Cluster-glass state and photon-induced effects in perovskite manganite ($\text{La}_{0.3}\text{Nd}_{0.7}$) $_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ films

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The electrical and magnetic properties and photon-induced effects in perovskite manganite thin films of ($\text{La}_{0.3}\text{Nd}_{0.7}$) $_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ with the tolerance factor $t \approx 0.908$ are investigated. The persistent photoconductivity (PPC) effect induced by a He-Ne laser with a photon energy of ~ 2 eV and the normal photoconductivity (NPC) effect induced by a mercury lamp with photon energy near 3 eV are observed at low temperatures where the system is in (or close to) the cluster-glass (CG) state. The observed resistivity relaxations induced by laser illumination and magnetic field indicate that the CG state is unstable. The PPC, NPC, and magnetic effects are originated from different mechanisms and can be understood based on the particular CG-state-related phase separations.

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I. INTRODUCTION

Since the discovery of the colossal magnetoresistance (CMR) effect in doped perovskite manganites $R_{1-x}A_x\text{MnO}_3$ (where R is trivalent rare-earth ions and A is divalent alkaline-earth ions), their electronic states and transport properties have been extensively studied to elucidate the unusual transport behaviors.¹ The rich structural, electronic, and magnetic phase diagrams are related to intricate interplay between spin, charge, orbital, and lattice degrees of freedom, which offer typical research systems the ability to study fundamental problems in condensed-matter physics. Furthermore, the peculiar properties existing in the doped manganites, such as the CMR effect, have a potential application in sensor and storage devices.

Recently, the discovery of intrinsic phase separation, especially the coexistence in the neighborhood of the ferromagnetic (FM) metallic phase and nonferromagnetic insulator phase, in the perovskite manganites has become an important issue.¹⁻⁵ Percolative conduction through metallic regions in the insulating matrix is believed to play an important role in electrical transport and to be the origin of the CMR effect.⁴ So far, the most theoretical and experimental studies are focused on the intrinsic charge inhomogeneity and phase separation related to the doping concentration.

Fewer studies are on phase separation related to lattice distortions. However, it is well known that the size mismatch caused by different ion radii between perovskite A and B sites can result in a considerable structure deformation that in turn affects transport properties.⁶⁻⁸ Teresa *et al.*⁷ and Terai *et al.*⁸ have systematically investigated (La-Tb) $_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ and (La-Dy) $_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ polycrystalline compounds, respectively, and obtained similar electronic and magnetic phase diagrams. Their research demonstrates that the systems are in a spin-glass insulator state at low temperatures below ~ 50 K when the tolerance factor t , a geometry parameter describing the mismatch of perovskite A- and B-site ions, is smaller than ~ 0.907 . This implies a possible phase separation. We, therefore, speculate that the phase in the vicinity of $t \sim 0.907$ should be unstable and sensitive to external stimulations such as temperature, magnetic field, electric field, pressure, and photon irradiation.

In our previous work,⁹ the interesting phenomenon of a persistent photoconductivity (PPC) effect induced by a He-Ne laser at low temperatures below ~ 50 K in thin films of ($\text{La}_{0.3}\text{Nd}_{0.7}$) $_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ (LNCMO) with the tolerance factor $t \sim 0.908$ was briefly reported. When the laser starts to illuminate on the film the resistivity decreases rapidly and significantly at the beginning, and the prolonged illumination eventually slows the rate of decreasing resistivity. However,

it is surprising to note that a further decrease in resistivity persists even after the laser is switched off and the measurement is stopped, which indicates that the PPC effect observed here is distinctly different from the PPC effect observed in conventional semiconductors in which the photoconductivity recovers though often very slowly when the light is switched off. The magnetization measurements indicate that the sample lies in a cluster-glass (CG) state in the temperature region below ~ 50 K. The PPC effect is believed to be closely related to the magnetic properties. In this paper, the relaxations of resistivity induced by laser illumination and magnetic field are investigated in detail. In addition, a normal photoconductivity (NPC) effect induced by a mercury lamp (Hg lamp) at low temperatures is observed. It has been found that the laser illumination, Hg-lamp illumination, and applied magnetic field affect the sample resistivity via different mechanisms. The results are explained based on the phase separation in the CG state.

II. EXPERIMENT

LNCMO films with thickness of 300 nm were epitaxially grown on SrTiO_3 (001) substrates by an off-axis dc magnetron sputtering technique reported previously.^{9,10} The substrates were held at 750°C in a mixture atmosphere of Ar/O_2 with the total pressure of 0.25 Torr during deposition. After deposition, the thin films were postannealed in a 760-Torr oxygen atmosphere at 600°C for 1 h. The x-ray-diffraction spectra showed that the films had no extra phases. The full width at half maximum value of $\sim 0.5^\circ$ obtained from a 2θ scan of the (002) reflection peak indicated an excellent structural order of the films.

A pair of silver electrodes separated by a $\sim 150\text{-}\mu\text{m}$ span were vacuum evaporated on the surface of the sample. The resistivity of the sample was measured in the temperature range from 20 to 300 K, with applied magnetic field up to 7 T. The Hg lamp with wavelengths of 546.1, 435.8, and 404.7 nm was also used to illuminate the samples in addition to the He-Ne laser used in our previous work.⁹

III. RESULTS AND DISCUSSIONS

Figure 1 shows the temperature dependence of the resistivity $\rho(T)$ of LNCMO thin film in warming and cooling modes in the dark and with the laser and Hg-lamp illumination in real time. The changing rate of the temperature was controlled at 1.5 K/min for both warming and cooling modes. The illumination-induced heating effect was taken into account.⁹ In the high-temperature range above ~ 130 K the $\rho(T)$ curves overlap and $\rho(T)$ increases monotonically as temperature decreases. A strong hysteresis effect corresponding to warming and cooling modes is observed below ~ 130 K for $\rho(T)$ in the dark. In the cooling mode $\rho(T)$ curves with laser and Hg-lamp illumination begin to separate from those in the dark at temperatures of ~ 50 and ~ 56 K, respectively. A semiconducting-metallic transition in $\rho(T)$ is observed at about 40 K with Hg-lamp illumination. While $\rho(T)$ curves in the dark and with laser illumination exhibit semiconductorlike behavior in the measured temperature

FIG. 1. Temperature dependence of the resistivity, $\rho(T)$, measured in warming and cooling modes in the dark and with laser and Hg-lamp illumination in real time. The power densities of the He-Ne laser and the Hg lamp are ~ 78 and ~ 10 mW/cm^2 , respectively.

range of 20–300 K, we note that the resistivity change [i.e., the deviation from the dark $\rho(T)$] induced by laser illumination at low temperatures cannot be recovered by switching off the laser. This is the PPC effect described in our previous work.⁹ However, it is worth emphasizing that the resistivity change induced by the Hg lamp at low temperatures can be recovered by switching off the Hg lamp. This is the NPC effect newly observed in the same sample. Further detailed investigations are presented below.

Figure 2 shows the relaxation of the resistivity of the sample after 15 min of laser illumination (filled circles) at 30 K. It can be seen that the resistivity fluctuates but slowly decreases during a period of about 3 h after the laser is switched off. When the laser is back on again the resistivity starts to decrease more rapidly and smoothly. The resistivity as a function of time measured before any illumination (open

FIG. 2. Time dependence of the resistivity measured after laser illumination (corresponding to the left scale) and before laser illumination (corresponding to the right scale).

FIG. 3. Time dependence of the resistivity with Hg-lamp illumination switched on (denoted as “Hg-on” with down arrows) and off (denoted as “Hg-off” with up arrows) at different temperatures indicated.

circles) indicates that it is stable without external stimulations.

Lights from the Hg lamp with different wavelengths obtained by optical filters are used to illuminate the sample. No photon-induced resistivity change is observed using the Hg-lamp emission at 546.1 nm, which may be caused by the weak power density of the Hg lamp and a low absorption of the sample at this position of wavelength. However, the photon-induced resistivity change is found using Hg-lamp emissions at 435.8 and 404.7 nm. Figure 3 illustrates the resistivity change induced by Hg-lamp illumination at the different temperatures indicated. The arrows pointing down and up in the figure indicate the switching on (“Hg-on”) and off (“Hg-off”) of the Hg-lamp, respectively. For temperatures above 60 K no photon-induced resistivity change can be observed. But below 60 K as the temperature decreases the photon-induced resistivity change becomes increasingly evident. Unlike the PPC effect observed with laser illumination (see Fig. 2), the photon-induced resistivity change observed here with the Hg lamp can more or less fully recover to its original value after switching off the Hg lamp. We call this photon-induced resistivity change an NPC effect.

Figure 4 shows the dependence of the sample resistivity on applied magnetic field, $\rho(H)$, at 30 K. A strong hysteresis effect is observed for the $\rho(H)$ curves corresponding to a sweeping up and down of the magnetic fields, respectively. The sweeping rate of the field is 0.8 T/min. In case of sweeping up, the resistivity changes little while the field is less than about 2 T, but decreases rapidly once the field is increased further. In the case of sweeping down, the resistivity increases relatively slowly and cannot recover to the original zero-field value. The detailed resistivity relaxations at fixed

FIG. 4. Magnetic-field dependence of the resistivity $\rho(H)$, at 30 K in sweeping-down and up modes. The inset shows the resistivity relaxation with time at 30 K at 7 and 0 T after a sweeping loop up to 7 T.

magnetic fields of 7 and 0 T (after a normal sweeping loop up to 7 and back to 0 T) is shown in the inset of Fig. 4. When the field is fixed at 7 T the resistivity continues to decrease as time goes on. Similarly, when the field is swept down to zero the resistivity continues to increase as time goes on. Our experiments have shown that similar resistivity relaxations can be observed clearly at temperatures below ~ 130 K and for the magnetic-field sweeping loop above ~ 2 T. When the applied magnetic field is below 2 T, the resistivity relaxation is not obvious, i.e., the resistivity decreases slightly at the beginning, quickly reaches a saturation value under magnetic field, but can more or less recover to its original value after sweeping down the field to zero.

Figure 5(a) shows the influences of the combined laser illumination and an applied low magnetic field ($H=0.6$ T) on the sample resistivity at 25 K. As is shown, a magnetic field of 0.6 T is applied at first (denoted H -on). The resistivity decreases and reaches a saturation value showing the negative magnetoresistance effect. When laser illumination (denoted laser-on) starts with the applied magnetic field switched on, the resistivity decreases rapidly at the beginning and slows down afterwards. While the laser illumination continues the applied magnetic field is switched off (denoted H -off). The resistivity decrease caused by the applied magnetic field is recovered gradually. When the magnetic field is applied a second time the negative magnetoresistance effect appears again. Finally, laser illumination is switched off with the applied magnetic field switched on. It is clearly shown that the PPC effect is unaffected by the magnetic field.

The influences of the combined laser and Hg-lamp illumination on the sample resistivity at 25 K are illustrated in Fig. 5(b). After an initial period of laser illumination the PPC effect occurs. Then the NPC effect is observed with a period of Hg-lamp illumination. We note that the resistivity only recovers to the value before Hg-lamp illumination; the PPC effect is unaffected by Hg-lamp illumination. Further prolonged laser illumination (~ 20 min) results in the further decrease and near saturation of the sample resistivity. At this stage, the NPC effect can still be observed with a period of

FIG. 5. Time dependence of the resistivity on (a) the combined laser illumination and a low magnetic field of 0.6 T and (b) the combined laser and Hg-lamp illumination at 25 K.

Hg-lamp illumination. The resistivity also recovers to the value before Hg-lamp illumination; the PPC effect is unaffected by Hg-lamp illumination. These results in Figs. 5(a) and 5(b) suggest that the PPC effect induced by the laser, the NPC effect induced by the Hg lamp, and the negative magnetoresistance effect are independent of each other and have different physical origins.

It is well known that a large lattice distortion is induced when the tolerance factor t is decreased by substituting La^{3+} with a smaller ion radius Nd^{3+} in $\text{La}_{2/3}\text{Ca}_{1/3}\text{MnO}_3$. This large lattice distortion weakens the double exchange interaction due to the reduction of the electron transfer integral between adjacent Mn^{3+} and Mn^{4+} ions¹¹ and results in carrier localization because of enhanced electron-phonon interaction. Furthermore, a disordered arrangement of Nd^{3+} and La^{3+} ions in the perovskite A site results in the formation of the local random potential field, intrinsic charge inhomogeneity, and then, in turn, it causes a possible phase separation.⁴ From the hysteresis phenomenon observed in Fig. 1 and previous measurements of magnetic properties⁹ of the sample we can conclude that the phase separation occurs at the temperature of $\sim 130 \text{ K}$, where the short-range FM order related to the double exchange interaction in rich-charge regions starts to build up. However, the matrix of poor-charge regions is a nonferromagnetic phase due to the carrier localization caused by the lattice distortion. In comparison with previous work on $\text{La}_{3/8-y}\text{Pr}_y\text{Ca}_{3/8}\text{MnO}_3$ (when $y \geq 0.3$) (Ref. 5) and $(\text{La}_{0.5}\text{Nd}_{0.5})_{2/3}\text{Ca}_{1/3}\text{MnO}_3$,¹² in which a phase separation with the coexistence of a short-range FM phase and a charge-ordering (CO) phase is found, we believe

FIG. 6. Schematic diagrams illustrate CG state and photon-inducing FM cluster growth model. (a) The system in the CG state before laser illumination; (b) FM clusters growing under laser illumination; (c) the boundaries of FM clusters in CG state modified and further relaxed after laser illumination.

that the nonferromagnetic matrix in our sample is a CO phase and the hysteresis phenomena observed in Figs. 1 and 4 are indications for the occurrence of a CO phase. The subtle competition between FM and CO interaction (that may be related to the long-range Coulomb interaction¹²) results in a CG state formation at temperatures below $\sim 50 \text{ K}$. The CG state at temperatures below $\sim 50 \text{ K}$ is illustrated schematically in Fig. 6(a). The FM-metallic clusters with different orientations of magnetic domains are frozen in a disorderly fashion and embedded into a matrix of a nonferromagnetic CO phase.

It has been reported that the oxygen deficiency of the samples can result in PPC and NPC effects in some perovskite manganites and high- T_C superconductors.¹³ However, the sample investigated in our experiments is annealed in an oxygen atmosphere, which can dispel the oxygen deficiencies and improve the properties of the films.¹⁴ Therefore, the photon-induced effects observed in our sample are unlikely to be originated from the oxygen deficiency.

The PPC effect observed in our sample can be qualitatively explained in terms of a photon-induced FM cluster growth model as shown in Fig. 6(b). In the CG state, it is believed that the percolative conduction through FM-metallic clusters in the CO state insulating matrix is mainly responsible for the current conduction. According to a previous study of optical conductivity spectroscopy in the system of $\text{Nd}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ there is a broad band centered around 1 eV in the conductivity spectrum at low temperatures,¹⁵ which is identified as charge-transfer excitation energy of an electron from the lower Jahn-Teller split e_g level of a Mn^{3+} ion to the e_g level of an adjacent Mn^{4+} ion. The photon energy of the He-Ne laser (1.96 eV) used in our experiment lies in the high-energy tail of the absorption band. Further investigations indicate that the PPC effect can be induced by a broad range of photon energy. In fact, we find that the PPC effect is more evident when the low photon energy near $\sim 1 \text{ eV}$ (found by using a tungsten lamp) is used to illuminate the sample instead of the He-Ne laser with the same power densities. However, a weak PPC effect but no NPC effect is observed when a Nd-yttrium aluminum garnet (YAG) pulse laser with the wavelength of 532 nm and average power density of $\sim 80 \text{ mW/cm}^2$ is used to illuminate the sample, which indicates that the photon excitation responsible for the PPC effect becomes gradually ineffective with increasing

photon energies. The higher photon energies from the Hg lamp with the wavelengths of 435.8, and 404.7 nm lie outside the absorption band and cannot promote this excitation. The incident photons promote e_g electrons to transfer from Mn^{3+} ions to the adjacent Mn^{4+} ions through an O_{2p} orbit in the FM clusters, which can enhance the double exchange interaction and result in the growth of FM-metallic clusters and an increase in percolative conduction paths. According to percolation theory¹⁶ percolative conduction can be expressed as $\Sigma \propto (P - P_c)^\mu$, where Σ is the conductance, P is the occupation probability of the conduction sites, P_c is the critical value of P for the percolative conduction, and μ is the critical exponent that depends on the dimensionality of the system. Therefore, a small change in FM cluster sizes can cause a large change in the conduction. In our experiment, the laser illumination causes the FM cluster to grow and we observe a rapid decrease of resistivity upon illumination.⁹ Since the FM domains are frozen at temperatures < 50 K, the CG state of the system remains after tuning off the laser. The photon-induced conductivity changes therefore persist, the PPC effect is observed, and the PPC effect only appears at temperatures below ~ 50 K.⁹ Furthermore, in the CG state the phase boundary is inherently unstable.^{17,18} The further decrease of the resistivity after the laser is switched off (see Fig. 2) can be explained as a result of the continued relaxation of the CG state as shown in Fig. 6(c). In other words, the CG state is more stable before any external stimulation but becomes very unstable afterwards. This is revealed by the resistivity fluctuations observed in Fig. 2. The continual relaxation after laser illumination is related to the further modification on the boundary of the FM cluster. Besides the percolative conduction mechanism, the tunneling conduction through the CO-insulating region between the neighboring FM clusters is also important. However, a constant current of 5 nA is employed in our experiments for the resistivity measurements at low temperatures so the tunneling conduction can be ignored.

The NPC effect observed in Fig. 3 clearly has a different origin in comparison with the PPC effect. The NPC effect can only be induced by the illumination of photons with energies near 3 eV or more. But photons with higher energies cannot promote charge transfer from Mn^{3+} ions to the adjacent Mn^{4+} ions in the FM clusters, which is responsible for the PPC effect. In addition, the time decay behavior in NPC cannot be fitted by the stretched exponential function used to describe the PPC effect.⁹ The behaviors of stretched exponential decay are observed in many disorder systems, and they usually characterize a relaxation from nonequilibrium to equilibrium systems.^{19–21} So we suggest that the origin of the NPC effect is related to the CO phase of the matrix. The photon illumination with higher energy mainly causes the charge-transfer excitation from O_{2p} to $\text{Mn}3d e_g$, which can disturb the regular ordering of $d_{3x^2-r^2}$ and $d_{3y^2-r^2}$ orbitals.²² So the strong carrier localization caused by the CO interaction can be ionized by the illumination of photons. The delocalization of carriers results in the increase of conductivity. In the homogeneous CO phase the relaxation time of the photon-ionized carrier is normally very short and the stable photoconductivity could not be built up. While in the CG

state, the FM-metallic clusters with different orientations of magnetic domains are frozen in a disorderly fashion and embedded into the matrix with the CO phase. The inhomogeneous microscopic electrical fields are formed in the system. This may result in the increase of relaxation time of the photon-ionized carrier and in turn the observation of an NPC effect at the temperatures near and below the frozen temperature ~ 50 K. We believe that the relaxation time of photon-ionized carrier is closely related to the temperature, the size of the FM cluster, and the cluster's distribution. The semiconducting-metallic transition observed in $\rho(T)$ at low temperature with Hg-lamp illumination (see the curve of the open circles in Fig. 1) indicates the increase of relaxation time with decreasing temperatures. In other words, the NPC effect originates from the increase of charge mobility induced by the photon illumination. However, the PPC effect results from the increase of conduction paths caused by FM cluster growth induced by the photon.

On the other hand, the decrease of resistivity caused by low magnetic field [see Fig. 5(a)] originates mainly from the decrease of disorderly scattering of FM domain boundaries due to the orientation alignment of FM domains in the applied field. No obvious resistivity relaxation is observed under low field. This implies that the size of the FM cluster in the CG state is unaffected under low magnetic field. However, when the magnetic field is higher than 2 T, the matrix with the CO-insulating phase begins to “melt” (or “collapse”) and changes into a FM-metallic phase. This results in a considerable decrease in resistivity as shown in Fig. 4. In comparison with a previous study of perovskite manganites with the CO phase,²³ the magnetic field needed to melt the CO phase is significantly lower here, indicating that the CO phase is unstable in our samples. The relaxations of resistivity observed (see Fig. 4, inset) under the stimulation of high magnetic fields are related to the dynamic behaviors of the collapsing and recovering CO state in this material. So the mechanism of the resistivity relaxation induced by high magnetic field is different from that by laser illumination.

Although the PPC and NPC effects originate from different mechanisms, they are related to the magnetic properties in the system with the particular CG state. We speculate that the CG state formation in $(\text{La-R})_{2/3}\text{Ca}_{1/3}\text{MnO}_3$ (where R are rare-earth ions with a smaller ion radius) (Refs. 7 and 8) with t close to ~ 0.907 is a common feature. In comparison with the systems with smaller t factors, the CG state in our systems is unstable and the phase transition in the region of $t \sim 0.907$ may easily occur due to external stimulations. The behavior of relaxation and photon-induced effects should exist in similar systems with the tolerance factor in the vicinity of 0.907. Very recently, the FM cluster growth induced by a Nd-YAG pulse laser with a wavelength of 532 nm in the thin film of a slightly Cr-doped CO state, $\text{Pr}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$, was clearly observed by Okimoto *et al.*²² We presume that the different phenomena of photon induction and responding of wavelengths are strongly related to the magnetic and microstructural properties of the systems. In the future, different systems will provide expedient methods to control the phase transition by using external stimulations, which may have

some implications for applications such as optical switches and magnetic sensores.

IV. CONCLUSION

The Hg-lamp illumination-induced NPC effect and the laser illumination-induced PPC effect are observed at temperatures near and below ~ 50 K in perovskite manganite films of $(\text{La}_{0.3}\text{Nd}_{0.7})_{2/3}\text{Ca}_{1/3}\text{MnO}_3$. The resistivity relaxations induced by laser and magnetic field are also observed. These effects can coexist and are independent of each other. Their origins can be explained in terms of the CG state formed by the FM-metallic clusters with different orientations of magnetic domains frozen in a disorderly fashion and embedded into the matrix with the CO phase as the temperature decreases to below ~ 50 K. The PPC effect originates from

photon-induced charge transfer in FM domains resulting in FM cluster growth, while the NPC effect originates from photon-induced ionization of localized carriers in the CO phase. Finally, a magnetic-field-induced resistivity relaxation effect is related to the dynamic behaviors of the collapsing and recovering of the CO state.

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¹For a review and related references, see *Colossal Magnetoresistive Oxides*, edited by Y. Tokura (Gordon and Breach, New York, 1999).

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