Transport and magnetic properties of laser ablated $La_{0.7}Ce_{0.3}MnO_3$ films on $LaAlO_3$

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$La_{0.7}Ce_{0.3}MnO_3$ is a relatively new addition to the family of colossal magnetoresistive manganites, in which the cerium ion is believed to be in the Ce^{4+} state. In this article, we report the magnetotransport properties of laser ablated $La_{0.7}Ce_{0.3}MnO_3$ films on $LaAlO_3$, and the effect of varying the ambient oxygen pressure during growth and the film thickness. We observe that the transport and magnetic properties of the film depend on the oxygen pressure, surface morphology, film thickness, and epitaxial strain. The films were characterized by x-ray diffraction using a four-circle goniometer. We observe an increase in the metal-insulator transition temperature with decreasing oxygen pressure. This is in direct contrast to the oxygen pressure dependence of $La_{0.7}Ca_{0.3}MnO_3$ films and suggests the electron doped nature of the $La_{0.7}Ce_{0.3}MnO_3$ system. With decreasing film thickness we observe an increase in the metal-insulator transition temperature. This is associated with a compression of the unit cell in the *a-b* plane due to epitaxial strain. On codoping with 50% Ca at the Ce site, the system ($La_{0.7}Ca_{0.15}Ce_{0.15}MnO_3$) is driven into an insulating state suggesting that the electrons generated by Ce^{4+} are compensated by the holes generated by Ca^{2+}, thus making the average valence at the rare-earth site 3+ as in the parent material $LaMnO_3$. © *1999 American Institute of Physics.* [S0021-8979(99)03522-7]

I. INTRODUCTION

Hole doped rare-earth manganites of the form $RMnO_3$ (R=rare-earth) have attracted a lot of attention in recent times due to their interesting magnetotransport properties arising from spin charge coupling. The parent material is a charge transfer insulator[1] in which the Mn^{3+} moments form a layered antiferromagnetic structure. The electronic configuration of Mn^{3+} is $t_{2g}^3 e_g^1$, where the three t_{2g} electrons are tightly bound with a net spin of 3/2. Hole doping is achieved in these compounds by substituting a bivalent cation such as Ca, Sr, Pb at the rare-earth site. About 30% doped compounds such as $La_{0.7}Ca_{0.3}MnO_3$ show ferromagnetism associated with a metal-insulator transition and a large negative magnetoresistance close to the ferromagnetic transition temperature (T_c). Such exotic properties of these material arise due to the strong on-site Hund's rule coupling between the localized t_{2g}^3 electrons and the electrons in the e_g band. This gives rise to a coupling between neighboring Mn^{3+}/Mn^{4+} pairs via Zener double exchange[2] mechanism. According to this mechanism[3] the hopping probability of an electron between two neighboring Mn^{3+}/Mn^{4+} pairs is proportional to $\cos(\theta/2)$. $La_{0.7}Ce_{0.3}MnO_3$ is unique in this family of compounds since the trivalent La is partially replaced by Ce instead of a bivalent cation. This compound has an orthorhombic structure with $a \approx b \approx \sqrt{2}c$. It shows a metal-insulator transition[4,5] and ferromagnetism associated with large negative magnetoresistance similar to $La_{0.7}Ca_{0.3}MnO_3$. To explain the observed behavior Mandal and Das[6] have argued that Ce could exist in Ce^{4+} state like in CeO_2 thus doping electrons instead of holes in the parent material. According to this picture the double exchange in this compound operates between neighboring Mn^{2+}/Mn^{3+} pairs giving rise to metal-insulator transition and ferromagnetism. From the theoretical point of view, the double exchange mechanism is symmetric with respect to electron or hole doping in the parent compound. A metal-insulator transition and ferromagnetism are also observed in the nominally electron doped layered manganite $La_{1.8}Y_{0.5}Ca_{0.7}Mn_2O_7$.[7,8]

In this article, we report a detailed study of $La_{0.7}Ce_{0.3}MnO_3$ films deposited by pulsed laser ablation on $LaAlO_3$. We also prepared $La_{0.7}Ca_{0.3}MnO_3$ films for comparison. We compare the effect of ambient oxygen pressure during growth on $La_{0.7}Ce_{0.3}MnO_3$ and $La_{0.7}Ca_{0.3}MnO_3$ films. To address the question of the valence state of Ce, we prepared films of nominal composition $La_{0.7}(Ce_{0.15}Ca_{0.15})MnO_3$. This compound goes into an insulating state when the film is grown at 100 mTorr oxygen pressure suggesting that the electrons generated by Ce^{4+} compensate the holes generated by Ca^{2+}. Finally, we study the effect of epitaxial strain on the metal-insulator transition temperature (T_p) in films of four different thickness.

II. EXPERIMENT

Films of $La_{0.7}Ce_{0.3}MnO_3$, $La_{0.7}Ca_{0.3}MnO_3$, and $La_{0.7}Ce_{0.15}Ca_{0.15}MnO_3$ were prepared by pulsed laser deposition using a KrF excimer laser in an oxygen atmosphere.

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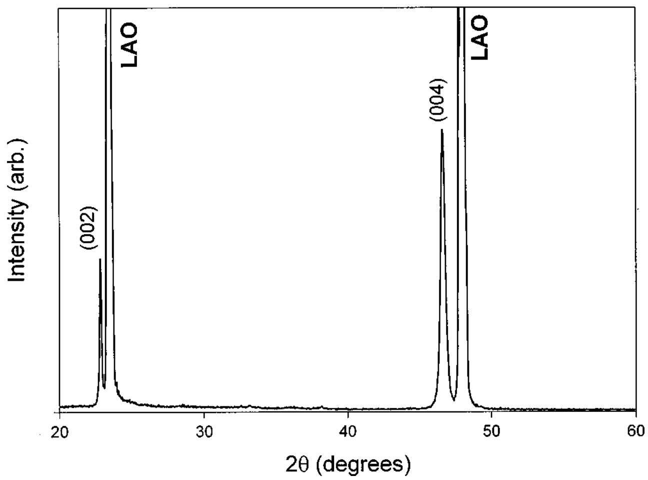

FIG. 1. X-ray θ–2θ scan of the $La_{0.7}Ce_{0.3}MnO_3$ film grown at 400 mTorr showing the (00ℓ) peaks of the orthorhombic structure. The peaks marked LAO are the $LaAlO_3$ substrate peaks.

The substrate ($LaAlO_3$) temperature was kept between 750 and 770 °C for all the films. The laser energy density was approximately 30 mJ/mm^2 with a repetition rate of 10 Hz. Films were grown at three different oxygen pressures: 100, 200, 400 mTorr. Films used for the thickness dependence study were all grown at 400 mTorr. After deposition, the laser ablation chamber was vented with high purity oxygen and the substrate cooled down to room temperature. The lattice parameters of the films were measured by x-ray diffraction on a high-resolution four-circle goniometer. The film thickness was measured using stylus method on a Dektak profilometer. Resistance and magnetoresistance were measured by conventional four-probe technique. Magnetization was measured on a Quantum Design superconducting quantum interference device (SQUID) magnetometer. Surface morphology was observed using a Digital Instrument atomic force microscope.

III. RESULTS AND DISCUSSION

A. Effect of ambient oxygen pressure during growth

To study the effect of oxygen pressure on the metal-insulator transition temperature (T_p) and the ferromagnetic transition temperature (T_c), films of $La_{0.7}Ce_{0.3}MnO_3$ were grown at three different oxygen pressures: 100, 200, and 400 mTorr. Other parameters such as deposition time, laser energy, and substrate temperature were kept constant for all the films. For comparison, $La_{0.7}Ca_{0.3}MnO_3$ films were also grown under identical conditions. All the films were seen to have $c\perp$(film plane) from the x-ray θ–2θ scan. Figure 1 shows a representative x-ray diffraction θ–2θ scans of the $La_{0.7}Ce_{0.3}MnO_3$ film grown at 400 mTorr showing the (00ℓ) peaks. Figures 2(a)–2(f) show the atomic force microscope (AFM) images of the three $La_{0.7}Ce_{0.3}MnO_3$ and $La_{0.7}Ca_{0.3}MnO_3$ film surfaces. There is a degradation of the surface morphology and decrease in grain size for the $La_{0.7}Ce_{0.3}MnO_3$ film grown at low oxygen pressure (100 mTorr) [Fig. 2(c)]. Such a degradation in the surface morphology at 100 mTorr oxygen pressure is also observed in $La_{0.7}Ca_{0.3}MnO_3$ films [Fig. 2(f)]. Figures 3(a)–3(c) show the resistance versus temperature ($R-T$) curves for the three $La_{0.7}Ce_{0.3}MnO_3$ films. It is interesting to note that the metal-insulator transition temperature (T_p) and the ferromagnetic transition temperature (T_c) increases from 246 K for the film grown at 400 mTorr to 272 K for the film grown at 100 mTorr. However, the effect of ambient oxygen pressure on T_p in $La_{0.7}Ce_{0.3}MnO_3$ is opposite to that in $La_{0.7}Ca_{0.3}MnO_3$. Figures 3(d)–3(f) show the $R-T$ curves for $La_{0.7}Ca_{0.3}MnO_3$ films grown at the same pressures. In the case of $La_{0.7}Ca_{0.3}MnO_3$ the metal-insulator transition broadens drastically with decreasing oxygen pressure while there is also a small decrease in T_p. We would like to point out that this property might be useful from the technological point of view since it allows $La_{0.7}Ce_{0.3}MnO_3$ films to be synthesized in a wider range of oxygen pressures without compromising on the metal-insulator transition width. Before elaborating further on these results, we shall discuss the effect of codoping Ce and Ca in $La_{0.7}Ce_{0.15}Ca_{0.15}MnO_3$.

In spite of the preliminary picture proposed in Ref. 6, electron doping in $La_{0.7}Ce_{0.3}MnO_3$ via Ce^{4+} has been a questionable issue since the data of thermoelectric power (TEP) in the bulk material is similar to that in hole doped manganites. However, the analysis of TEP data in these materials could be very complex, in particular, where the TEP changes sign as a function of temperature.[6] To resolve this problem, we prepared films of the nominal composition $La_{0.7}Ce_{0.15}Ca_{0.15}MnO_3$. If Ce is in the 4+ state, one would expect the holes generated by Ca^{2+} to be compensated by the electrons generated by Ce^{4+}, thus driving the system into an insulating state, as in the parent compound $LaMnO_3$. The difficulty is that the undoped compound (like $LaMnO_3$) has a tendency to get overoxygenated, generating cation vacancies[9–11] and it can show a metal-insulator transition just like the doped material.[12] However, if the compound $La_{0.7}Ce_{0.15}Ca_{0.15}MnO_3$ is compensated for electrons and holes the effect of varying ambient oxygen pressure during growth should be much more drastic than in $La_{0.7}Ca_{0.3}MnO_3$ or $La_{0.7}Ce_{0.3}MnO_3$ films since one should be able to suppress the carriers by decreasing the oxygen pressure. In Figs. 4(a) and 4(b), we show the $R-T$ curve for $La_{0.7}Ce_{0.15}Ca_{0.15}MnO_3$ films grown at 400 and 100 mTorr oxygen pressures. The film grown at 400 mTorr shows a metal-insulator transition around 230 K. However, the film grown at 100 mTorr becomes an insulator having resistance four orders of magnitude larger at 55 K than the films grown at 400 mTorr. Comparing this result with the effect of varying oxygen pressure in the same range in $La_{0.7}Ca_{0.3}MnO_3$ and $La_{0.7}Ce_{0.3}MnO_3$ (Fig. 3), we see that the low temperature metallic phase in these two compounds is never fully suppressed. The drastic increase in the resistance and the suppression of metallicity in the $La_{0.7}Ce_{0.15}Ca_{0.15}MnO_3$ film grown at 100 mTorr thus indicates that electrons and holes are compensated in this compound.

Having thus established that Ce is in 4+ state, one can now look more carefully at the data on $La_{0.7}Ce_{0.3}MnO_3$ and $La_{0.7}Ca_{0.3}MnO_3$ in Fig. 3. First, the increase in T_p with decreasing oxygen pressure in $La_{0.7}Ce_{0.3}MnO_3$ can be understood as follows. In the parent compound $LaMnO_3$, the Mn

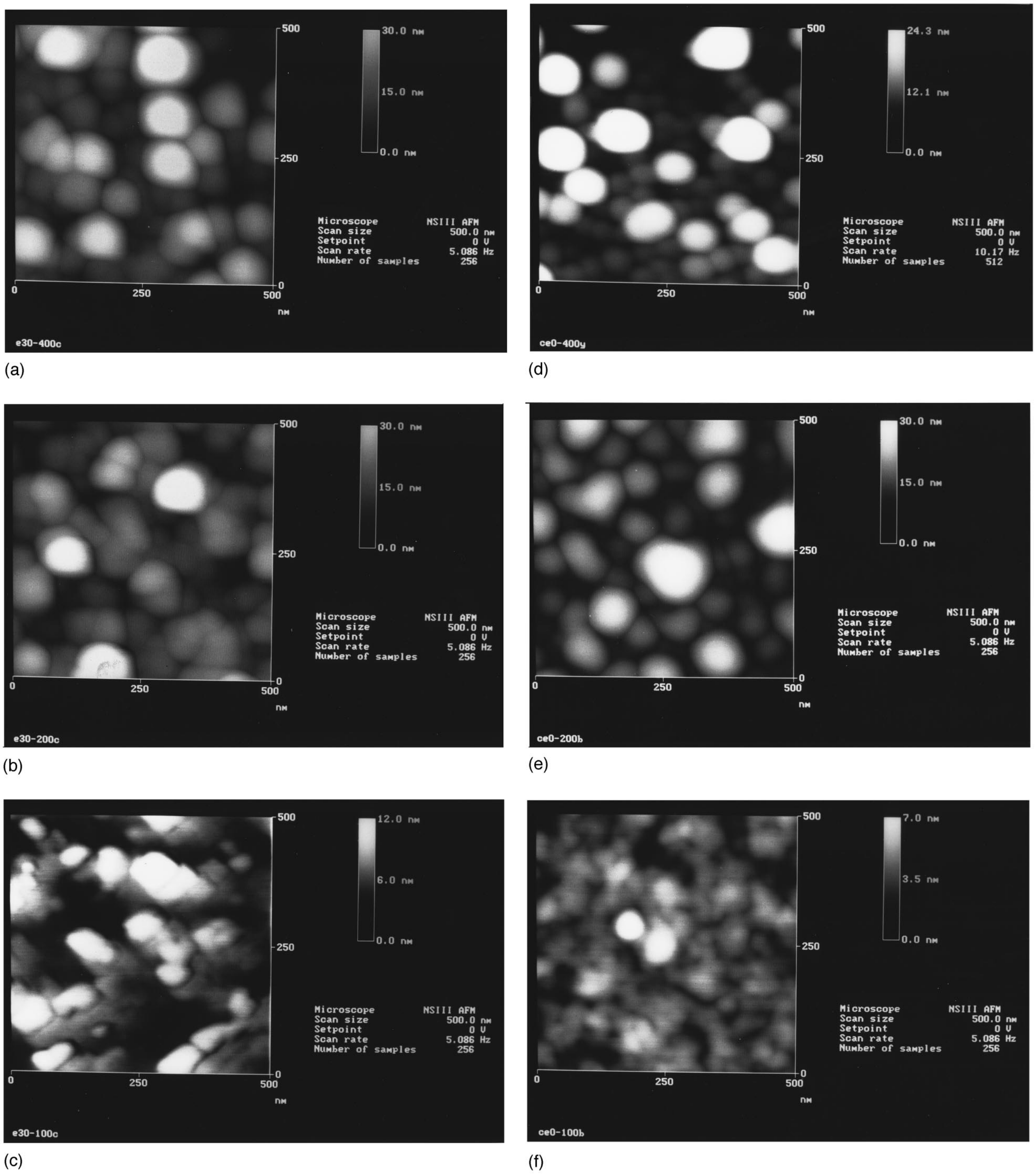

FIG. 2. AFM photograph of the $La_{0.7}Ce_{0.3}MnO_3$ films grown at (a) 400 mTorr, (b) 200 mTorr, (c) 100 mTorr. AFM photograph of the $La_{0.7}Ca_{0.3}MnO_3$ films grown at (d) 400 mTorr, (e) 200 mTorr, (f) 100 mTorr.

ion has a filled t_{2g} orbital with three electrons and one electron in the e_g orbital. The average occupancy of the e_g orbital (n) is therefore 1. The twofold degeneracy of the e_g orbital is lifted due to the Jahn–Teller distortion in $LaMnO_3$, which makes the material an insulator. In $La_{0.7}Ca_{0.3}MnO_3$, the parent compound is doped with holes and the average occupancy $n<1$. On the other hand, in $La_{0.7}Ce_{0.3}MnO_3$, the parent compound is doped with electrons and $1<n<2$. Since the oxygen content of the film depends on the ambient oxygen pressure during growth, we assume that the $La_{0.7}Ce_{0.3}MnO_3$ film has the composition $La_{0.7}Ce_{0.3}MnO_{3-\delta}$. (Not too much significance should be attached to the negative sign of δ since it is not known whether this material gets overoxygenated or under oxygenated. Here, δ is used to compare films with different oxygen stoichiometry.) For this composition, the average valence of Mn is 2.7-2δ. This means that when δ is increased the average manganese valence approaches 2. This in turn increases

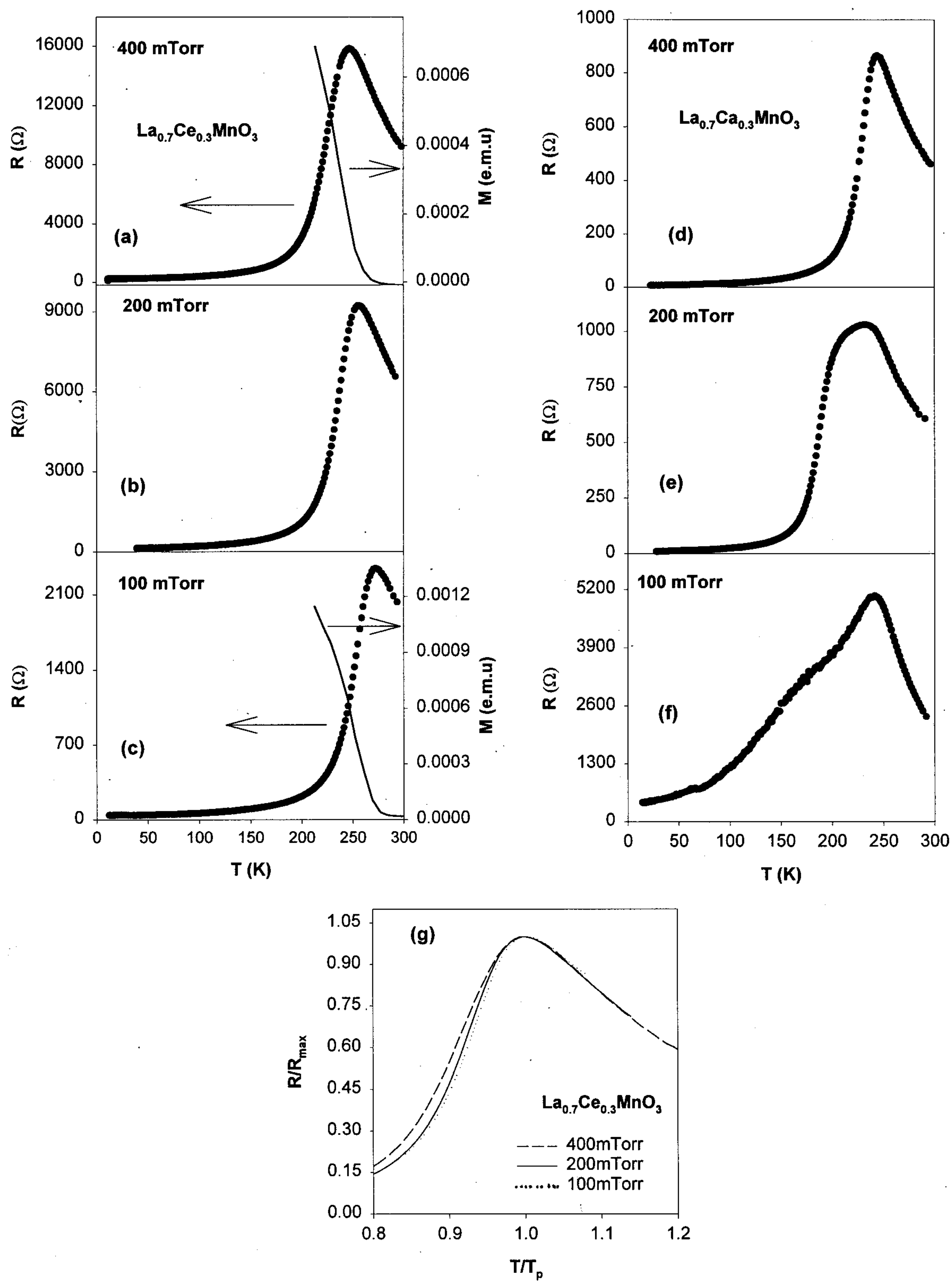

FIG. 3. Resistance vs temperature of $La_{0.7}Ce_{0.3}MnO_3$ grown at (a) 400 mTorr, (b) 200 mTorr, (c) 100 mTorr. Resistance vs temperature of $La_{0.7}Ca_{0.3}MnO_3$ grown at (d) 400 mTorr, (e) 200 mTorr, (f) 100 mTorr; (g) shows the R/R_{max} vs T/T_p for the three $La_{0.7}Ce_{0.3}MnO_3$ films, where R_{max} is the resistance value at T_p.

the electron doping at the e_g orbital. This is in contrast to the situation in the hole doped $La_{0.7}Ca_{0.3}MnO_{3-\delta}$, where the average Mn valence is 3.3-2δ. Here, increasing δ means that the Mn valence will reduce towards 3 thus decreasing hole doping. Thus, in terms of doping, the reducing oxygen pressure has opposite effects in the two compounds. Since T_p and T_c are known to be sensitive functions of doping in these materials, we can qualitatively explain the increase in T_p in one case and decrease in the other. The other difference between $La_{0.7}Ca_{0.3}MnO_3$ and $La_{0.7}Ce_{0.3}MnO_3$ films is that the width of the metal-insulator transition is more insensitive to the decrease in the oxygen pressure in $La_{0.7}Ce_{0.3}MnO_3$. For

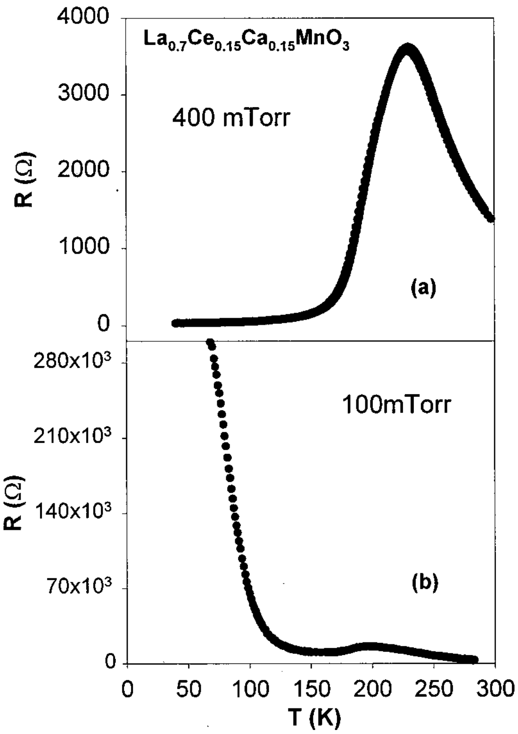

FIG. 4. Resistance vs temperature for $La_{0.7}Ca_{0.3}Ce_{0.15}MnO_3$ films grown at (a) 400 mTorr and (b) 100 mTorr oxygen pressure.

the $La_{0.7}Ca_{0.3}MnO_3$ film grown at 100 mTorr, the metal-insulator transition is extremely wide [Fig. 3(f)]. On the other hand, the width of the transition remains almost constant (with a slight decrease at lower oxygen pressures) with decrease in oxygen pressure for $La_{0.7}Ce_{0.3}MnO_3$ films [Fig. 3(g)]. The large broadening in the metal-insulator transition in $La_{0.7}Ca_{0.3}MnO_3$ suggests that the $La_{0.7}Ca_{0.3}MnO_3$ films become inhomogeneously oxidized at lower oxygen pressures. We believe that the $La_{0.7}Ce_{0.3}MnO_3$ film is more homogeneous in terms of its oxygen stoichiometry due to the high oxygen affinity of cerium. This robustness of the metal-insulator transition might be advantageous in applications where a sharp metal-insulator transition and a large temperature coefficient of resistivity are desirable; for example, in bolometric application.[13,14]

B. The effect of epitaxial strain on the metal-insulator transition (T_p) and magnetoresistance

In order to study the effect of epitaxial strain on the transport properties of this material, we synthesized films of varying thickness in the range of 500–3200 Å. For the thinnest film, the thickness was estimated from the deposition time. The substrate temperature was kept at 760 °C and the ambient oxygen pressure was kept at 400 mTorr during growth. These values were chosen since grain size and surface morphology were best for these parameters. Figures 5(a)–5(c) show the surface morphology for films of various thicknesses. Thicker films have smaller grain size possibly due to strain relaxation. In Fig. 6(a) we show the $R-T$ curves close to the metal-insulator transition. There is a gradual increase in T_p with decreasing film thickness. However, we see that there is not much change in the width of the metal-insulator transition in this thickness range. This is shown in Fig. 6(b) where R/R_{max} is plotted as a function of T/T_p. Figure 6(c) shows the magnetoresistance $\{MR \sim \Delta\rho/\rho_0 \sim [\rho(H)-\rho(0)]/\rho(0)\}$ at 300 K for the four samples. There is a gradual increase in the magnetoresistance with decreasing film thickness.

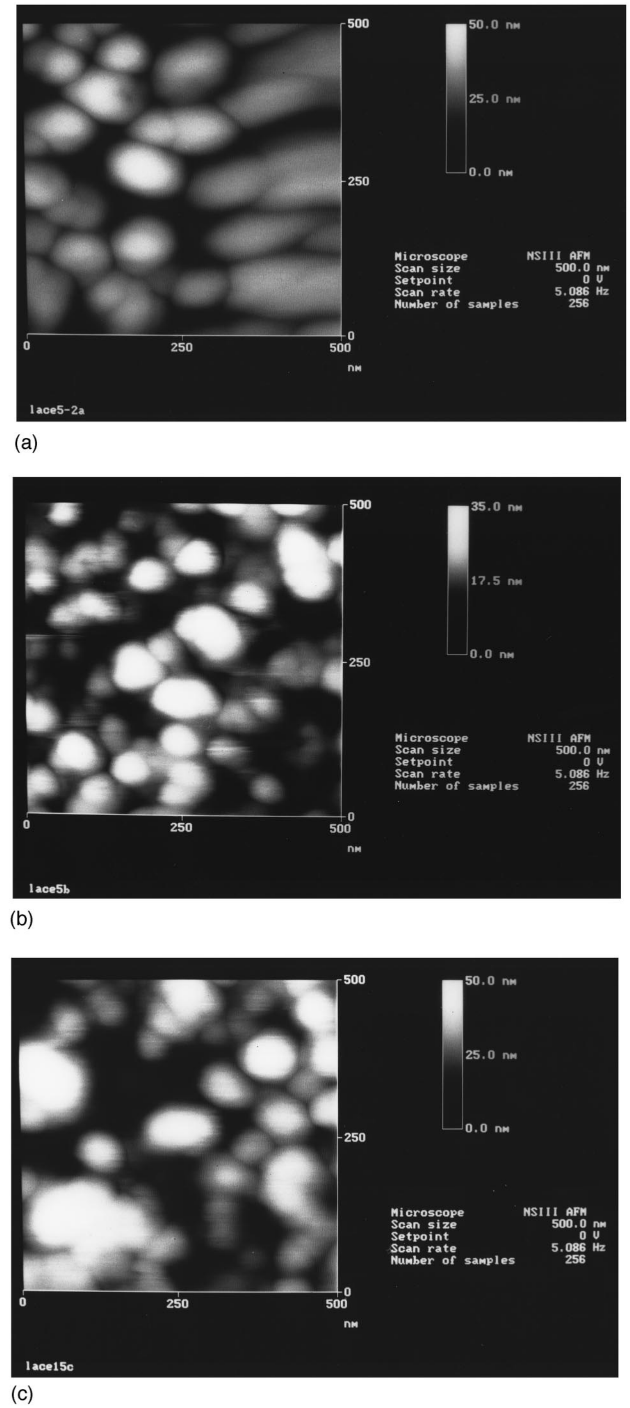

FIG. 5. Surface morphology of $La_{0.7}Ce_{0.3}MnO_3$ films of various thickness: (a) 500 Å, (b) 1000 Å, (c) 3200 Å.

In order to understand this observation, we measured the lattice constants of the four films using x-ray diffraction on a four-circle high-resolution goniometer. The lattice constants

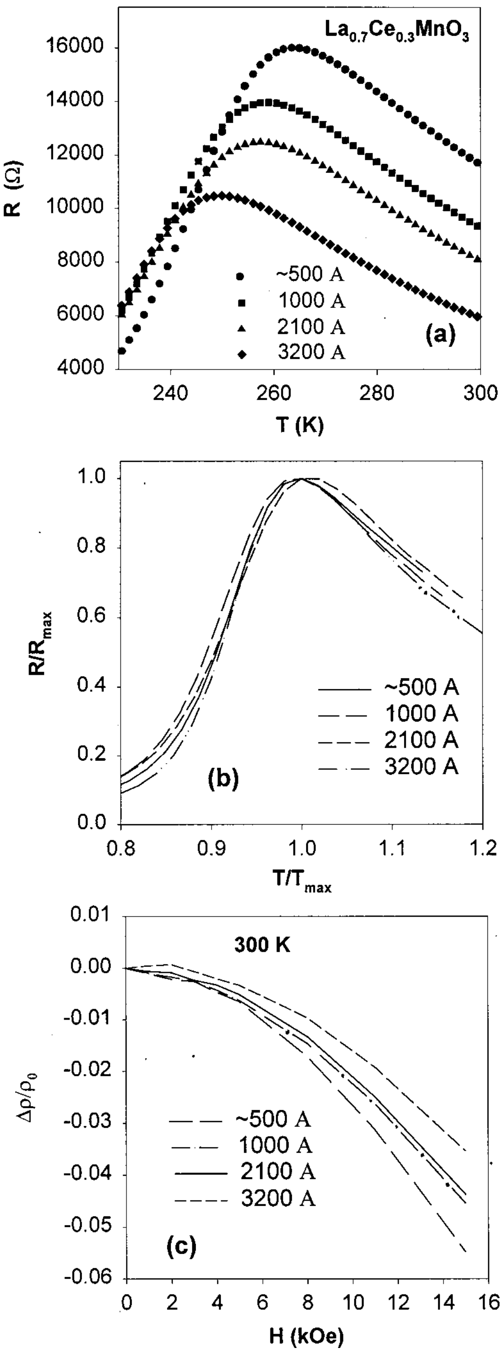

FIG. 6. (a) Resistance vs temperature around T_p for films of various thickness; (b) R/R_{max} vs T/T_p for films of various thickness; (c) MR vs field at 300 K for films of various thickness.

along with the cell volume and T_p for films of various thickness are summarized in Table I. There is a gradual compression in the in-plane (*a-b*) lattice constants and an expansion in the *c* parameter with decreasing thickness. This is understandable since the pseudocubic lattice parameter of $LaAlO_3$ is smaller than the pseudocubic lattice parameters of $La_{0.7}Ce_{0.3}MnO_3$. To understand the increase in T_p with decrease in thickness, we note that T_p in the bulk compound increases with applied pressure.[15] We know that T_p (and T_c) in doped manganites is governed by by the bandwidth (W) of the e_g band which is given by the semiempirical formula[16]

TABLE I. Evolution of lattice parameters, cell volume and T_p with thickness variation in $La_{0.7}Ce_{0.3}MnO_3$ films.

Thickness[a] (Å)	*a* (Å)	*b* (Å)	*c* (Å)	*V* (Å^3)	T_p (K)
500	5.518	5.459	7.852	236.52	263
1000±100	5.529	5.466	7.841	236.09	259
2100±100	5.548	5.464	7.836	237.54	257
3200±100	5.561	5.512	7.802	239.15	250

[a]All thickness values except the lowest one were measured on a Dektak profilometer. The lowest thickness was estimated from the time of deposition using the calibration from the thicker ones.

$$W \propto \frac{\cos\omega}{d_{\text{Mn–O}}^{3.5}},$$

where ω is the ''tilt'' angle in the plane of the Mn–O–Mn bond (given by $\omega=\frac{1}{2}[\pi-\langle\text{Mn–O–Mn}\rangle]$), and $d_{\text{Mn–O}}$ is the Mn–O bond length. Application of an isotropic pressure in bulk samples reduces $d_{\text{Mn–O}}$ but keeps ω almost constant, thus increasing W and T_c. On the other hand, when a chemical pressure is applied by modifying the size of the cation at the rare-earth site, $\cos\omega$ decreases more rapidly than $d_{\text{Mn–O}}$ thus reducing W and T_c. We believe that the effect of compressive epitaxial stress in films is similar to the applied pressure in the bulk sample. However, the situation is more complicated here due to the anisotropic nature of the strain in the film which give rise to different values of $d_{\text{Mn–O}}$ and ω in different directions. Rao *et al.*[17] have reported the effect of epitaxial strain in $La_{0.8}Ca_{0.2}MnO_3$ films of varying thickness. Their study revealed that T_p decreases with compressive in-plane epitaxial strain. Blamire *et al.*[18] have reported similar results on laser ablated $La_{0.7}Ca_{0.3}MnO_3$ films on $LaAlO_3$ and $SrTiO_3$. The puzzling fact is that in bulk $La_{0.7}Ca_{0.3}MnO_3$ also T_p increases with applied pressure.[16] However, a close inspection of their $R-T$ data (Ref. 18) for various film thicknesses reveal that the metal-insulator transition becomes extremely broad in addition to the decrease in T_p with decreasing film thickness. One reason for this behavior could be that very thin films of $La_{0.7}Ca_{0.3}MnO_3$ tend to lose oxygen, which leads to a reduction in its T_p.[17]

Figures 7(a)–7(d) show the $R-T$ curves close to the metal-insulator transition for the films with varying thickness at different fields up to 1.5 T. All the films have a large MR ($>60\%$) close to the metal-insulator transition at 1.5 T [Fig. 7(e)]. Note that above 250 K, the thinner films have larger MR. This could be useful since a large MR at temperatures close to room temperature is required for the device application of these films.

IV. CONCLUSION

We have reported the magnetic and transport properties of laser ablated $La_{0.7}Ce_{0.3}MnO_3$ films on $LaAlO_3$ substrates. By codoping with Ca (in $La_{0.7}Ca_{0.15}Ce_{0.15}MnO_3$) we have established that in this system the parent compound ($LaMnO_3$) is doped with electrons in contrast to the hole doped compounds such as $La_{0.7}Ca_{0.3}MnO_3$, where holes are doped by substitution of bivalent Ca^{2+} at the rare-earth site. This calls for further investigations of the electronic band

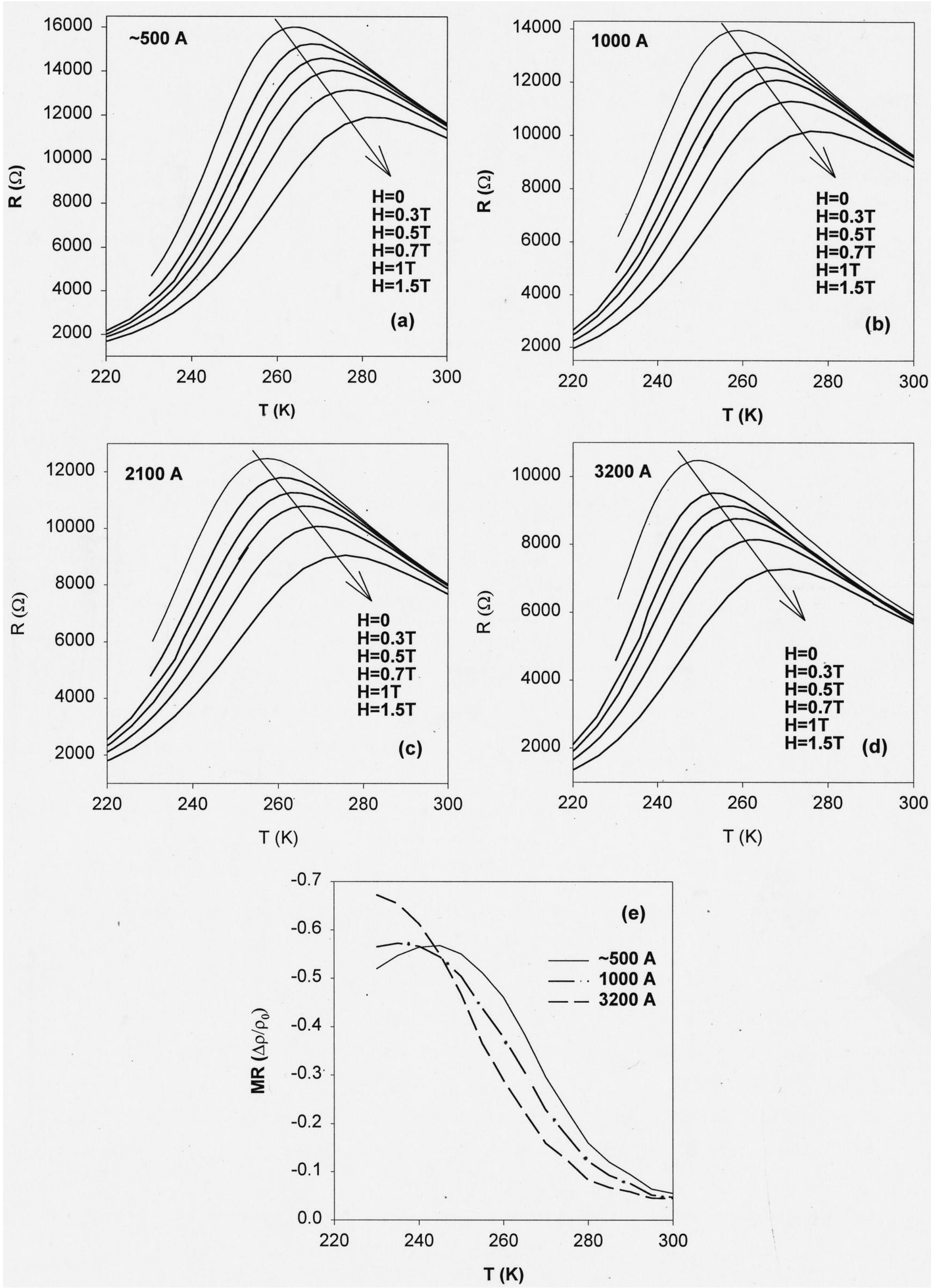

FIG. 7. (a)–(d) Resistance vs temperature at different fields for various film thickness. (e) MR at 1.5 T as a function of temperature for films of various thickness.

structure in this compound since both Jahn–Teller distortion (splitting the degeneracy of the e_g band) and strong Hund's rule (polarizing the conduction band) play a vital role in the electronic properties of this compound. We have compared the effect of ambient oxygen pressure during growth in $La_{0.7}Ca_{0.3}MnO_3$ and $La_{0.7}Ce_{0.3}MnO_3$. We find that the latter is much more stable with respect to variation in oxygen pressure. Thickness dependence studies reveal that the metal-insulator transition temperature increases with decreasing thickness. This can be attributed to the compressive in-plane epitaxial strain in the films. However, the most interesting observation in this study is that the properties of $La_{0.7}Ce_{0.3}MnO_3$ provide us a scheme to tune the metal-insulator transition temperature over a moderate range by varying oxygen pressure during growth and sample thickness, without compromising on the sharpness of the metal-insulator transition.

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