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The role of magnetoelastic strain on orbital control and transport properties in an $LaTiO_3$–$CoFe_2O_4$ heterostructure

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Abstract
Epitaxial heterostructures of $CoFe_2O_4/LaTiO_3/LaAlO_3$ have been successfully prepared by using the pulsed laser deposition technique. The magnetoresistance (MR) of the samples is negative and linear with field at $H \geqslant 2$ T, exhibiting no dependence on field directions. Nevertheless, when $H < 2$ T, the MR is negative in a field parallel to the sample plane, but positive in that along the film normal. This novel observed anisotropic MR is explained in terms of the magnetic anisotropy in the ferrimagnetic layer, as well as the magnetoelastic coupling between the two. In fields of different directions, the top $CoFe_2O_4$ layer contracts or expands in the sample plane due to the significant magnetostriction effect, changing its resistance accordingly and exerting compressive or tensile strains on the bottom $LaTiO_3$ layer. Apparently the orbital status and the one-electron bandwidth in the $LaTiO_3$ layer are altered, which leads to a change in resistance.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Manipulation of the orbital orientation of the 3d electrons in transition metals is interesting not only for a basic understanding of physical interactions, but also for future device applications. The titanate $LaTiO_3$ (LTO) (Ti^{3+}, $3d^1$) is regarded as a prototypical example of Mott–Hubbard insulator, which undergoes a G-type antiferromagnetic ordering at $T_N = 146$ K with an ordered moment of 0.45 μ_B [1]. The Jahn–Teller distortion, though not particularly strong, in conjunction with the crystal field from the displaced La ions induced by the $GdFeO_3$-type distortion [2, 3], lifts the Ti t_{2g} orbital degeneracy by about 0.12–0.30 eV [4], so that the orbital degree of freedom is frozen at low temperatures and the system becomes an orbital glass. The influence of spin–orbital coupling must also be considered important in its orbital physics though [5]. Therefore the lattice distortion promotes selective orbital occupation of the single d electron in LTO, hinting that a strain field can lead to some extent of orbital ordering (OO) [6], analogous to the effect of an external magnetic field on the orientation of spins.

On the other hand, $CoFe_2O_4$ (CFO) is a typical ferrimagnetic oxide with an inverse spinel structure, which exhibits excellent magnetic properties, e.g. high Curie temperature of 520 °C, large magnetocrystalline anisotropy, high coercivity and high saturation magnetization. Additionally, this material exhibits a significantly high magnetostriction effect. The magnetostriction coefficient is quite large (e.g. $\lambda_{100} \sim -670 \times 10^6$ and $\lambda_{111} \sim 120 \times 10^6$ or $\lambda_S \sim -110 \times 10^6$ for polycrystals [7]). Recent studies reveal that films of CFO nanopillars embedded with 3D epitaxy in a $BiFeO_3$ matrix can be ablated

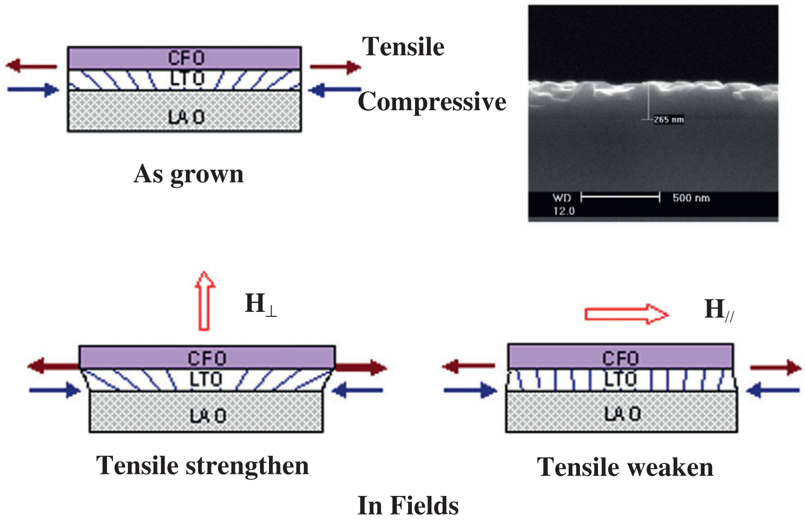

Figure 1. Scheme of the as-grown heterostructure and its strain status in perpendicular and parallel magnetic fields. The top right corner shows an SEM image of the sample cross section.

directly from a composite target, demonstrating the high chemical compatibility and stability of CFO [8, 9].

An epitaxial bilayer of LTO and CFO seems very plausible since the lattice misfit between the two materials is trivial [10]. Due to the large magnetostriction effect of CFO, an applied magnetic field will induce a change in its shape, which should dynamically alter the misfit strain in the LTO layer, as illustrated in figure 1, and thus influence the orbital status as well as the electrical conductivity in the latter [11]. In this paper, we report our efforts in preparing the LTO/CFO heterostructure, and the experimental investigation on its magnetotransport properties in fields of different directions. A novel anisotropic magnetoresistive (MR) effect was observed and explained in terms of the magnetoelastic coupling between the two layers. A possible strategy of tuning the orbital status in 3d materials by applying a magnetic field is cautiously put forward and discussed.

2. Experimental details

The films were prepared by using the pulsed laser deposition (PLD) technique (KrF excimer laser with $\lambda = 248$ nm). The LTO layer was grown on a (100)$LaAlO_3$ (LAO) single-crystal substrate at 750 °C from a ceramic $LaTiO_{3.5}$ target. The deposition lasted for 60 min at a laser repetition rate of 3 Hz. The laser energy density adopted in this experiment was $\sim$4.0 J cm^{-2}. Generally speaking, a stoichiometric LTO of Ti^{3+} can only be acquired in an extremely reducing atmosphere. The titanates have a strong tendency to incorporate La and Ti vacancies, which are frequently denoted by excess oxygen in the formula, $LaTiO_{3+\delta/2}$ [12]. To minimize the doping level δ, we grew the film in background vacuum $\leqslant 3.0 \times 10^{-6}$ Torr [13–15]. After deposition the film was cooled down at a ramp rate of 40 °C min^{-1} to room temperature. To avoid oxidation of the LTO layer, the top CFO film was also grown in high vacuum at 650 °C from a ceramic target of the nominal composition [16]. After a 30 min deposition at 7 Hz, the heterostructure was annealed *in situ* for 30 min at an elevated temperature of 850 °C, and then cooled down to room temperature at 30 °C min^{-1}. The scanning electron microscopy study has been quite awkward due to the magnetism of the CFO layer and the LTO layer is even indiscernible (see figure 1). The thickness of the CFO layer is around 260 nm and that of the LTO layer is estimated to be 230 nm, assuming an identical growth rate. The magnetotransport properties of the samples were measured by a four-probe method in an Oxford superconducting quantum interference (SQUID) measuring system. The electrodes were indium-soldered and baked afterwards at 80 °C for 30 min. Our attempts to grow LTO epitaxially on top of CFO failed due to the rather rough surface of the latter.

3. Results and discussion

3.1. Strain status in the as-grown heterostructure

The single-layer LTO ($LaTiO_{3+\delta/2}$) films grown on LAO substrates exhibit perfect in-plane and out-of-plane epitaxy, as is evident in the top panel of figure 2. Deposited in such a high vacuum, the films have an oxygen concentration very close to 3.0. The ideal $LaTiO_3$ bulk belongs to a space group *pbnm* with lattice parameters $a = 5.622$, $b = 5.613$ and $c = 7.915$ Å (PDF no. 49-426), showing trivial orthorhombic distortions from cubic. Epitaxially grown on LAO, the film becomes tetragonal and the crystallographic relationship is LTO(001)[100] ∥ LAO(001)[100]. The out-of-plane parameter is around 3.96 Å. Therefore assuming an in-plane parameter the same as LAO, 3.79 Å, the film is compressively stressed in the film plane. Accordingly, the unit cell volume shrinks by more than 10%. The rocking curve of LTO(002) is of good Gaussian shape, and its full width at half-maximum (FWHM) is only 0.17°, as shown in the left inset. The right inset depicts ϕ scans of (111) reflections from the film and the substrate, where the FWHM for LTO is only 0.98°. All the results verify the very high crystallinity of the LTO films.

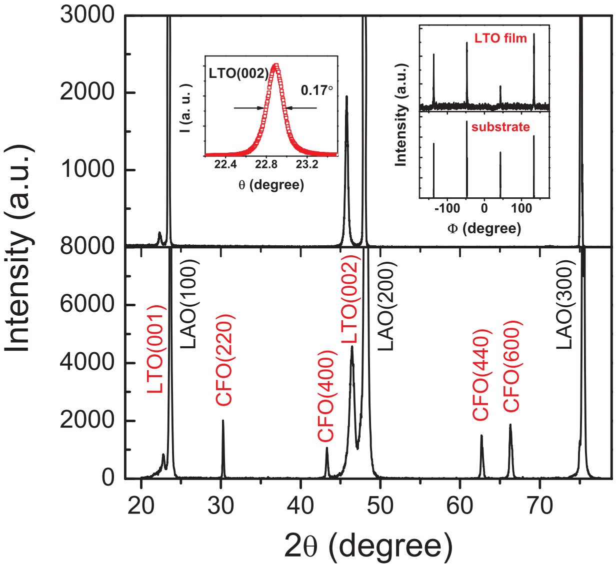

Figure 2. XRD spectra of the LTO single layer (top) and the CFO/LTO bilayer (bottom) grown on (100)$LaAlO_3$ substrates. Insets are the rocking curve of LTO(002) (left) and ϕ scan of (111) reflections of the LTO film and the substrate (right) in the single layer.

Grown on LTO, the $CoFe_2O_4$ (CFO) film shows two distinct out-of-plane orientations, (100) and (110), as shown in the bottom panel of figure 2. The CFO bulk is cubic ($a = 8.3919$ Å) with a $Fd3m$ symmetry (PDF no. 22-1086). The out-of-plane parameter of the CFO film on LTO, as derived from the spectrum, is also ~8.39 Å; while the value of d_{110} derived is ~5.94 Å, which implies a consistent lattice constant of ~8.40 Å. Therefore the misfit stress in the CFO layer, if it exists, has caused little distortion, perhaps due to its relatively large Young's modulus of 1.5×10^{12} dyn cm^{-2}. Nevertheless, as one may have noticed, peaks of the LTO layer in the heterostructure right-shifts to higher angles. The lattice constant derived is around 3.92 Å, substantially reduced, indicating a tensile stress from the upper CFO layer.

An atomically smooth surface of the bottom layer is a prerequisite for an epitaxial growth of the top layer. As seen in figure 3, the surface of the LTO layer is very smooth, noting that the height scale of the image is only 5 nm. The film is quite dense with a grain size less than 10 nm. In contrast, the top CFO layer is very rough as shown in the top panel, probably due to the high temperature annealing process. Bar-like grains of size $\sim 100 \times 500$ nm^2 are clearly seen, which are roughly aligned along the [110] or [$1\bar{1}0$] directions of the LTO film, suggesting a good in-plane epitaxy of CFO. The crystallographic relationship between the two layers therefore can be determined as CFO(100+110)[001] ∥ LTO(001)[110+$1\bar{1}0$], so two CFO unit cells may probably take the space of three LTO unit cells. Calculated from bulk values, the lattice misfit is ~−0.4%. But, due to the compression effect from the LAO substrate, the in-plane lattice parameter of LTO has been remarkably reduced. Assuming an in-plane parameter of 3.79 Å, the same as the LAO substrate, the lattice misfit between LTO and CFO increases to 4.4%. The mismatch strain

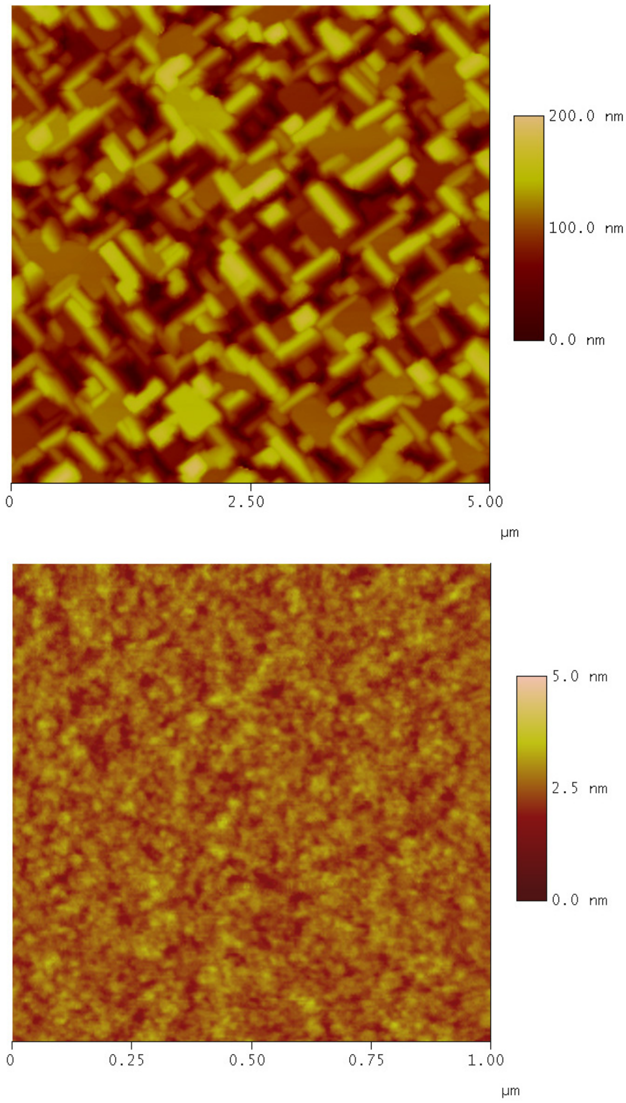

Figure 3. AFM images of the top CFO layer (top) and the bottom LTO layer (bottom). The scan areas for the two images are 5×5 and 1×1 μm^2, respectively.

is accommodated mostly by the LTO layer, as revealed by x-ray results, which then expands at the upper interface. It is the reason for the reduced out-of-plane lattice parameter in LTO after the deposition of the CFO layer. Since the LTO film is also subject to a compressive stress in the bottom interface, it is hence essentially distorted, as depicted in figure 1.

3.2. Magnetic and transport characterizations

As shown in figure 4(a), the single-layer LTO grown on LAO behaves as metallic and the temperature dependence of its resistance can be well described by a small-polaron coherent conduction model, as we have previously proposed [15]. Due to the relatively large ion radius of La^{3+}, the $LaTiO_3$ possesses a small electron correlation strength U/W, where U is the on-site Coulomb energy and W is the one-electron bandwidth [17]. Therefore it is located in the immediate vicinity of the metal–insulator boundary, with a small bandgap

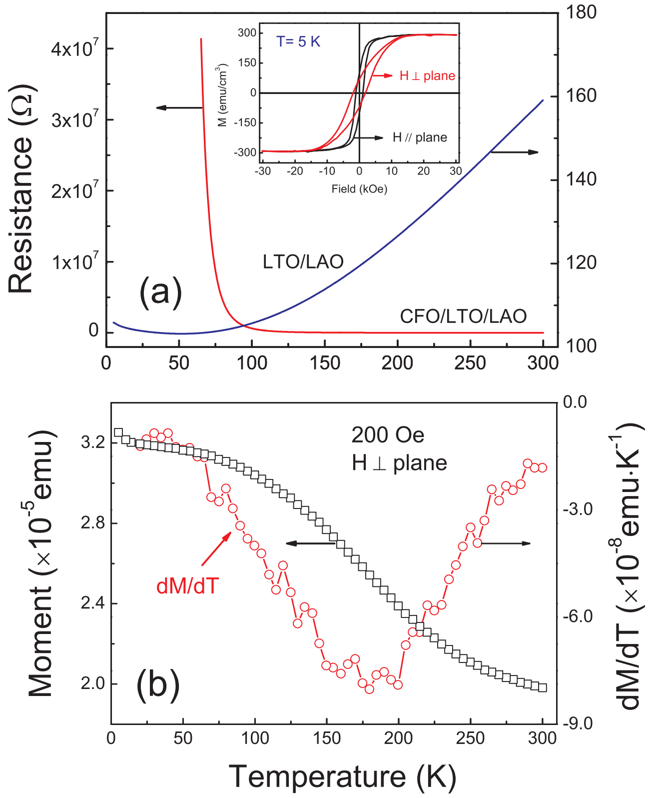

Figure 4. (a) R–T curves of a single-layer LTO film on LAO and the CFO/LTO/LAO heterostructure; (b) the M–T and dM/dT–T curves of the heterostructure measured in a 200 Oe perpendicular field. The inset shows magnetic hysteresis loops at 5 K in perpendicular and parallel fields.

of 0.2 eV. A slight oxygen excess of $\delta \sim 0.03$ would induce metallic behavior [18], as observed here in this experiment. Certainly, after deposition of the CFO layer, the transport behavior in the buried LTO layer is somewhat different from its single-layer counterpart due to the opposite strain in its up and down interface, which definitely further splits the t_{2g} level affecting the transport properties, and may even induce a metal–insulator transition. For the heterostructure, the resistance at room temperature increases by one order of magnitude. The value dramatically increases with decreasing temperature and rockets up below 80 K. We found that a single CFO layer grown on LAO substrates in vacuum becomes semiconducting itself and exhibiting a similar R–T behavior. Therefore the electrical resistance of the heterostructure actually is contributed by a series connection of the top CFO and the buried LTO layer. Consequently, the effect of misfit strain on the LTO layer cannot be directly observed.

Figure 4(b) are the M–T and dM/dT–T curves of the heterostructure, measured in a 200 Oe field applied perpendicular to the sample plane. Grown in high vacuum the CFO film is oxygen-deficient. As a result, though it exhibits a substantial magnetic moment at room temperature, the dM/dT curve finds a peak only around 180 K. The film shows a strong magnetic anisotropy, as presented in the inset, which should arise primarily from the magnetoelastic coupling with the underlying LTO layer [19], in addition to the demagnetization field. Due to the negative λ of CFO, the compressive misfit strain in the CFO layer causes the easy axes to lie in the film plane and also leads to the bar-like grain shape. As is clearly seen, at 5 K the magnetic hysteresis loop saturates below 2 T and the saturation moment M_s is around 300 emu cm^{-3}, comparable to previous reported values for stoichiometric films [20, 10]. On the whole, though the CFO film on LTO is oxygen-deficient to some extent and hence semiconducting, most of its magnetic properties are retained, including the large magnetorestriction effect.

3.3. Anisotropic magnetoresistance

The most striking observation in this experiment is seen in figure 5, where the magnetoresistance (MR) of the heterostructure in perpendicular (red) and parallel (black) magnetic fields at four different temperatures (300, 150, 90 and 65 K) are presented. The sample resistance becomes too large to be measured if the temperature is further reduced. Apparently at all temperatures, in fields higher than 2 T, the MR exhibits a negative linear field dependence with a roughly identical slope in both directions, whereas in fields below 2 T, the MR is negative in parallel fields but positive in perpendicular fields. An anisotropic magnetoresistance (AMR) effect is clearly observed. At 300 K where the magnetization is weak, the positive MR is hardly recognized, while at 60 K it is almost submerged by noises, because at this temperature the extremely large sample resistance can only be measured with a very small current of 10 nA. The AMR signal at intermediate temperatures is relatively more distinct.

In order to gain insight into the underlying mechanism of this novel AMR effect, we also carried out MR measurements in a single-layer CFO film at 90 and 65 K, and the results at 90 K are plotted in figure 6. The single-layer CFO film has been grown on an LAO substrate in conditions comparable to that of the heterostructure, which possesses a single out-of-plane orientation of (001) though. As clearly seen, the single layer behaves roughly the same as the bilayer, except some small deviations particularly in low fields. We also did MR measurements in a single-layer LTO at 300 and 60 K, but found no field dependence of the resistance. Therefore, the high field negative MR in the bilayer can be attributed mainly to the CFO layer. The carriers induced by oxygen deficiency in this layer should be spin-polarized in the ferrimagnetic background. Thereby a magnetic field can suppress spin fluctuations and reduce the film resistance. As compared with that of the heterostructure, the slope in the single CFO layer is slightly smaller in perpendicular fields, but larger in parallel fields. This may be due to the larger magnetoelastic anisotropy in the former, induced by the stronger compressive interface strain from the LAO substrate. As for the AMR effect, we have hitherto found no relevant report in CFO, though the planar Hall effect has been studied in Fe_3O_4 (of the same crystal structure) films under single-domain circumstances [21]. Since the AMR ratio increases with the applying field and saturates roughly at H_S, it should closely relate to the magnetoelastic properties of the CFO layer.

As illustrated in figure 6(b), we divide the perpendicular MR–H curves into four segments. Segment 1 is due to the

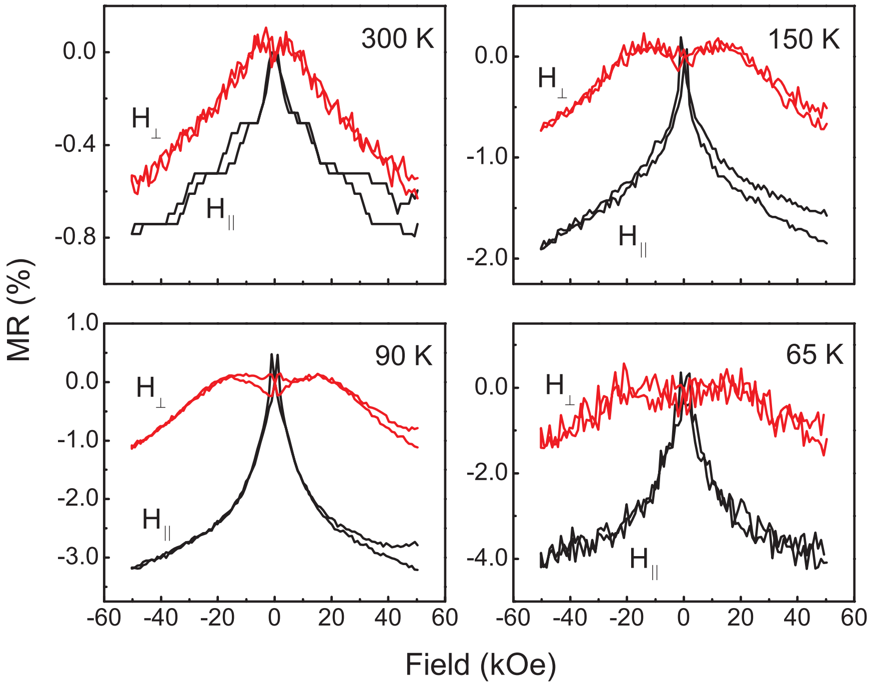

Figure 5. Magnetoresistance of the heterostructure at four different temperatures, in perpendicular (⊥) and parallel (∥) magnetic fields.

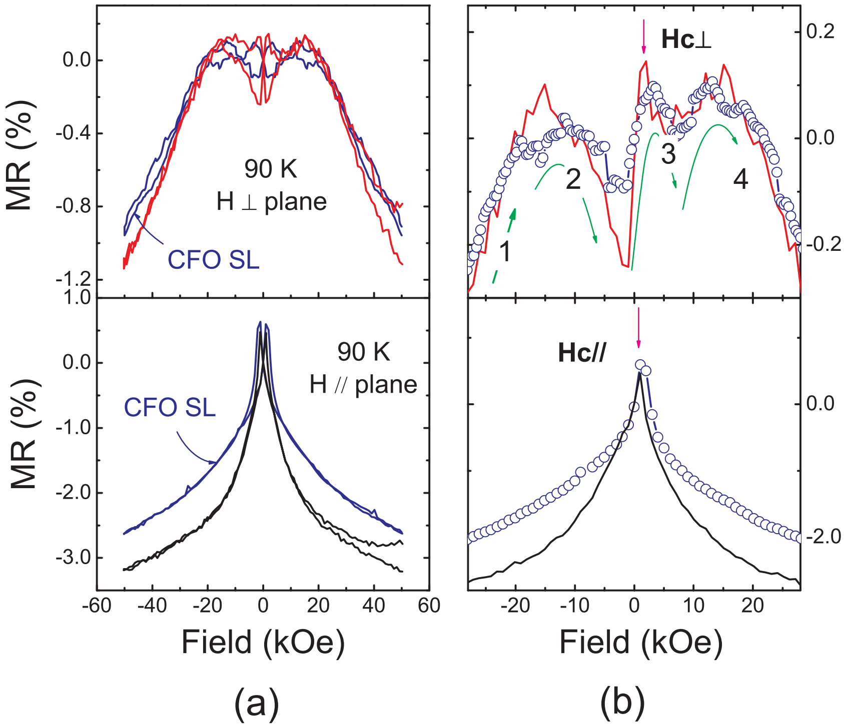

Figure 6. (a) The MR–H curves of the heterostructure and a CFO single layer (SL) measured at 90 K in perpendicular and parallel fields, respectively. (b) The low field expansion of (a). For clarity only one branch of each loop is shown.

suppression of spin fluctuations, as mentioned above. In segment 2, where the field approaches the anisotropic field, the upright magnetic spins in the CFO layer start to collapse onto the film plane and gradually rotate to the in-plane easy axes, which diminishes the spin fluctuations and leads to the resistance decline. Then the resistance finds a peak in the positive coercivity field, as a result of the magnetization reversal, which constitutes segment 3. Segment 4 is actually the inverse process of segment 2. That is, the positive MR in the CFO single layer is originated from the magnetoelastic anisotropy and the curve is distorted by the enhanced spin scattering near the coercivity field. Such fine structures are absent in the parallel curves, where the spins lie in the film plane.

Certainly, the small differences at low fields between the single layer and the heterostructure curves are due to the interfacial magnetoelastic coupling. When the field is applied out-of-plane, the CFO film expands in the film plane, so that the tensile strain that the bottom LTO layer suffered will surely be enhanced (see figure 1). As a result, the LTO lattice is further distorted, which causes the t_{2g}-level splitting and the orbital ordering to some extent. Actually, a lattice distortion may also affect the transfer interaction of the conducting electrons through the buckling of the Ti–O–Ti bonds. That is, the one-electron bandwidth W will also be narrowed upon lattice distortion [17]. Therefore the resistance of LTO increases with the perpendicular magnetic field. In contrast, when the field is applied in-plane, the tensile strain the LTO layer suffered will be alleviated, which leads to a lower resistance. This AMR signal, however, is fairly weak because in the overall resistance the contribution from the LTO layer is minor.

Therefore, the change of orbital status and electrical resistance in the LTO layer through interface coupling by an applied magnetic field has been demonstrated, though not as impressive as we have previously expected. It is obscured by the magnetoelastic anisotropy in the CFO layer. This, to the best of our knowledge, represents a first trial experiment on this strategy.

3.4. Discussion

One may have noticed that the positive and negative signals from the interface coupling are apparently asymmetric, being $\sim$0.15% and 0.67%, respectively. Since the in-plane magnetic field was applied along LTO[100], the strain it induced in the CFO layer can be estimated according to $e_{\parallel} = \lambda_{110} = \frac{1}{4}\lambda_{100} + \frac{3}{4}\lambda_{111} = -77.5 \times 10^{-6}$. In a perpendicular field, the strain in the field direction is roughly $e = \frac{1}{2}(\frac{1}{4}\lambda_{100} + \frac{3}{4}\lambda_{111} + \lambda_{100}) = -374 \times 10^{-6}$, considering the mixed out-of-plane orientations of CFO, and thus in the film plane it is around $e_{\perp} = -\frac{1}{2}e = 187 \times 10^{-6}$. Therefore, according to the above estimations, the MR ratio induced by the perpendicular field should be larger, but actually it is not. Possible reasons may include: (1) the effective field applied is smaller in the perpendicular case, due to the demagnetization effect. But since the MR value saturates in the two directions at roughly the same field, this effect can be neglected. (2) The resistance enhancement in the CFO layer at the coercivity fields, which superimposes on the AMR effect and impairs the positive signal while it enhances the negative one. This might be the most appropriate explanation. On the other hand, the magnetoelastic stress that the CFO layer exerts on the LTO layer can be estimated using $\sigma = Y \cdot e$, where Y is the Young's modulus ($\sim$150 GPa). Taking e as 100×10^{-6}, the involved stress σ is only around 15 MPa, not quite high enough. This also interprets the weak AFM signal from the interface coupling. Moreover, since the exact fraction of the LTO resistance is unknown, the pressure susceptibility of LTO is still not available at present.

4. Conclusions

In summary, high quality epitaxial bilayers of CFO/LTO have been grown on (100)LAO single-crystal substrates. Due to the large negative magnetostriction effect of the CFO material, the misfit strain at the interface stabilizes a magnetic easy plane in the CFO layer, which induces an AMR effect. Moreover, the CFO film contracts in the field direction and thus dynamically alters the misfit strain in the bottom LTO layer. This strain acts on the orbital status in LTO just like a magnetic field on spins in a magnet, thus also presumably changing its resistance. The two contributions offer the heterostructure magnetoresistance of different signs in perpendicular and parallel magnetic fields in the low field region. Obviously, the magnetoelastic energy can couple the order parameters of spin in CFO and orbital in LTO together. This work proposes a strategy to tune the orbital status in transition metal oxides by a magnetic field.

Acknowledgments

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