Magnetic and magnetotransport properties of ultrathin \( \text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3 \) epitaxial films embedded in \( \text{SrRuO}_3 \)

F Bern\(^1\), M Ziese\(^1\), I Vrejoiu\(^4\), X Li\(^1\) and P A van Aken\(^3\)

\(^{1}\) Universität Leipzig, Fakultät für Physik und Geowissenschaften, Abteilung Supraleitung und Magnetismus, Linnéstraße 5, D-04103 Leipzig, Germany

\(^{2}\) Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, D-70569 Stuttgart, Germany

\(^{3}\) Max-Planck-Institut für Festkörperforschung, Stuttgart Center for Electron Microscopy, Heisenbergstrasse 1, D-70569 Stuttgart, Germany

\(^{4}\) Present address: Universität zu Köln, II. Physikalisches Institut, Zülpicher Str. 77, D-50937 Köln, Germany.

E-mail: ziese@physik.uni-leipzig.de and vrejoiu@ph2.uni-koeln.de

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Abstract

The structural, magnetic and magnetotransport properties of \( \text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3 \) layers interfaced with \( \text{SrRuO}_3 \) layers were studied. High quality trilayers with coherent interfaces were fabricated by pulsed laser deposition. The thickness of the embedded \( \text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3 \) layer was varied between one and five unit cells, whereas the embedding \( \text{SrRuO}_3 \) layers were kept at a constant thickness of three unit cells. In this embedded geometry \( \text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3 \) layers are ferromagnetic, even if only one unit cell thick. Magnetization and anomalous Hall effect curves show an antiferromagnetic coupling of the layers that leads to an intricate magnetic field dependence. When this is disentangled, it can be shown that the magnetic and transport properties are mainly dominated by the corresponding properties of the constituent materials.

Extensive research effort was devoted to the study of thin ferromagnetic films grown on various substrates [1–3]. Although restricted to ferromagnetic films, this research field is very wide ranging from elemental metallic films and their spin structures [4] or their multiferroic properties [5] to oxide films and their complex interplay between spin, structure, orbital and electronic properties [6]. There is one underlying feature in these systems; thin films—unless these are free-standing—have an underlying asymmetry being adjacent to the substrate on one side and to vacuum (or often more realistically: to an unknown variety of adsorbates) on the other side. Depending on the properties of the substrate, this proximity might induce obvious changes of the film properties, e.g. exchange biasing of a ferromagnetic film in contact with an antiferromagnetic substrate [7]. The issue of magnetic proximity, however, might be far more subtle, extending to less obvious changes [8]. The situation might be similar to proximity effects in superconductors [9], albeit with the difference that the relevant interaction length scales are sensitive to coupling type and interface characteristics [10]. Apart from subtle proximity effects one has to consider the effect of strain and, for perovskite-like oxides, the effect of the oxygen octahedral tilt and rotation mismatch at interfaces between dissimilar materials.

In this work we study the magnetic and magnetotransport properties of embedded \( \text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3 \) (LBMO) films with thicknesses ranging from one to five unit cells (uc). In recent years a series of studies on exposed thin manganite films [11–14] showed that these films lost their ferromagnetism and metallicity below a critical thickness of about eight unit cells [14]. This was attributed to the strain exerted by the substrate (either \( \text{SrTiO}_3 \) or \( \text{LaAlO}_3 \)) and the surface symmetry breaking; a tetragonal distortion of the unit cell would lead to an energetic splitting of the \( d^2 \) and \( d_{x^2-y^2} \) orbitals, destabilizing the double exchange interaction, while stabilizing the superexchange interaction and thus tipping the balance between ferro- and antiferromagnetism [15, 16]. We have shown before that \( \text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3 \) (LSMO) layers in \( \text{LSMO}/\text{SrRuO}_3 \) superlattices retain their ferromagnetism down to a thickness of at least two unit cells [17]. This result was criticized, since extended electronic states might form in these superlattices. Moreover, superlattices consisting of 15 bilayers might not be in a homogeneous strain state. Similar results were obtained for \( \text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3/\text{CaRuO}_3 \) superlattices [18, 19]...
and for La$_{0.7}$Sr$_{0.3}$MnO$_3$/TbMnO$_3$ superlattices [20], albeit with a rather small Curie temperature in the latter system. Here we report results on single LBMO layers embedded in SrRuO$_3$ (SRO) layers. In this embedded configuration the LBMO layers retain their ferromagnetic properties down to a film thickness of one unit cell. Further, the antiferromagnetic interaction between LBMO and SRO is studied at these small film thicknesses.

The trilayers were fabricated by pulsed laser deposition (KrF laser, 248 nm) from stoichiometric polycrystalline targets onto slightly vicinal (0.1°) SrTiO$_3$ (100) substrates. Prior to deposition substrates were etched in buffered HF acid and annealed at 950 °C for 2 h in air to achieve a continuous TiO$_2$ surface termination. Substrate temperature and oxygen partial pressure during deposition were 700 °C and 0.14 mbar, respectively. The growth was monitored by a reflective high energy electron diffraction (RHEED) system, thus giving direct control on the layer thicknesses. LBMO was chosen to minimize the strain; the LBMO lattice constant (0.3910 nm) is larger than that of LSMO (0.388 nm), and closer to the lattice constants of SrTiO$_3$ (0.3905 nm) and SRO (0.3930 nm). Various sets of samples were grown. In this work we focus on a sample series with SRO layers of constant thickness of 3 unit cells (3uc) embedding LBMO layers of varying thickness between 5 uc and 1 uc.

These trilayer nanostructures were studied by high angle annular dark field imaging (HAADF) and electron energy loss spectroscopy (EELS) at 200 keV in a c$_z$-corrected scanning transmission electron microscope (STEM) (JEOL ARM 200CF). Magnetization measurements were made with Quantum Design models MPMS-7 and MPMS-XL SQUID magnetometers. The diamagnetic contribution from the substrate was subtracted from the data. All substrates showed a ferromagnetic magnetization contribution [21] of the same magnitude as the ferromagnetic layers; therefore, the magnetization versus magnetic field measurements could not be evaluated. The resistance and Hall effect measurements were made in van der Pauw configuration using a He flow cryostat with a magnetic solenoid up to 8 T. The Hall resistivity was calculated from the Hall resistance using the combined thickness of embedded and embedding layers.

Figure 1(a) shows a HAADF image of the 3 uc thick embedded LBMO film, where no structural defects were observed. In bulk, SrRuO$_3$ is orthorhombic [22] and La$_{0.7}$Ba$_{0.3}$MnO$_3$ rhombohedral [23]. SrRuO$_3$ thin films grown on SrTiO$_3$ (100) were found to be monoclinic [24, 25] with a crossover to tetragonal symmetry at very small film thickness [26]. From these data we believe that the present SRO layers are monoclinic, whereas the
LBMO layers have tetragonal symmetry [27]. Further, the trilayer is strained so that the in-plane lattice parameters of the SRO and LBMO layers adopt the value of the STO substrate lattice parameter.

The EELS chemical mapping of the trilayer is also shown in figure 1 (b), where the atomic distribution of Ti, La, Ba, Mn across the thin film can be seen. Inhomogeneous distribution of La and Mn at the interface adjacent to the SRO layers was observed with a diminished intensity for the La top and Mn bottom atomic planes, indicating interface chemical roughness of around one atomic plane with intermixing between Sr and La, Mn and Ru. Moreover, Ba atoms are hardly seen, because of the low concentration; in the Ba mapping only one intense atomic spot is clearly seen, whose intensity is inversely correlated to the corresponding La atom at the same position, indicating the substitution of La with Ba. However this locally probed chemical inhomogeneity does not affect the overall properties of the layer on a large scale. According to the fine structure of Mn-$L_{2,3}$ edges in the EELS spectra of figure 1(c), no chemical shift is observed among these three Mn atomic planes and the Mn valence is estimated to be around $3.5$. Identical Mn valence consistency was also observed in a 9 u.c. thick LBMO film.

Magnetization data are shown in figures 2(a) and (b) with a magnetic field of 0.1 T applied during field cooling either parallel or perpendicular to the film plane, respectively. The temperature dependence of the magnetization data is similar to that of LSMO/SRO superlattices [28]: coming from high temperatures the LBMO film undergoes a paramagnetic-to-ferromagnetic transition; on further cooling the SRO layers become ferromagnetic and orient their magnetization in general antiparallel to the LBMO magnetization. This is due to the antiferromagnetic coupling between Mn$^{3+/4+}$ and Ru$^{4+}$ spins mediated by the intermediate oxygen ion [28]. From the magnetization measurements in the two field directions one obtains a qualitative idea of the magnetic anisotropies. At high temperatures, above the Curie temperature of the SRO layers, the in-plane LBMO magnetization is always larger than the out-of-plane LBMO magnetization; therefore, the magnetic hard axis of the LBMO layers is along the layer normal. At low temperatures in the ferromagnetic phase of the SRO

![Figure 2](image_url)
layers, we believe the anisotropy of the LBMO layers to be mainly unchanged. The SRO layers tend to have larger out-of-plane than in-plane magnetization and thus a magnetic easy axis along the direction of the layer normal. This is in agreement with single SRO films [25]. As criterion for the determination of the Curie temperature the inflection point of the magnetization for a magnetic field of 0.1 T applied along the easy axis was used. This yields the values in figure 2(c). The Curie temperatures of the SRO layers are roughly independent of the LBMO film thickness; this is expected, since the individual SRO layer thickness was always three unit cells. The Curie temperatures of the LBMO films decreases from 272 K down to 169 K for the 1 unit cell thick LBMO film. We conclude that we succeeded in fabricating samples with an embedded ferromagnetic LBMO film. The Curie temperature decreases significantly below the bulk value of 330 K, possibly due to finite size effects.

The resistivity of the embedded LBMO films was dominated by conduction processes in the SRO layers, since SRO has a rather high conductivity; the resistivity as a function of temperature is therefore similar to that of single SRO films and is not shown here. We have noted before that in LSMO/SRO heterostructures the longitudinal resistivity is in general dominated by the SRO layers; however, the Hall resistivity might yield indirect information on the conductivity and magnetic state of the LBMO films [17], since it is determined by a normal Hall effect proportional to the magnetic induction and an anomalous contribution proportional to the magnetization due to the spin–orbit interactions: \( R_{xy} = \mu_0 (R_\parallel M + R_\perp H) \) with the ordinary and anomalous Hall coefficients \( R_\parallel \) and \( R_\perp \), the applied field \( H \) and the magnetization component \( M \) perpendicular to the film.

Figure 3 shows the Hall effect of the embedded LBMO films as a function of magnetic field at temperatures above the Curie temperature of the SRO layers. At 150 K a sharp non-hysteretic anomalous Hall effect is seen in magnetic fields below 2 T which is due to the ferromagnetic LBMO films. This contribution can only be seen, when the conductance of LBMO film and embedding SRO layers is of the same magnitude; this shows that the LBMO films are both ferromagnetic and conducting. At 150 K, the high field Hall effect is dominated by both the Hall effect of LBMO and the Hall effect of SRO in the paramagnetic phase that is weakly nonlinear in magnetic field [29]. At the higher temperatures of 200 K and 250 K the anomalous Hall effect contribution of the embedded films remains, but becomes nonlinear for the 1 and 2 unit cell thick films as they enter the paramagnetic phase at 169 K and 217 K, respectively.
The Hall resistivity at low temperatures has a complex magnetic field dependence that reflects the interplay between antiferromagnetic coupling between LBMO and SRO layers, the Zeeman energy and the magnetocrystalline anisotropy. It is known that SRO is an electron, LBMO a hole conductor; further the anomalous Hall constant of LBMO is negative [30], whereas that of orthorhombic SRO has a more complicated temperature dependence, being negative at low and positive at higher temperatures [29, 31, 32]. In contrast, the anomalous Hall coefficient of tetragonal SRO is positive in the whole temperature range [29]. With this in mind, one might interpret the Hall resistivity curves shown in figure 4 as follows. Both samples show switching events in large magnetic fields up to 4 T; these are undoubtedly from the SRO layers, since LBMO is a soft ferromagnet. In case of the 1 uc thick embedded LBMO film at 30 K and 50 K the hysteresis curves follow standard magnetization curves of a hard ferromagnet with the magnetic field applied close to or along the easy axes. The anomalous Hall constant, \( R_A \), which is obtained from extrapolation of the linear high field part to zero field, is positive. This indicates that the embedding SRO layers might have tetragonal symmetry. However, since the \( \langle 100 \rangle \)-axes of tetragonal SRO are magnetically hard, the latter conclusion is inconsistent with the fact that the film normal is an easy axis, see also the magnetization data in figure 2. At 3 K the anomalous Hall constant is negative, but small; between 2 T and 4 T an incongruent reversal process occurs. Overall, the behavior of the anomalous Hall constant of this sample is similar to that of Pr\(_{0.7}\)Ca\(_{0.3}\)MnO\(_3\)/SRO superlattices with orthorhombic SRO layers [32]. It is instructive to compare the data of the 1 and 5 uc thick embedded films in figure 4. In case of the 5 uc thick embedded film the anomalous Hall constant (as determined by extrapolation) is negative at low temperatures. However, the curves, especially those at 30 K and 50 K, show a complex field dependence. Whereas the magnetization of all layers is oriented along the magnetic field at high fields, at low fields a reversal of the SRO magnetization occurs at temperatures of 30 K and 50 K. This is due to the fact that the value of the LBMO magnetization is larger than that of the SRO layers, such that the LBMO magnetization is stabilized along the field direction. Driven by the antiferromagnetic coupling, below about 2 T the SRO magnetization rotates antiparallel to the applied field. Therefore the actual value of the anomalous Hall effect at zero field has the opposite sign compared to the extrapolated value. This behaviour is schematically illustrated in figure 4(b) by arrows showing the magnetization direction of the individual layers in the different configurations along a field sweep. At 10 K the rotation of the SRO magnetization is incomplete as indicated by the dips in the
curve; probably the large magnetocrystalline anisotropy stabilizes the SRO magnetization direction at this temperature. With this interpretation we might return to the interpretation of the data in figure 4(a). In that case the SRO magnetization is larger than the LBMO magnetization, such that the former might be stabilized along the field direction by the Zeeman energy. The LBMO magnetization might then gradually rotate with respect to the field into an antiparallel direction in zero field; in conjunction with the negative anomalous Hall constant of LBMO this might result in a positive anomalous Hall constant of the embedded film. On the other hand, for a 1 unit cell thick embedded LBMO film it might be misleading to interpret the experimental results by an interplay of independent SRO and LBMO layers; instead it might be more appropriate to start from a hybrid magnetic and electronic configuration of the heterostructure.

The anomalous Hall constants $\mu_0 R_A$ of the two layers were determined by comparing the extrapolation of the high field Hall effect to zero field with the value at zero field based on the assumption of AF-coupling: the carrier density was calculated from the slope $R_H$ of the high field Hall effect using $n = 1/\epsilon R_H$. The corresponding values are shown in figure 5. The separation of the two anomalous Hall constants is not satisfactory in case of the 3 uc thick film due to a magnetization rotation process at low fields, see below. The temperature dependence of the anomalous Hall constant attributed to SRO is reminiscent of ultrathin orthorhombic SRO films [29, 31, 32], while the LBMO films contribute a negative, weakly temperature dependent term. Astonishingly, the anomalous Hall effect of the present embedded LBMO films is much easier to understand as a superposition of the contributions from the constituent layers than is the case in LSMO/SRO superlattices [17, 33]. In the superlattices, in the limit of ultrathin layer thicknesses, a negative anomalous Hall constant with a strikingly different temperature dependence appeared, see stars in figure 5(a). This we interpreted as arising from an interface mediated hole gas. This interpretation is corroborated by the present data, since the number of interfaces in the embedded films is much smaller than in the superlattices. The carrier density appears to indicate a transition from electron dominated conduction at low temperatures to hole

5 At high field $\mu_0 R_A = \mu_0 R_{A1} + \mu_0 R_{A2}$ at zero field $\mu_0 R_A(0) = \mu_0 R_{A1} - \mu_0 R_{A2}$.

Figure 5. (a) Anomalous Hall constant and (b) carrier density of the embedded LBMO films as a function of temperature. In (a) symbols refer to $R_{MA}$ of the SRO layers and dotted lines to the LBMO layers. For comparison, the parameters of a [2 uc LBMO / 2 uc SRO]$_{22}$ superlattice (solid stars, right axis in (a)) as well as a 5 nm (12 uc) thick SRO film (open stars) are shown.
dominated conduction at high temperatures. One has to keep in mind, however, that the high field slope of single SRO films also shows a sign change from negative to positive values \cite{32, 34}. The latter is due to the positive contribution of the anomalous Hall effect in the paramagnetic phase that is difficult to disentangle from the ordinary Hall effect above the Curie temperature of SRO. The behaviour of the carrier concentration of the embedded LBMO films is also in contrast to that of LSMO/SRO superlattices which show hole conduction for all temperatures \cite{33}. This again shows that the superlattices, in the limit of ultrathin layers, are dominated by interfacial conduction processes.

The low temperature magnetoresistance is shown in figure 6 for magnetic fields applied perpendicular and parallel to the films. For in-plane field the current density $j$ was either parallel (longitudinal configuration) or perpendicular (transverse configuration) to the magnetic field; current density and magnetic field were applied along the edges of the substrate, i.e. either along [100] or [010] of the LBMO film. For out-of-plane fields, see figure 6(a), a large hysteresis was observed with coercive fields of 2.6 T (1 uc) and 3.1 T (3 uc); in case of the 5 uc thick embedded film two coercive fields of 0.7 T and 4.5 T were observed that were associated with a partial magnetization reversal at 0.7 T and a full magnetization reversal of the SRO layers at 4.5 T, see also the Hall effect data in figure 4. In contrast, the in-plane magnetoresistance curves shown in figure 6(b) were largely reversible. From the field dependence we might conclude that the film normal is a magnetic easy axis, such that the application of an in-plane field leads to a reversible domain rotation. For in-plane fields there is a strong dependence on the relative direction between magnetic field and current density. This is due to the spin–orbit interaction that leads to anisotropic magnetoresistance. For out-of-plane fields there is no significant change in the magnetoresistance for two mutually perpendicular directions of the current density, i.e. magnetocrystalline effects play a minor role. Only in the case of the 3 uc film the effective magnetic anisotropy of the coupled layers leads to an in-plane magnetization during the reversal process and thus to anisotropy. The magnetoresistance is not saturated in a field of 8 T due to the large magnetocrystalline anisotropy of SRO \cite{25}.

![Figure 6. Magnetoresistance of the embedded LBMO films at 10 K with the magnetic field applied (a) perpendicular and (b) parallel to the film. In (b) measurements in the longitudinal (H // j) and in the transverse (H ⊥ j) configuration are shown. $j$ denotes the current density. Lines and symbols in (a) give the two in-plane directions indicating magnetization reversal via an in-plane easy axis for the 3 uc film.](image-url)
In summary, we have studied the structural, magnetic and magnetotransport properties of La$_{0.7}$Ba$_{0.3}$MnO$_3$ films embedded in SrRuO$_3$ layers. In this embedded state the manganese films retain their ferromagnetic and conducting properties down to a thickness of 1 unit cell with a sizable Curie temperature of about 170 K. Magnetization, magnetoconductance and Hall effect can in a first approximation be understood as arising from a superposition of the corresponding properties of the two constituent materials. The antiferromagnetic coupling along the Mn-O-Ru bond still persists, even for a LBMO film that is only one unit cell thick. In case of La$_{0.7}$Sr$_{0.3}$MnO$_3$/SrRuO$_3$ superlattices, theoretical work [35] indicates that the stabilization of the ferromagnetic state is due to distortions of the MnO$_6$ octahedra and a splitting of the $e_g$ levels into a lower lying 3$z^2-r^2$ and an energetically higher $x^2-y^2$ state. These distortions have been attributed to the local chemical environment rather than compressive strain, thus underlining the importance of the interface. Furthermore, our experimental finding of the effect in the LBMO/SRO system, with moderate strain as compared to the LSMO/SRO case supports this view. As a second ingredient, the charge transfer at the interface mediates a robust double exchange interaction between $x^2-y^2$ orbitals.

The present results, in comparison to previous results on La$_{0.7}$Sr$_{0.3}$MnO$_3$/SrRuO$_3$ superlattices, see [17, 33], highlight that in the superlattices a new electronic ferromagnetic state has formed, presumably related to an interfacial carrier gas, that cannot simply be understood by superposing the results of the individual layers. Future work will address a further minimization of the SRO layer thickness as well as the possibilities of electrostatic modulation of the magnetic and transport properties of these embedded films.

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