the calculation program, the value of D is set at $T = 1173$ K, and for other temperatures it is automatically recalculated according to relation (3). Diffusion equation for OC in the form of

$$dn_{\mathrm{O}}/dt = d/dy(D \cdot dn_{\mathrm{O}}/dy) \qquad (4)$$

was numerically solved with boundary conditions at the outer film interface $n_{\mathrm{O}}(0, t)$ and at the film-substrate interface $dn_{\mathrm{O}}(d, t)/dy = 0$. The initial condition with respect to OC for films of different thicknesses was set according to the experimental data.

Another feature concerns the choice of the boundary condition at the outer interface of the film. Despite the relatively high partial pressure of oxygen in the air atmosphere [19], the value of OC = 3 was used to solve equation (4) at $T = 873$ K. The fact is that in films of thickness $d = 25-75$ nm, as we found earlier, see, for example, [23] [1], there is a „stabilization effect“ OC = 3. The effect is that in the process of sequential saturation of the film material with oxygen in air (under conditions of excess partial pressure of oxygen in the gas medium [19]) at relatively low temperatures of heat treatment ($T < 973$ K) the maximum achievable value $T_{\mathrm{C}}(z = x - 2\delta)$ corresponds to $\delta \approx 0$ (or OC ≈ 3). In our opinion, the effect is caused by mechanical stresses in the epitaxial film (in this case, „compressive“), caused by a small mismatch between the crystalline parameters of the film and the substrate. It consists in the presence of an energy barrier in the vicinity of the outer interface that prevents the excess oxygen atom from being embedded in the film crystal lattice and the formation of a cation-deficient cell. I.e., the film state with OC = 3 is the most energetically stable, and mechanical stresses (in this case, „compressive“) enhance this effect. As a result, heat treatment of the films in air at a relatively low temperature (873 K) does not lead to the formation of excess OC. The temperature factor, e. g., $T = 1073$ K, is important for the formation of the oxygen excessive state.

As it turned out, a good approximation of the experimental data by the theoretical curves without taking into account the dependence of the diffusion coefficient on the film thickness, as well as in work [21], is not obtained. The calculated curves poorly reproduce the appearance of the time-saturated experimental dependences $\delta(t)$. The calculated curves in Fig. 1, *a–c* are obtained considering this dependence $D(y)$, parameter values for three curves correspond to $U_1 = 2.7$ eV, $\Delta U = 0.37$ eV, $y_0 = 0.5d$.

Fig. 3 shows the dependence of the oxygen diffusion coefficient in the vicinity of the outer film interface $D(y = 0)$ for films of different thicknesses.

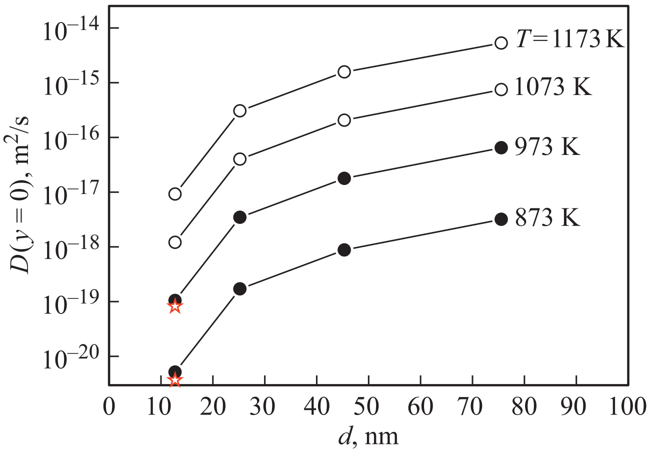

Figure 3. Dependence of the diffusion coefficient corresponding to the external interface of the films ($y = 0$) on the thickness of the films. The unfilled circles represent the calculated data within the described model.

3. Discussion

As follows from the data in Fig. 3, as the film thickness decreases, the diffusion coefficient markedly decreases. From the physical point of view, the effect is clear. Indeed, the crystal cell size of manganites depends on the OC, and the size of the oxygen-deficient cell is larger than the complete [28]. The transition of an oxygen atom from a complete cell to a neighboring oxygen vacancy is associated with redistribution of mechanical stresses. For this reason, the diffusion process of a mechanically stressed film is energetically more costly. I.e., the fact that the diffusion coefficient decreases with decreasing film thickness and increasing mechanical stress is expected.

The appearance of the obtained dependence $D(y = 0)$ in Fig. 3, demonstrating an increase in the rate of change as the film thickness decreases, is also expected based on the spatial distribution of mechanical distortions of the crystal lattice over the film thickness. Indeed (see work [29]), c — the lattice parameter of $\mathrm{La_{0.7}Sr_{0.3}MnO_{3-\delta}}$-films depends significantly on the material of single-crystalline substrates (in particular, $\mathrm{SrTiO_3}$, $\mathrm{NdGaO_3}$ and $\mathrm{LaAlO_3}$) and changes relatively little at 100–300 nm film thicknesses. Relatively large and abrupt changes in the crystal parameter of the LSMO-film on an $\mathrm{NdGaO_3}$-substrate occur at a distance of $d < 20-30$ nm from the film-substrate interface. This trend is evident in the dependence $D(d)$ in Fig. 3 and on a qualitative level testifies to the connection of the nature of this dependence with the mechanical stresses of the film material.

The greatest mechanical stresses are present in our 12.6 nm-thick film, which raises the question of the adequacy of calculating the oxygen index value using the $T_{\mathrm{C}} - \delta$ diagram with a fixed numerical correction. In the region of thicknesses $d = 25-100$ nm, the possible error due to small changes in mechanical stresses, judging from

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[1] Effect of oxygen content „stabilization“ was especially considered in the paper [33].

our data, is not critical. At the same time, the calculation of the oxygen index in the film, which is 12.6 nm thick, seems to overestimate δ (curve *1* in Fig. 1, *d*). Indeed, the OC = 3 in the film is not achieved after prolonged heat treatment at $T = 873$ K, as well as when the temperature is increased to 973 and 1073 K. Further continuation of the heat treatment at $T = 1073$ K leads to a radical decrease in conductivity and the value of T_C, which indicates the formation of significant excess OC. If we assume that the effect is due to mechanical stresses in the lattice only, and that OC = 3 is achieved after heat treatment at $T = 973$ K, then the correction value to be made in the diagram $T_C - \delta$ should be increased to 31 K. In this case, the experimental data approximate well the theoretical curve (solid curve *2* in Fig. 1, *d*), with fixed parameters — $y_0 = 0.5d$, $\Delta U = 0.37$ eV, $U_1 = 2.7$ eV, which were used for films of greater thickness. In Fig. 3, the corresponding values of $D(y = 0)$ are marked with asterisks. As can be seen, making a larger correction in the diagram $T_C - z$ to calculate the oxygen index slightly shifts the numerical values of the diffusion coefficient, but it does not change the qualitative view of the dependence $D(y = 0)$ on the film thickness.

The indirect method at the level of more–less allows to characterize the change of OC in films of different quality, but for quantitative assessment, it is important to have high quality films with respect to the crystal structure and chemical composition. The $T_C - \delta$ diagram must be calibrated. The reason for the lower temperature values T_C in films, 20−100 nm thick, compared to bulk monocrystalline samples, may be due to several factors. In addition to the mechanical stresses of the film and OC, T_C is affected by various imperfections in the form of deviations from the stoichiometry of the film material by cationic composition, non-uniform distribution of elements in the micro- and macroscale, surface roughness and the presence of other substrate defects, that is, unrecoverable defects associated with the imperfection of a particular technology. OC variation in oxygen-deficient films can be reversible, but organizing a procedure that ensures the achievement of stoichiometric OC in LSMO films also proves to be a difficult task, since it requires taking into account the effects described above. In particular, it can be stated that the result of heat treatment under the same conditions for films of different thicknesses and on different substrates will be different with respect to OC. The application of higher temperatures $T = 1073-1173$ K during heat treatment of films causes the formation of excess OC, while the concentration of oxygen vacancies in the volume decreases and cation-deficient cells are formed in the near-surface layer of the film.

The films of greater thickness $d = 150-300$ nm, produced by the DC-magnetron method, are characterized by a less perfect crystal structure, so the OC variation in them should be considered separately. In particularly, in our relatively thick films, the formation of excess OC during heat treatment in air also occurs at relatively low temperatures (at $T = 873$ K). Films of smaller (a few nanometers) thickness should also be considered separately, since they are not yet two-dimensional, but no longer a three-dimensional system [31,32].

Conclusion

Oxygen diffusion in epitaxial oxygen-deficient $La_{0.8}Sr_{0.2}MnO_{3-\delta}$ films of 12.6−75.4 nm thickness during step-by-step heat treatment in air was studied. Quantification of changes in the oxygen index of the film material was carried out by the indirect method.

It was found that the value of the oxygen diffusion coefficient decreases significantly along the film thickness depending on the distance from the outer interface, and that the values of $D(y = 0)$ in the vicinity of the outer interface decrease as the film thickness decreases. The effect is due to the action of mechanical stresses caused by a small discrepancy between the crystalline parameters of the film material and the substrate, and is observed both in the case of tensile and compressive mechanical stresses.

It is shown that the indirect method of quantifying the OC for films with a thickness of ~ 12 nm overestimates the oxygen index and works well for LSMO films on $NdGaO_3$-substrates with a thickness of 25−75 nm.

Conflict of interest

The authors declare that they have no conflict of interest.

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