MAGNETISM AND MAGNETOTRANSPORT IN HALF- AND OVER-DOPED MANGANITES: IMPACT OF SUBSTRATE INDUCED STRAIN AND POLYCRYSTALLINE DISORDER

Synopsis of the thesis submitted in fulfillment of the requirement for the Degree of

DOCTOR OF PHILOSOPHY

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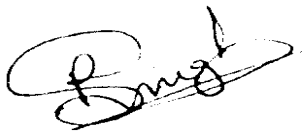
CERTIFICATE

This is to certify that subject matter presented in this synopsis entitled, **"Magnetism and magnetotransport in half- and over-doped manganites: Impact of substrate induced strain and polycrystalline disorder"**, submitted by **Pawan Kumar** at **Jaypee Institute of Information Technology (Deemed University), NOIDA, Uttar Pradesh, India,** is the original contribution of the candidate. This work has not been submitted elsewhere for any other degree or diploma.



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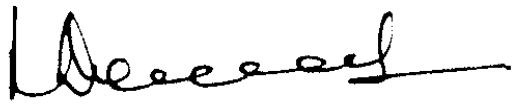

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SYNOPSIS

Over the last two decades, the doped rare earth perovskite manganites that are chemically represented by $RE_{1-x}AE_xMnO_3$ (where, RE is rare earth La, Nd, Pr, etc. and AE is the alkaline earth elements Ca, Ba, Sr etc.) have been the centre of interest for the materials science community [1-6]. The interest in these systems arises not only from the prospect of their application in magnetic data storage/recording, memories, magnetic/temperature sensors etc. but also due to the occurrence of variety of interesting physical phenomena, e.g., paramagnetism (PM), ferromagnetism (FM), antiferromagnetism (AFM), charge and orbital ordering (CO/OO), insulator metal transition (IMT), etc. that causes strong phase separation (PS). The competing lattice, charge and spin degrees of freedom make the phase diagram of these compounds very rich. The huge drop in the resistivity under the influence of a magnetic field in manganites yields a very large magnetoresistance (MR), much larger than that observed in giant magnetoresistance (GMR) materials [7-11]. Hence it was termed as colossal magnetoresistance (CMR) [9].

The real essence of large MR due to PS in manganites [12-14] is explained in terms of the relative increase in metallic ferromagnetic (FM) phases with respect to insulator charge ordered-antiferromagnetic (CO-AFM) phase due to the application of a magnetic field. At half and over-doping ($0.50 \leq x \leq 1$) in intermediate (i.e. $Nd_{1-x}Sr_xMnO_3$, $La_{1-x}Ca_xMnO_3$ etc.) and narrow bandwidth (i.e. $Pr_{1-x}Ca_xMnO_3$, $Sm_{1-x}Sr_xMnO_3$ etc.) manganites, a variety of magnetic and electronic phases such as paramagnetic insulator (PMI), ferromagnetic metal (FMM), antiferromagnetic insulator (AFMI) and charge ordered (CO)/orbital ordered (OO) are observed on different temperature scales [6,15-17]. The occurrence of such phases has been attributed to the strong coupling between spin, charge, lattice and orbital degrees of freedom [2,4,11,16,17]. Due to the narrow stability range of these phases, strong phase separation is often seen as intrinsic and consequently the CO phases can be transformed into FM by external perturbations such as magnetic and electric field [18]. Further strong inter diffusion across the phase boundaries is also seen [18-22].

It has been shown that, the AFM charge order insulating (AFM-COI) phase and the concomitant metal–insulator (MI) transition at T_{CO}, that appear in bulk samples are modified in

thin films, indicating the important role of strain induced by film/substrate lattice mismatch [10,11,23,24]. It is argued that, the strain prevents the occurrence of the lattice distortions necessary to accommodate and drive the CO state [24]. In chapter 3, we have shown that for near half doping in $Nd_{1-x}Sr_xMnO_3$ the strong phase coexistence/phase separation is observed in polycrystalline thin films.

Apart from CMR, another important property of doped rare earth manganites is anisotropic magnetoresistance (AMR) due to anisotropic magnetocrystalline nature that results in the dependence of resistivity on the angle between the applied magnetic field (H) and the direction of transport current (J) [25–33]. The AMR of manganites thin film can be utilized for storing memory in two different directions (in-plane and out-of-plane) within the same used area. Nonspherical charge distribution possesses an orientation dependent energy due to its interaction with the surrounding lattice electric field, and through the spin–orbit interaction this leads to an angular dependent contribution from the total moment to the overall energy of the system, resulting in magnetocrystalline anisotropy [25].

Thin films, by virtue of the substrate induced strain and strain relaxation offer several additional degrees of freedom to modify and tune the physical properties of these materials. The effect of biaxial distortion induced by underlying substrate strain is expected to be fundamentally different from the effect of bulk (compressive) strain. This biaxial strain strongly affects the subtle interplay between spin, charge, structural, and orbital degrees of freedom. Therefore, the clarification of the detailed role of biaxial strain is essential. Extensive details are given in some recent reviews [10, 11].

In manganite thin films the non-spherical charge distribution around Mn ions gets further modified by substrate induced strain and lattice defects [10,11]. A reduction in dimensionality (e.g., in thin films) of such a system enhances the easy axis magnetic anisotropy and a decrease in electrostatic screening, which favors directional tunneling of electron through the insulating stripe domain walls formed at the phase-separated FM domain boundaries [31]. In manganites both strain and magnetocrystalline anisotropy are reasoned for the occurrence of AMR near the insulator–metal transition temperature (T_{IM}) but detailed explanation for this is still debatable [26]. Apart from the strain and hence the film thickness other factors such as the structural

defects, spin disorder, nature of the magnetic ground state, phase coexistence, etc. are also expected to play a crucial role. So far the low field anisotropic magnetoresistance (LF-AMR) has not been explored and investigated in manganites with mixed ground state having strong phase coexistence. In the chapter 4 of proposed thesis, we have studied the impact of compressive strain and polcrystallinity on the out-of-plane AMR in nearly half doped manganite $Nd_{0.51}Sr_{0.49}MnO_3$ (NSMO) thin films that show strong phase coexistence.

In nano-manganites, when the size is reduced below 100 nm, the charge and the orbitally ordered (CO/OO) ground state with antiferromagnetic (AFM) spin order have been shown to become unstable, and this gives rise to a ferromagnetic (FM) ground state [4, 34]. Size-induced transition from the AFM/CO state to the weak ferromagnetic (WFM) state was observed in both nanowires [34] and nanoparticles [35,36]. Lu et al. [4] showed that destabilization of the AFM charge ordered (AFM-CO) state and the formation of a FM order could result in an enhancement of the magnetization by two orders of magnitude. The WFM ground state resulting in nanomanganites from the destabilization the AFM ground state has been regarded to be direct consequence of size reduction because when the size is small enough (e.g., 20 nm), the effect of surface spin disordering becomes more evident. However, a WFM induced by the destabilization of the AFM order has also been reported in single crystals [2,11], as well as epitaxial thin films [16]. This suggests that the evolution of the WFM state out of the AFM-CO state cannot be attributed to downsizing to nanometric scale alone and that some additional effects, such as orbital disordering, may also be equally important [11,16-18]. Material downsizing can have a more dramatic effect in the vicinity of bicritical regions in manganites. Among the manganites, $Nd_{1-x}Sr_xMnO_3$ (NSMO) possesses several bicritical regions [11]. In the case of NSMO, the AFM phase spans the whole half- and over-doped region ($0.50 \leq x \leq 1$) wherein the A-type AFM (antiferromagnetically coupled two- dimensional ferromagnetic order) occurs up to $x \sim 0.63$. Above that, C-type AFM (antiferromagnetically coupled one-dimensional ferromagnetic order) takes over. The phase boundary at $x \sim 0.63$ that separates the A- and the C-type AFM phases has been shown to possess a fairly large FM contribution [2,11,16]. In single crystals and epitaxial thin films [2,11,16], this FM and the associated transition have been attributed to the strong orbital fluctuation/disorder in the proximity of the A-C phase boundary. Nanocrystalline thin

films, by virtue of their large surface-to-volume ratios and local epitaxy, can provide an additional degree of freedom to tune and to complement the WFM at the A-type and C-type AFM (A-C AFM) phase boundary in NSMO. However, nanocrystalline thin films of this material have not been investigated so far. In the chapter 5 of proposed thesis, we have shown that the A-type AFM (A-AFM) phase in nanocrystalline thin films (having crystallite size < 20 nm) of over-doped $Nd_{1-x}Sr_xMnO_3$ ($x \sim 0.60 - 0.62$) can be destabilized, resulting in the occurrence of a FM phase. In addition to the small magnetic moment, small remanence, large coercivity, and the exchange bias (EB) effect suggests the presence of a FM phase.

The objective of the present work is to study the impact of substrate induced strain and polycrystalline disorder on magnetism and magnetotransport property in half and over doped manganites thin films. The thickness of these films is generally in the range of 10-300 nm. The compounds and compositions were chosen judiciously to make the impact of strain or polycrystalline disorder visible. Here, we provide a brief sketch of the proposed doctoral thesis. The thesis is divided into six chapters and one appendix. The **chapter-I,** gives a brief and up-to-date introduction to the state of the art of manganite and their thin films. The **chapter-II,** describes briefly the experimental techniques employed during the present work. Single crystalline and some polycrystalline thin films have been prepared by DC magnetron sputtering technique, while other polycrystalline thin films have been deposited by nebulized chemical spray pyrolysis. Thickness analysis has been carried out by surface profiler and atomic force microscopy. Structure/microstructure and morphology of these films has been studied by X-ray diffraction/high resolution transmission electron microscopy (HRTEM) and atomic force microscopy (AFM), respectively. Magnetic properties were explored using DC magnetization employing a commercial magnetic properties measurement system (MPMS-Quantum Design). Resistivity and low field magneto-resistance measurements up to 0.5 T magnetic field was done using a homemade setup and high magnetic field (7 T) magnetotransport was carried out using commercial physical properties measurement system (PPMS-Quantum Design). **Chapter-III** incorporates the work on magnetotransport in $Nd_{1-x}Sr_xMnO_3$ thin films. **Chapter-IV** details the work on the AMR in single crystalline and polycrystalline thin films of $Nd_{1-x}Sr_xMnO_3$ system. **Chapter-V** contains the work on occurrence of weak FM in overdoped $Nd_{1-x}Sr_xMnO_3$

polycrystalline thin films. **Chapter-VI** incorporates the work on structure and magnetotransport in $La_{1-x}Ca_xMnO_3$ polycrystalline thin films, in the vicinity of half doping. The **appendix-A** contains a brief report on the thickness dependent properties of $Nd_{0.50}Sr_{0.50}MnO_3$ single crystalline thin films. Summary of all the chapters is given below.

Chapter-I describes, the fundamental structural and electronic properties of the doped rare earth manganites. The occurrence of the various phenomena such as PMI, FMM, FM insulating (FMI), AFM insulating (AFMI), AFM metallic (AFMM), CO/OO and IMT and their origin has been elaborated and discussed in the framework of the divalent doping as well temperature scale. The fundamental interactions that are believed to govern the above phenomena such as the Jahn Teller (J-T) effect, FM-double exchange (FM-DE), AFM-superexchange (AF-SE), etc. have also been described. The phenomenon of phase coexistence /phase separation, which is now believed to be intrinsic to these materials and one of the key features to explain the range of effects observed, is also discussed. In addition, the state of the art of bulk as well as thin film manganites has been discussed in this chapter.

In **Chapter-II,** the fundamental aspect of thin films and the growth models have been described. The effect of substrate induced strain and its consequences have been elaborated. Thin film deposition techniques have been briefly described and the technique of DC magnetron sputtering, which has been used to grow single crystalline and some polycrystalline thin films in the present study is described in detail. Nebulized chemical spray pyrolysis that is used to prepare polycrystalline thin films has been also briefed. Characterization techniques, such as, X-ray diffraction, transmission and scanning electron microscopy techniques, atomic force microscopy, etc., magnetization and magnetotransport measurement techniques have been also described.

In **Chapter-III,** we have studied the impact of magnetic phase coexistence/phase separation on the magnetic and electrical transport properties of $Nd_{1-x}Sr_xMnO_3$ (x = 0.49) polycrystalline thin films of thickness ~ 100 nm, prepared by dc magnetron sputtering on (100) oriented single crystal Y-stabilized ZrO_2 substrates. It has been shown that near half doping the strong phase coexistence/phase separation is observed in polycrystalline thin films. The present films show the paramagnetic (PM) to ferromagnetic (FM) transition at Curie temperature (T_C) ~ 260 K, which is slightly larger than that reported for single crystals/epitaxial thin films of similar composition

(T_C~ 250 K) and is followed by A-type antiferromagnetic (AFM) ordering at temperature (T_N^A) ~ 200 K and charge ordered (CO) phase having the CE type spin order at Neel temperature (T_N^{CO}) ~ 110 K. As reported, the strong CO-AFM transition observed at T_{CO} ~ 150 K at half doping is softened by a small decrease in the Sr concentration and the disorder due to the polycrystalline nature of these films. The multiple magnetic phase transitions are suggestive of strong phase coexistence. In this study the insulator-like to metal-like transition temperature T_{IM} ~ 140 K is considerably lower than the T_C and remains independent of the external magnetic field up to 70 kOe. The occurrence of competing magnetic phases (phase separation) leads to percolative electrical transport in polycrystalline $Nd_{0.51}Sr_{0.49}MnO_3$ films. This suggests that electrical transport is dominated by grain boundaries and has percolation type characteristics due to the existence of metallic (FM + A-AFM) and insulating (CO-AFM + Grain boundary (GB)) paths. The metallic clusters are from the FM and A-AFM phases while the insulating contribution arises mainly from the grain boundaries and the CE- type CO-AFM state. The electrical transport is strongly influenced by the presence of grain boundaries as evidenced by the occurrence of a magnetic field independent $T_{IM} << T_C$. As a consequence of the GB disorder and electronic phase separation a significant low field MR (~31% at H = 4 kOe and 58 K) as well as high field CMR (~80% at H = 70 kOe) can be achieved in the polycrystalline thin films of the same material.

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Chapter-IV contains a comparative study of magnetism and magnetotransport properties of single crystalline and polycrystalline thin films of phase separated nearly half-doped manganite $Nd_{0.51}Sr_{0.49}MnO_3$. We have studied the in-plane and out-of-plane magnetization (M), low field magnetoresistance (LFMR) and anisotropic magnetoresistance (AMR) in single crystalline and polycrystalline $Nd_{0.51}Sr_{0.49}MnO_3$ thin films. On-axis DC magnetron sputtering was used to deposit the single crystalline films (~30 and ~100 nm in thickness) on (001) oriented single crystal $LaAlO_3$ (LAO) and polycrystalline films (100 nm) on (100) oriented Yttria-stabilized ZrO_2 (YSZ) substrates. In all the films the magnetic easy axis lies in the plane of the film. The in plane and out-of-plane magnetotransport properties of these films differ significantly. All films show large LFMR when H$\|$I but the same is drastically reduced in the H$\perp$I configuration. A large low field AMR is observed in all the films. The large LF-AMR (13–15% at 3.4 kOe) in both

varieties of films suggests that the shape anisotropy dominates over the surface and magnetocrystalline anisotropies. AMR shows a peak below the insulator metal transition temperature in the single crystalline films, while the same increases monotonically in the polycrystalline film. The LF-AMR of the single crystalline films show a weak dependence of the films on film thickness (and hence the degree of strain) and could be related to the thickness dependent variation in the domain wall thickness. Relatively larger low field AMR (~20% at T = 78 K and H = 1.7 kOe) in the polycrystalline films suggests the dominance of the shape anisotropy. The magnetic field dependence of AMR shows a peak. Large AMR observed in the present thin films could be attributed to the occurrence of multiple magnetic phases that causes strong phase fluctuations.

(Results of this work published in Journal of Magnetism and Magnetic Materials, vol. 323, pp. 2564–2568, 2011)

In **Chapter-V** half- and over-doped $Nd_{1-x}Sr_xMnO_3$ (x ~ 0.50 –0.62) polycrystalline thin films having an average crystallite size of ~15 nm have been studied in relation to their magnetic and magnetotransport properties. This chapter demonstrates that an anomalous weak ferromagnetism occurs in the composition range $0.50 \leq x \leq 0.62$ in nanostructured thin films of $Nd_{1-x}Sr_xMnO_3$ (NSMO) manganite. Nanocrystalline thin films of half- and over-doped manganite $Nd_{1-x}Sr_xMnO_3$ (x ~ 0.50, 0.55, 0.60 and 0.62) are grown on single-crystal $LaAlO_3$ (001) substrates by using a nebulized chemical spray pyrolysis technique. These single phase films possess an average crystallite size ~15 nm, and the width of the grain boundaries is ~1-2 nm. In the composition range $0.50 \leq x \leq 0.62$, the ground state of NSMO is an A-type antiferromagnetic (AFM) metallic state. However, in the present films, a broad paramagnetic (PM)-to-ferromagnetic (FM) transition is observed in all the compositions, at T_C ~ 226 K for x = 0.50 and at 235 K for rest of the samples. All the films show a well-defined M-H hysteresis loop in the lower temperature regime. The coercivity (H_C) of these films is found to be much larger than those having compositions in the range $0.35 < x < 0.45$. Further, the asymmetry in the coercivity suggests the presence of a weak exchange bias effect in these films. The FM ground state in these films is observed to have a smaller magnetic moment per Mn atom than the expected full moment from the rigid model; we term this as a weak ferromagnetic (WFM) state arising due to the destabilization of the AFM ordering. We propose a possible scenario based on the combined effect of spin reorganization due

to reduced crystallite size and enhanced orbital disordering in nanosized manganites to explain the observed anomalous weak ferromagnetism in the A-type AFM spin ordered region.

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Chapter-VI presents the study on the coexistence of magneto-electronic phases and hence phase separations in $La_{1-x}Ca_xMnO_3$ thin films in the composition range $0.45 \leq x \leq 0.60$. We have studied the structural, microstructural, magnetic and magnetotransport properties of highly oriented $La_{1-x}Ca_xMnO_3$ ($0.45 \leq x \leq 0.60$) polycrystalline thin films. The oriented polycrystalline thin films were prepared by chemical nebulized spray pyrolysis technique on (001) oriented single crystalline substrate $LaAlO_3$ (LAO). We have shown that, like the nanostructured manganites, the AFM-COI state can be destroyed even in oriented polycrystalline thin films. The lattice parameters have been found to decrease with increase in Ca content and the structural/microstructural disorder is also seen to increase concomitantly. High resolution transmission electron microscopy (HRTEM) investigations show that there is an increase in the microstructural disorder with increase in Ca content. As the Ca content is increased from $x = 0.45$ to 0.55, the paramagnetic insulator (PMI) to ferromagnetic metal (FMM) transition temperatures (T_C/T_{IM}) are suppressed. This is accompanied by (i) appreciable suppression of the T_C, (ii) sharp reduction in the magnetic moment, (iii) occurrence of a peak in the magnetization below T_C, (iv) stronger bifurcation of the zero field cooled-field cooled (ZFC-FC) magnetization, and (v) decline in the magnetization in the lower temperature regime. The broadening of the PMI-FMM transition and simultaneous decrease in the magnetic moment shows that the observed variations are due to the increase (decrease) in the AFM-COI (FMM) phase fraction at higher Ca content. This also confirmed by the decrease in magnitude of the MR and the accompanying broadening of its temperature dependence. At $x = 0.60$, the FM metallic phase disappears and a weak FM insulator like phase is observed, which is attributed to the ordering of the FM clusters and disordering of the AFM state due to the occurrence of structural defects and surface effects. This is also confirmed by the absence of any peak in the temperature dependent MR of this film. In the vicinity of $x = 0.55$, the magnetic ground state resembles metamagnetic spin cluster glass. This is also confirmed by the occurrence of a hysteresis loop in the resistivity of the $x = 0.55$ measured during the cooling and warming cycle. The transition broadening up to $x = 0.55$ and the vanishing

of the two transitions in x = 0.60 film along with the simultaneous decrease in the magnetic moment and enhanced resistivity shows that the observed variations are due to the increase in the antiferromagnetic-charge ordered (AFM-CO) phase fraction at higher Ca content. This also confirmed by the decrease in magnitude of the magnetoresistance and its temperature dependence. Our results demonstrate that, there is strong phase separation in the overdoped region ($0.50 < x \leq 0.60$) due to the presence of multiple magneto-electric phases. Significant deviation from the bulk properties such as the charge order destabilization and hysteretic insulator to metal transition (IMT) in the vicinity of half doping and weak ferromagnetism in the overdoped regime is caused by (i) substrate induced compressive strain, (ii) oxygen vacancies due to high temperature annealing, and (iii) enhanced microstructural disorder (as evidenced by our HRTEM results).

(Results of this work published in Journal of Alloys and Compounds, vol. 531, pp. 23-29 2012)

Appendix-A presents the structural, morphological, magnetic and magnetotransport characteristics of $Nd_{0.50}Sr_{0.50}MnO_3$ thin films deposited on (001) oriented $LaAlO_3$ (LAO) single crystal substrates. All the films are found to be single crystalline. At lower thicknesses the films are strongly strained and as the film thickness increases the strain is observed to relax gradually. The out of plane lattice parameter decreases nonlinearly from 0.38979 nm to 0.38358 nm as the film thickness increases from 10 nm to 300 nm. The PM-FM as well as the CO transition temperature is seen to increase with film thickness. The T_C increases from 207 K to 250 K and T_{CO} increases from 132 K to 200 K as the film thickness increases from 10 nm to 300 nm. However, only the 300 nm film shows metallic transition. The magnetic moment is also observed to increase gradually with film thickness, hence suggesting the enhancement in the FM fraction. The enhanced magnetic moment and IMT at higher film thickness has been explained in terms of the defects that could be caused by strain relaxation.

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1. Kumar P., Prasad R., Srivastava A. K., Vijayan N., Dwivedi R. K., Singh H. K., *"Microstructural and magnetotransport properties of $La_{1-x}Ca_xMnO_3$ ($0.45 \leq x \leq 0.60$) thin films"*, J. Alloys Compd., vol. 531, pp. 23-29, March 2012.
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