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Research articles

Observation of ferromagnetism in a thin $SrIrO_3$ film contacting with a $La_{0.7}Sr_{0.3}MnO_3$ film

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A B S T R A C T

In this paper, we demonstrate for the first time an experimental observation of ferromagnetic ordering at temperatures below 60 K in $SrIrO_3$ film which was the upper layer in $SrIrO_3/La_{0.7}Sr_{0.3}MnO_3$ bilayers. Epitaxial bilayers were deposited on (1 1 0) $NdGaO_3$ substrates, after which the temperature dependences of the electron spin resonance spectra of the fabricated heterostructures were investigated. The analysis of these spectra showed that under certain conditions, the magnetization of the $SrIrO_3$ layer reached a value of ~1 μ_B/Ir, comparable to the magnetization of the $La_{0.7}Sr_{0.3}MnO_3$ layer that transfers into a ferromagnetic state at temperatures of 320–350 K.

1. Introduction

Transition metal oxides are widely used in the manufacture of film structures, which in turn are considered as promising elements of spintronics. In the last decade, much attention has been attended to the study of iridium oxides (iridates) as typical representatives of 5d transition metals [1,2]. Such interest is due to the presence of a strong spin-orbit coupling in these materials, which, in turn, opens up opportunities for the manifestation of various topological and significant spin-transport effects. Among the strontium iridates $Sr_{n+1}Ir_nO_{3n+1}$, a special place is occupied by $SrIrO_3$, which, in contrast to the substantially layered Sr_2IrO_4 insulators with one separate IrO_2 plane (n = 1) and $Sr_3Ir_2O_7$ (n = 2), has a continuous alternation of IrO_2 planes (n = ∞). $SrIrO_3$ is the paramagnetic semimetal throughout the temperature range used in this work [3,4]. At the same time, in films from 5d transition metals, the energy of spin-orbit coupling is comparable with the correlation and crystal field energies. As a result, even minor disagreements arising from contact with other layers stimulates various epitaxial stresses, which significantly affect different correlations and in particular the magnetic properties of oxide films of these metals [5,6]. For example experimental data have been published that indicate the appearance of ferromagnetic ordering in thin layers of superlattices in which SrIrO3 layers alternate with other oxide perovskites [7,8]. This stimulated us to search for additional evidence of the appearance of ferromagnetism in $SrIrO_3$ films (SIO). This work presents new data on the magnetic characteristics of $SrIrO_3$ and $La_{0.7}Sr_{0.3}MnO_3$ (LSMO) thin films in heterostructurs $SrIrO_3/La_{0.7}Sr_{0.3}MnO_3/NdGaO_3$ heterostructures in a wide temperature range.

2. Samples and measurement procedures

The member of 3d transition metals LSMO manganite was chosen as a ferromagnetic layer, because its crystallographic parameters [8,9] almost coincide with the SIO parameters [10]. This fact allows growing high-quality epitaxial heterostructures. At the same time, the Curie temperature (T_C) for LSMO films is near room temperature (usually lies in the range of 350–370 K, but begins to decrease noticeably with a film thickness < 15 nm) [11]. All these facts make it possible to carry out research in the conditions of variable magnetization of LSMO using conventional methods for changing the temperature.

In this research we used thin-film epitaxial heterostructurs from $La_{0.7}Sr_{0.3}MnO_3$ and $SrIrO_3$ grown by magnetron sputtering on single-crystal $NdGaO_3$ (NGO) neodymium galate substrates. For the deposition, the (1 1 0) NGO plane was used, which made it possible to minimize the mismatch of the crystallographic parameters between the film material and the substrate. First, an LSMO layer was deposited on the substrate, and then, without removing the sample from the chamber (in situ), a SIO layer was sputtered onto it. Heterostructures with two different thicknesses of LSMO were investigated: SIO(10 nm)/LSMO (4 nm)/NGO (sample S-1) and SIO(10 nm)/LSMO(10 nm)/NGO (sample S-2). The quality of our heterostructures was checked using X-ray diffraction (XRD) analysis.

The presence of clear peaks from both the NGO substrate and from both film layers in Fig. 1 indicates on the epitaxial relation for the heterostructure in c-direction: (0 0 1)SIO||(0 0 1)LSMO||(1 1 0)NGO. XRD investigation of the obtained heterostructures showed that the growth of the layers occurred by the "cube-to-cube" mechanism with

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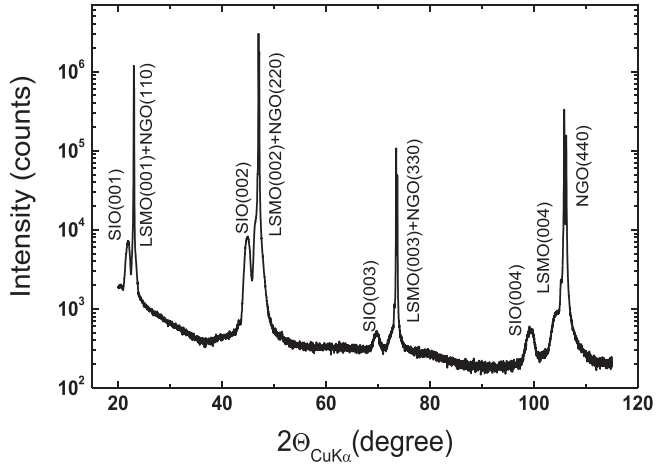

Fig. 1. The 2θ − ω XRD pattern of SIO(10 nm)/LSMO(10 nm) heterostructur deposited on (1 1 0) NGO substrate.

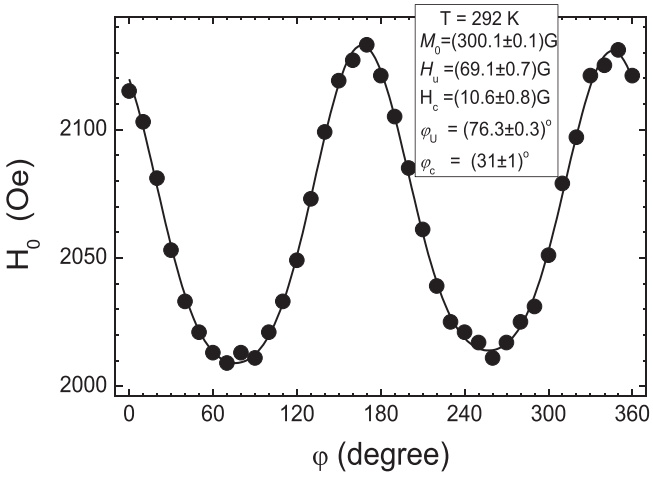

Fig. 2. The angular dependence of the magnitude of the resonance field H_0 for the S-2 sample at T = 292 K. Points are experiment data. Solid line is the fitting curve by using relation (1). The fitting parameters are shown at inset.

epitaxial relations: and [1 0 0]SIO||[1 0 0]LSMO||[0 0 1]NGO. The high quality of the layers was proved by small value of a full width at half maximum of rocking curve (0.10°–0.12°) [12].

The magnetic properties of SIO/LSMO/NGO heterostructures were investigated using the electron spin resonance spectra in the temperature range 20–300 K. To record the magnetic resonance spectra, we used the Bruker ER 200 spectrometer operating in the X-band ($\omega/2\pi$ = 9.6 GHz) with the Oxford cryogenic ESR 900. All spectra were taken on samples with dimensions 2.5 × 2.5 mm^2 under the condition that the external constant magnetic field lay in the sample plane (parallel orientation), and the magnetic component of the microwave field was directed along the normal to the film. The sensitivity of our spectrometer does not allow registering the electron paramagnetic resonance (EPR) signal from the SIO layer with a thickness of 10 nm. This means that in the entire temperature range it was possible to observe only the ferromagnetic resonance (FMR) or the magnetic resonance signal near the onset of ferromagnetic ordering in the SIO/LSMO heterostructurs. Indeed, the magnetic resonance spectra of all our SIO/LSMO/NGO structures in the temperature range 100–300 K contained only single resonance line belonging to the LSMO ferromagnetic layer.

The FMR method allows simultaneously obtaining a whole set of magnetic characteristics of a substance: magnetization, the values of various types of magnetic anisotropy and the directions of their axes, as well as a number of others. The point is that under resonance conditions, the ration that connect the frequency of the microwave field and the magnitude of the external constant magnetic field H_0 contains all these quantities as parameters [13]. In particular, for the LSMO film, which has both biaxial planar magnetic anisotropy due to crystallographic symmetry and induced uniaxial planar magnetic anisotropy due to mechanical stresses due to the mismatch between the crystallographic parameters of LSMO and NGO, the resonance ratio in the parallel orientation has the following form [14]:

$$\left(\frac{\omega}{\gamma}\right)^2 = \left(4\pi M_0 + H_0 + \frac{2K_u}{M_0}\cos^2\varphi_u + \frac{2K_c}{M_0}\frac{1+\cos^2 2\varphi_c}{2}\right)\left(H_0 + \frac{2K_u}{M_0}\cos 2\varphi_u + \frac{2K_c}{M_0}\cos 4\varphi_c\right) \quad (1)$$

Here γ is the gyromagnetic ratio, M_0 is the equilibrium magnetization, K_u and K_c are the uniaxial and biaxial magnetic anisotropy constants, respectively, and φ_u and φ_c are the angles relative to the vector $\mathbf{H}_0$, under which the corresponding easy magnetization axes are directed. Note that the magnetic anisotropy constants determine the effective anisotropy magnetic fields: $H_u = 2K_u/M_0$ and $H_c = 2K_c/M_0$. Thus, registering the FMR spectra in parallel orientation at different angles of film rotation around the normal, we obtain the angular dependence of the resonance field H_0, which is described by relation (1), with the most appropriate parameters M_0, H_u, H_c, φ_u and φ_c.

3. Experimental results and discussion

The angular dependence of the magnitude of the resonance field H_0, taken at room temperature, is shown in Fig. 2. Here, the angle φ was measured from one of the edges of the substrate. Thus, the directions of the easy axes are also determined relative to this edge. The large number of points obtained makes it possible to determine, using the relation (1), all five fitting parameters with fairly good accuracy. The data on temperature changes in both the M_0 and magnetic anisotropy parameters were obtained by analogous processing of the angular dependences of the FMR spectra taken at different temperatures.

Next, we will call special attention to the change in the parameter M_0 in the heterostructures under study with decreasing temperature.

Fig. 3a shows the change in the parameter M_0 with decreasing temperature for the S-1 and S-2 heterostructures. Note that the error in

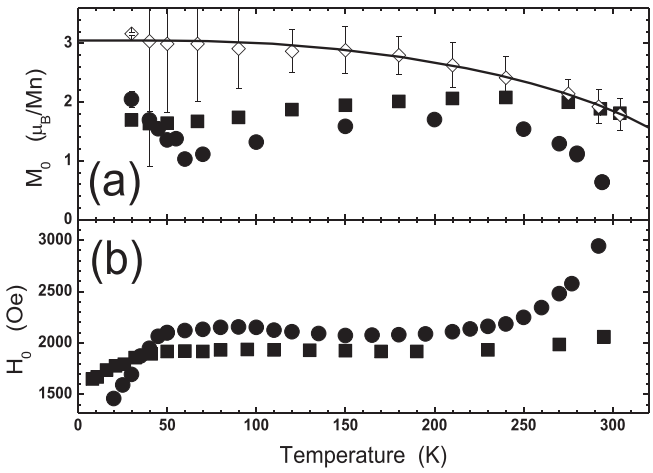

Fig. 3. Temperature dependences of M_0 parameter (a) and the magnitudes of the resonant field (b) of the FMR signal for both heterostructures, taken at the direction of the external magnetic field vector along the hard axis of the uniaxial magnetic anisotropy. Filled circles describe S-1 heterostructure. Filled squares describe S-2 heterostructure. Diamonds describe the temperature dependence of the magnetization at the S-2 structure, determined from the area of the FMR line. The solid curve is the calculation of the magnetization using the Weiss mean field theory.

the definition of the parameter M_0 does not exceed the size of the points. In addition, in Fig. 1a, open diamonds show the actual values of the magnetization for the S-2 structure, converted from the areas of the FMR lines and normalized to the M_0 value at T = 304 K. It also shows the curve calculated according to the Weiss mean field theory with the parameters T_C = 363 K and the value of saturated magnetization 3.05 μ_B/Mn. Both the diamonds and the curve describe a typical monotonic change in the magnetization of a single LSMO film with decreasing temperature [15,16]. It can be seen that for both structures, the change of the parameter M_0 with decreasing temperature is noticeably different from the real values of the magnetization.

First, when cooled from room temperature, a local maximum is observed with a subsequent decrease in the magnitude of M_0 over a considerable temperature range. Secondly, with further cooling for both heterostructures, a new increase in the parameter M_0 is observed, which begins near 60 K. The decrease of the parameter M_0 as the temperature decreases from 200 K to 60 K is associated with the presence of a conductive nonmagnetic layer in contact with the ferromagnetic LSMO layer. This effect will be discussed in another paper. Here we will discuss the reasons for the new sharp increase of the parameter M_0 in the two-layer structure of SIO/LSMO.

The point is that the parameter M_0 can be identified with the sample magnetization only in a single ferromagnetic film, for which expression (1) is obtained. If there is another layer in contact with the ferromagnetic film, an interlayer interaction may appear which will manifest itself as an additional term in the energy of the ferromagnetic film. As a result, expression (1) will no longer adequately describe the relationship between the frequency and the external magnetic field in FMR conditions. A similar picture was observed when analyzing the temperature dependence of the FMR spectra in the bilayers of SRO/LSMO [17] (here the SRO designation for $SrRuO_3$ was used). In [17], the FMR signal was also observed only from the LSMO layer and the initial analysis of the angular dependences of the FMR spectrum obtained at different temperatures was processed using relation (1). It was shown that the parameter M_0 adequately describes the magnetization of the LSMO spin system as the temperature decreases from room temperature to the Curie temperature for the SRO T_C^{SRO} = 150 K. With a further decrease in temperature (below the Curie temperature for SRO), the analysis of FMR using formula (1) led to an unreasonable increase in the parameter M_0. It was shown that for an adequate description of the FMR spectra one should take into account the energy of interlayer exchange [18], which occurs when the SRO transitions to the ferromagnetic state. Based on the results of [17], it can be assumed that, in the case of the SIO/LSMO bilayer, an increase in the parameter M_0, which starts near 60 K using relation (1), indicates the possibility of ferromagnetic ordering in the adjacent SIO layer near 60 K.

To test this hypothesis, more thorough studies of the FMR of both S-1 and S-2 were carried out in a narrow temperature range, where the development of ferromagnetic ordering in the SIO layer is expected. A more thorough analysis of the FMR spectra at temperatures below 100 K revealed the appearance of an additional magnetic resonance line, which differs from the FMR line of manganite. Fig. 4 shows several magnetic resonance spectra from the S-1 heterostructure in the temperature range (15–90) K. In all these spectra, there is a resonance line near the external magnetic field of 3400 Oe, which characterizes the EPR from the reference sample and does not belong to the heterostructure under study. Also on all spectra, the FMR line from the LSMO layer is visible. The resonance field for this line varies from 2100 Oe to 1300 Oe, which is caused by an increase in the magnetization of manganite with a decrease in temperature. And finally, as can be seen from Fig. 4, at temperatures below 70 K, a new resonance line appears, the origin of which can be explained by the ordering of the electron spin system in the SIO layer.

As noted above, the sensitivity of the spectrometer makes it possible to record the magnetic resonance from the spins in a film with a thickness of the order of 10 nm only if they are ordered. Magnetic

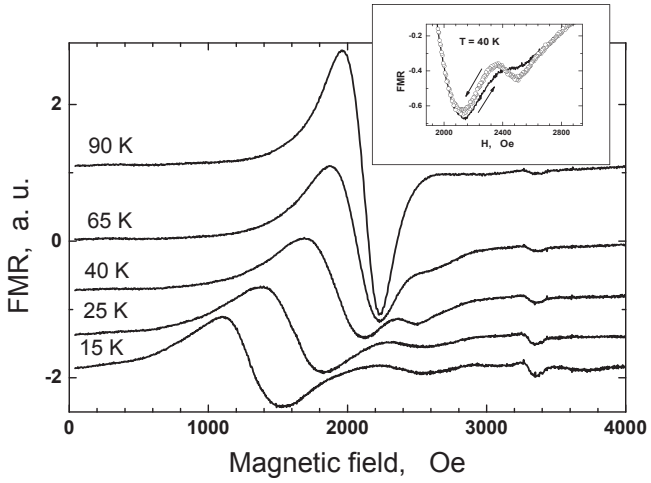

Fig. 4. Magnetic resonance spectra in the S-1 heterostructure, taken at several temperatures in the region of the supposed ferromagnetic ordering in the SIO layer. The inset shows a part of the spectrum obtained at 40 K, both in the case of an increase (solid curve) and a decrease (point) of the external magnetic field (the direction of the field change is duplicated by arrows).

resonance signals from a substrate do not occur in this temperature range. So we are dealing with the emergence of a new FMR signal in the heterostructur SIO/LSMO. An additional evidence of the fact that a new line belongs to the FMR is a narrowing of the line during a reverse sweep of the magnetic field (see inset in Fig. 4). Such a narrowing can be explained by the fact that an external magnetic field stimulates ferromagnetic ordering in the SIO film. Similar stimulation was observed before when recording the spectra of electron magnetic resonance in other manganites with a perovskite structure [19]. As a result, with the growth of the external field, the magnetic ordering becomes more homogeneous, which is reflected in the narrowing of the FMR line during the reverse course of the sweep.

Fig. 5 demonstrates the temperature dependences of the resonant fields for samples S-1 (Fig. 5a) and S-2 (Fig. 5b) in the temperature range in which the ferromagnetic ordering of the SIO layer develops. The Fig. 5a and b show that along with the typical behavior of the FMR spectrum from LSMO (compare with the temperature dependence of the resonant field in Fig. 2b) there is an additional FMR line (middle line in Fig. 4). The temperature dependence of resonance field for this line is characteristic of ferromagnetic ordering with temperature Curie around 60 K. Recall that unusual magnetic phenomena, including ferromagnetic ordering, were observed in this temperature range for thin SIO layers in contact with other perovskite-type oxides, including LSMO [7,8,20]. In Fig. 5 also the temperature dependences of the absorption line area I_{SIO} for the additional signal (see diamonds and right scale) is shown, normalized to the area of the FMR line from the LSMO layer I_{LSMO}..These dependences reflect the temperature changes in the magnetic moment in the additional resonating system, since the area of the FMR line is proportional to the total magnetic moment of the ordered spin system. Therefore, these experimental data also confirm that a new ferromagnetically ordered spin system appears in heterostructures S-1 and S-2. The magnetic moment can be recalculated into the magnetization by dividing by the layer thickness (the film areas in heterostructures are equal). As result the magnetization of the new ordered spin system reaches ~0.2 μ_B/Ir for the heterostructure S-1 and ~1 μ_B/Ir for the heterostructure S-2. The increase in magnetization in the second structure is obviously due to the increased magnetic moment of the LSMO layer, as compared with the first structure, as well as the possibly more favorable ratio between the energies of the spin-orbit coupling, the crystal field and mechanical stresses.

It can be assumed that the additional line of FMR refers to the part of the LSMO layer with a higher Curie temperature. However, there are

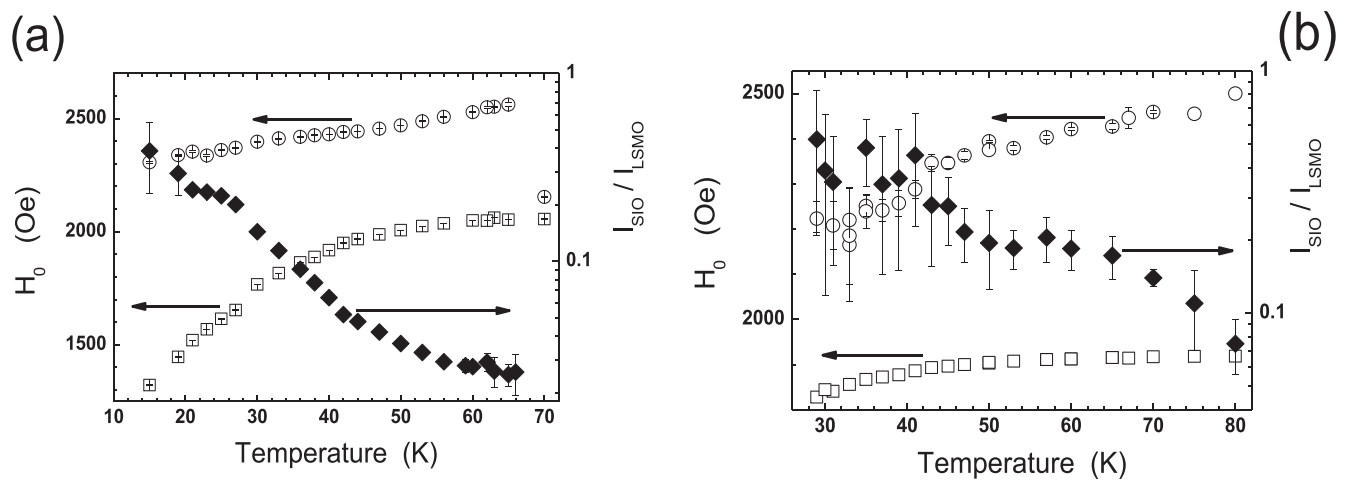

Fig. 5. (a) – temperature dependences of the resonant fields (left scale) of the LSMO line (open squares) and additional line (open circles), as well as the area of the absorption line of the additional magnetic resonance signal (right scale, filled diamonds), normalized to the area of LSMO line, for the heterostructure S-1. (b) – the same dependencies for the heterostructure S-2.

several facts that allow us to exclude such an assumption. In the process of setting up the deposition tool, we have obtained LSMO/NGO samples that contained two Mn spin systems with different T_C. Such samples gave FMR spectra containing two different absorption lines. We studied the temperature dependences of parameter M_0 for both spin systems in such samples. These dependences were monotonous and were well described by curves obtained from the Weiss mean field theory (see the curve in Fig. 3a), but, of course, with different Curie temperatures and with different values of saturated magnetization. We discussed above the conditions under which the parameter M_0 corresponds to the true value of the magnetization in a ferromagnetically ordered spin system (see explanation to Fig. 3). The difference in the magnitude of the parameter M_0 from the real value of magnetization (see the squares in Fig. 3a) clearly indicates an additional effect on the LSMO film. Ferromagnetic ordering in the SIO layer can add interlayer interaction energy to the LSMO free energy, which allows one to describe a sharp increase in the parameter M_0 at $T < 70$ K (also see explanation to Fig. 3).

As another argument in favor of our hypothesis, we studied the angular dependences of the resonance fields of the additional FMR line, which occurs at $T < 70$ K. As already mentioned above, analysis of such dependences allows, in particular, determining the directions of the easy magnetization axes. In Fig. 6a, one can see the temperature dependences for the directions of the easy magnetization axes of uniaxial magnetic anisotropy for both the LSMO layer and the additional spin system, to which we relate the additional FMR line. As a result, we obtained that the additional spin system also has planar uniaxial magnetic anisotropy, but the light axis of this anisotropy is rotated 90° relative to the direction of the axis of easy magnetization for the LSMO layer. This means that this line cannot belong to the LSMO layer. It should be noted that in Fig. 6a, the angles are measured from the direction [0 1 0] of the plane (0 0 1) in the LSMO film, and not from the edge of the substrate, as shown in Fig. 2.

Let's consider one more argument in favor of our statement that at $T < 70$ K the spin system of the LSMO layer is subjected to an additional effect that it does not experience at a higher temperature. Taking into account that the change in the parameter M_0 in the LSMO layer with decreasing temperature differs noticeably from the real values of magnetization (see Fig. 3a), we constructed similar dependences for the parameters H_u and H_c. In Fig. 6b, the dots show the temperature dependences of the values of H_u and H_c for the LSMO layer of heterostructure S-1, obtained from processing the angular dependences of the resonance fields using expression (1). The same figure shows the dependence that was constructed by averaging the temperature

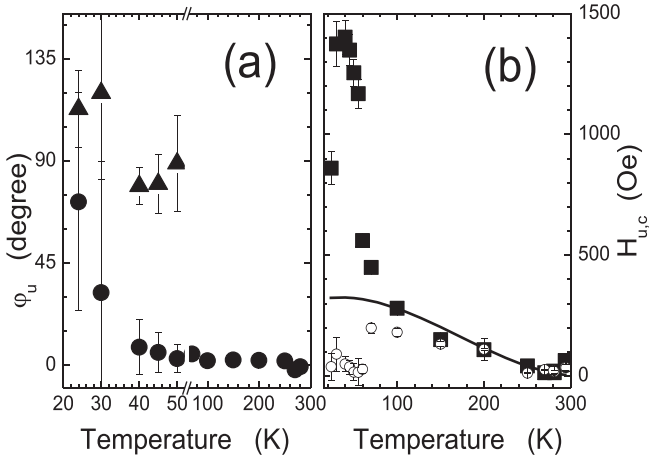

Fig. 6. (a) – temperature dependences of the directions of the easy magnetization axes in the LSMO (circles) and SIO (triangles) layers for the S-1 structure. The angles are measured from the direction [0 1 0] of the plane (0 0 1) in the LSMO film. (b) – the temperature dependence of the uniaxial magnetic anisotropy (open circles) and the biaxial magnetic anisotropy field (filled squares) in the LSMO layer; the dots are the experiment for the S-1 structure, the curve is the experimental H_c values for LSMO layer averaged over various film structures containing the LSMO layer (see text).

dependences of the H_c value obtained on four different film structures containing the LSMO layer: LSMO/NGO, SRO/LSMO/NGO, LSMO/SRO/NGO and LSMO/LMO/SRO/NGO [17] (here LMO means $LaMnO_3$). The largest deviation of the experimental points in [17] from the given curve is only 13% for the indicated four structures. Such coincidence of the temperature dependences of H_c in various film structures indicates a negligible effect of these layers on the crystallographic structure of LSMO. Note that a similar insensitivity to the temperature dependence of the H_c parameter in the LSMO film to various substrates was observed in [21]. From the Fig. 6b it can be seen that at $T < 70$ K the value of the parameter H_c in the heterostructure SIO/LSMO/NGO, determined using expression (1), is very different from the average dependence. The parameter H_u sharply changes the nature of the dependence in the same temperature range. Such a behavior of the parameters H_u and H_c can be explained by the interlayer exchange that occurs when the neighboring SIO layer transits into the ferromagnetic state. Similar dependences at $T < T_C^{SRO}$ two co-authors of this study have observed during work with bilayers SRO/LSMO, the results of which were published in [17].

Thus, we can state that, firstly, the additional absorption line that appears in our S-1 and S-2 heterostructures at T < 70 K does not belong to the LSMO layer. Secondly, this line belongs to the ferromagnetic layer directly in contact with the LSMO layer. So it belongs to the SIO layer. Third, the number of ferromagnetically ordered spins in the new ferromagnetic layer is comparable to the number of ferromagnetically ordered spins in the LSMO layer, since the areas of the FMR lines from two layers are comparable to each other. Consequently, the magnetic moments in the S-1 and S-2 heterostructures are ordered at a considerable thickness of the SIO layer, and not only close the interface.

It is known that at the contact of the manganite layer with SIO, the transfer of electrons from Ir to Mn occurs (see, for example [5,22]). The resulting excess concentration of charge carriers (electrons in manganite and holes in iridate), exceeding a certain critical value, provides to the emergence of ferromagnetic ordering in iridate [22]. It can be assumed that in our heterostructures the charge transfer provides a critical amount of doped carriers at a sufficient depth of the SIO layer. Nevertheless, we understand that for complete confidence it would be good to carry out estimations from first principles based for example on density functional theory, but this task is beyond the scope of this work.

4. Conclusion

Thus, in this work, experimental data are presented that demonstrate in the temperature range 60–70 K the occurrence of ferromagnetism in $SrIrO_3$ epitaxial films, which are part of bilayer SIO/LSMO deposited on the (1 1 0) plane of the NGO substrate. In this case, the magnitudes of the magnetization in the films of iridate are comparable with the magnetization of the manganite films. All this is of course a consequence of the large magnitude of the spin-orbit coupling in 5d transition metals. Our results are further proof of the diversity of the phase characteristics of 5d transition metals. Such diversity promises to observe new interesting phenomena in the future.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jmmm.2019.165979.

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