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Anomalous magnetotransport in $LaMn_{1-x}Te_xO_3$

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The LaMn_{1-x}Te_xO_{3+ δ} (x=0.1, 0.2, 0.3) were synthesized using solid-state reaction method for the first time. X-ray photoemission spectrum (XPS) shows that in the samples the Te ions have a valence of Te⁴⁺, and Mn³⁺ ions are partly converted into Mn²⁺ and Mn⁴⁺ due to the excess oxygen and Te doping. The magnetotransport associated with Mn²⁺, Mn³⁺ and Mn⁴⁺ was investigated. The experimental results show that the samples are insulator at 0 T when the amount of Mn³⁺ is much larger or less than the sum of Mn²⁺ and Mn⁴⁺; by contrast, the samples display metal to insulator transition with increasing temperature when the amount of Mn³⁺ is close to the sum of Mn⁴⁺. These anomalous magnetotransport behaviors were analyzed in the frame of the double-exchange (DE) mechanism.

colossal magnetoresistance, La-Te-Mn-O, magnetotransport

Doped LaMnO₃ have been widely studied due to a rich variety of physical properties^[1-4]. The prominent one is the colossal magnetoresistance (CMR) effect, a phenomenon of the resistive transition induced by the magnetic transition, and CMR is traditionally explained by the double exchange (DE)^[5] interaction. The key role in DE mechanism is played by Mn ions, the unique magnetic ions in LaMnO₃, which affect not only the transport behaviors of manganites by the network of Mn-O-Mn, but also the magnetism of materials by the spin variation of Mn ions.

As demonstrated by numerous studies, the valence of Mn ions in the manganites varies with the dopant, which is considered the intrinsic factor to understanding CMR effect. Mn ions are usually all trivalent (Mn^{3+}) in LaMnO₃.When La ions were substituted partially by divalent ions, such as Ca²⁺, Sr²⁺ etc., some Mn³⁺ would convert into Mn⁴⁺, and Mn³⁺-Mn⁴⁺ mixed valences are formed in the stable manganites of La_{1-x}A_xMnO₃^[5-8]. When La ions were substituted partially by tetravalent ions, such as Ce⁴⁺, Te⁴⁺ ... etc., some Mn³⁺ would con-

vert into Mn^{2+} , which leads to the $Mn^{2+}-Mn^{3+}$ mixed valence in $La_{1-x}A_xMnO_3$ (A=Ce⁴⁺, Te⁴⁺...)^[8,9]. The interactions between Mn^{4+} (or Mn^{2+}) ions and the neighboring Mn^{3+} ions via O^{2-} are believed to contribute to the DE effect.

The influence of the La-site substitution in manganites on DE mechanism is indirect, depending on the conversion of Mn^{3+} ions. The direct influence, undoubtedly, is the Mn-site substitution. Recently, most studies on the Mn-site substitution focus on the La_{1-x}A_xMnO₃ or LaMnO₃. It is found that the manganites with the formula of La_{1-x}A_xMn_{1-y}O₃ (A=Ca²⁺, Sr²⁺...; M=Zr²⁺, Cr³⁺, Ta⁴⁺,...) or LaMn_{1-x}B_xO₃ (B=T⁴⁺, Mo⁶⁺...) still remain Mn³⁺-Mn⁴⁺ (or Mn³⁺-Mn²⁺) mixed-valence, and undergo ferro- to para-magnetism (FM-PM) transition. However, both have the different magnetotransport behavior.

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La_{1-x}A_xMn_{1-y}O₃ usually undergo metal to insulator transition (MIT)^[10,11], but LaMn_{1-x}B_xO₃, such as LaMn_{1-x}- $Ti_xO_3^{[12]}$, LaMn_{0.096}Mo_{0.04}O₃^[13], LaMn_{0.096}Fe_{0.04}O₃^[14], etc., has no MIT, which are usually insulator at 0 T.

In addition, the manganites with or without the La and Mn site substitution are usually oxygen nonstochiometry (indicated by the deviation value δ which is a positive value and represents excess oxygen) in when the oxides are prepared in air or in the oxygen atmosphere. According to the studies of Tofield et al.^[15], van Roosmale et al.^[16] and the results from electron energy-loss spectroscopy (EELS) measured by Wang et al.^[17], it is widely accepted that the δ induces the conversion of Mn³⁺ ions into Mn⁴⁺ ions in the manganites. The amount of Mn⁴⁺ is depending on the value of δ with a relationship of [Mn⁴⁺]~ 2δ ^[18]. Obviously, the δ is also an important factor affecting DE effect^[19,20].

For the first time, we study lanthanum manganese oxides $LaMn_{1-x}Te_xO_{3+\delta}$ with excess oxygen. This material has three-kind valence of Mn^{2+} , Mn^{3+} and Mn^{4+} . In this paper, we focus on the magnetotransport of $LaMn_{1-x}Te_xO_{3+\delta}$ under different ratios of Mn^{2+} , Mn^{3+} and Mn^{4+} .

1 Experiment

Polycrystalline sample of LaMn_{1-x}Te_xO_{3+ δ} (x=0.1, 0.2, 0.3) was synthesized with the conventional solid-state reaction method. X-ray diffraction (XRD) of the powder sample was carried out by DMAX2400 diffractometer using Cu-K_{α} radiation at the room temperature, and XRD data obtained by the step scanning model with 0.02°/s were Rietveld refined by the program of DBW9411. XRD results show that the samples of x=0.1and x=0.2 are single phase, rhombohedral lattice with the space groups of $R\overline{3}C$. Where as the sample of x=0.3 is composed of two phases. The main phase is LaMn_{0.7}Te_{0.3}O_{3+ δ} with a symmetry of R₃C, and the additional phase is La2TeO6 with a symmetry of P212121. The main phase is much more than the additional phase according to the intensity of the maximum peak, the electronic structure of samples was measured by X-ray photoemission spectrometer (XPS) (EscaLab 220-IXL) with Al-K_{α} radiation and the scanning step of 0.05 eV. The oxygen content was analyzed by the Oxygen/Nitrogen Determinator (LECO TC-300) with the accuracy of 1 ppm, and all samples exhibit excess oxygen. The deviation value δ is about +0.09, +0.14 and +0.03 for *x*=0.1, 0.2 and 0.3, respectively. The magnetization (*M*) and the resistivity (ρ) of the samples were measured by the quantum design superconducting quantum interference device magnetometer (SQUID) MPMS-7 system. The temperature dependence of resistivity was recorded with the standard four-probe method.

2 Results and discussion

The XPS of LaMn_{0.9}Te_{0.1}O_{3.09} is showed in Figure 1 where the experimental data were analyzed by the XPSPeak program. The solid bold lines in Figure 1 represent the fitting results. The valence band (VB) spectrum of sample has a typical feature of two peaks, similar to that of LaMnO₃ doped on La-site. The corresponding value of two peaks is about 2.8 and 5.3 eV, respectively, near to 3 and 6 eV reported by Chainani et al.^[21] and Kowalczyk et al.^[22], indicating that the Mn_{3d} orbit and the O_{2p} orbit in LaMn_{1-x}Te_xO_{3+ δ} are hybridized extensively into Mn_{3d}-O_{2p} bonding states and nonbonding states. Hybridized orbits play a key role in the DE interaction. Besides, as shown in the inset of Figure 1, the Te- $3d_{5/2}$ core level is about 576.2 eV, close to 576.1 eV in TeO₂^[23] but 0.3 eV larger than that of La_{0.9}Te_{0.1}-MnO_{3.01}, indicating Te ions in LaMn_{1-x}Te_xO_{3+ δ} have 4+ valence states^[9]. In other words, LaMn_{1-x}Te_xO_{3+ δ} has a characteristic of Mn²⁺-Mn³⁺ mixed-valence state as reported in $La_{1-x}Te_{x}MnO_{3+\delta}$.

The temperature dependences of field-cooled (FC) magnetization (M) under the applied magnetic field of



Figure 1 X-ray photoemission spectrum of $LaMn_{0.9}Te_{0.1}O_3$ showing the valence band and Te-3d_{5/2} (inset).

0.01 T are plotted in Figure 2(a) for the samples of x=0.1and x=0.2, and Figure 2(b) for the sample of x=0.3. The FC curves of LaMn_{0.9}Te_{0.1}O_{3.09} show a single peak (Figure 2(a)), indicating that there is the antiferromagnetic phase at the low temperature. Further evidence stems from the measurement of AC magnetic susceptibility of sample of x=0.1, as shown in the inset of Figure 2(a) where the curve of susceptibility vs. temperature at 300 Hz exhibits a cusp in the low temperature region. On the one hand, with the increase of Te content, the antiferromagnetism of $LaMn_{1-x}Te_xO_{3+\delta}$ at the low temperature becomes less and less, even disappears, as shown by the experimental results of sample of x=02 (Figure 2(a)). When the Te content increases to 0.3, the *M*-*T* curve exhibits an obvious feature of the ferromagnetism, as shown in Figure 2(b). On the other hand, the maximum of magnetization of $LaMn_{1-x}Te_xO_{3+\delta}$ at the low temperature becomes more and more with the increase of Te content. Similar phenomenon was also observed in $LaMn_{1-x}M_xO_3$ (Mn=Ni²⁺, Zn²⁺, Li⁺, ...). Hébert et al.^[24] attributed it to the creation of Mn⁴⁺ species induced by the substitution of Mn by Ni or Zn or Li in LaMnO₃. As

a result, the DE interaction from $Mn^{4+}-Mn^{3+}$ favors the ferromagnetism of the samples, and depresses the A-type antiferromagnetism of LaMnO₃ matrix. However, the favorable ferromagnetism originating from Mn^{4+} - Mn^{3+} DE interaction will weaken when the content of dopant reaches a critical value.

The temperature variation of resistivity of LaMn_{0.9}-Te_{0.1}O_{3.09} under the applied field of 0 and 4 T is shown in Figures 3(a) and 3(b), respectively. Figure 3(a) shows that the ρ of LaMn_{0.9}Te_{0.1}O_{3.09} drops rapidly with the increase of temperature in the low temperature region, and then tends to a small value in the high temperature region, indicating an insulating behavior. The analogous variation of ρ -T has been reported in LaMn_{1-x}Sc_xO₃, $LaMn_{1-x}Mg_xO_3$, and $LaMn_{1-x}Fe_xO_3$, etc.^[12,14,25,26]. In addition, the contrasting samples of LaMnO_{3.11} and La_{0.9}Te_{0.1}MnO_{3.01} were fabricated under the same condition as LaMn_{1-x}Te_xO_{3+ δ_2} and the corresponding ρ -T curves of LaMnO_{3.11} and La_{0.9}Te_{0.1}MnO_{3.01} are shown in Figure 3(a) and in the inset of Figure 3(a), respectively. Figure 3(a) shows that LaMnO_{3.11} has a similar zero field ρ -T relationship as to that of LaMnO_{3.12}^[26,27] and





Figure 2 The temperature dependences of field-cooled magnetization under the applied magnetic field of 0.01 T. (a) $LaMn_{0.9}Te_{0.1}O_{3.09}$ and $LaMn_{0.8}Te_{0.2}O_{3.14}$, inset: The ac magnetic susceptibility of $LaMn_{0.9}Te_{0.1}-O_{3.09}$; (b) $LaMn_{0.7}Te_{0.3}O_{3.03}$.

Figure 3 The resistivity curves vs. temperature under the applied field of 0 T and 4 T. (a) $LaMn_{0.9}Te_{0.1}O_{3.09}$, $LaMnO_{3.11}$ and $La_{0.9}Te_{0.1}MnO_{3.01}$ (inset) at 0 T; (b) $LaMn_{0.9}Te_{0.1}O_{3.09}$ at 4 T.

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displays an insulating property similar to LaMn_{0.9}Te_{0.1}-O_{3.09}. However, the resistivity of LaMnO_{3.11} is much larger than that of LaMn_{0.9}Te_{0.1}O_{3.09} though both have a near oxygen content. On the other hand, La_{0.9}Te_{0.1}Mn- $O_{3.01}$, having the same Te-doping content as LaMn_{0.9}-Te_{0.1}O_{3.09}, exhibits a metal-insulator transition, which is different from LaMn_{0.9}Te_{0.1}O_{3.01}. Based on the results of Figure 3(a), some conclusion can be drawn: 1) The different site substitution by Te in LaMnO₃ would result in the different transport properties, which could help to judge the objective site of Te in LaMnO₃; 2) Te-doping on Mn-site in LaMnO₃ would significantly reduce the resistivity of LaMnO₃, but would not alter its transport behavior if the Te-doping content is too low. In fact, when Te content increases to 20%, the sample of LaMn_{0.8}Te_{0.2}O_{3.14} still remains insulating, as shown in Figure 4(a).



Figure 4 The resistivity curves vs. temperature of $LaMn_{0.8}Te_{0.2}O_{3.14}.$ (a) 0 T; (b) 4 T.

In contrast to the case of zero fields, at magnetic field of 4 T the magnetotransport of $LaMn_{0.9}Te_{0.1}O_{3.09}$ and $LaMn_{0.8}Te_{0.2}O_{3.14}$ underwent an essential change. As seen in Figures 3 (b) and 4(b), the ρ -T curves became a double peaks structure apart from a sharp fall at low temperature region.

The ρ -*T* relationship of LaMn_{0.7}Te_{0.3}O_{3.03} is shown in Figure 5. Compared with Figures 3 and 4, Figure 5 display several different features: 1) The resistivity of LaMn_{0.7}Te_{0.3}O_{3.03} is much lower than that of LaMn_{0.9}-Te_{0.1}O_{3.09} and LaMn_{0.8}Te_{0.2}O_{3.14}; 2) the ρ -*T* curves represent a single peak structure no matter what the applied field is 0 T (Figure 5(a)) or 4 T (Figure 5(b)). This is similar to the corresponding results of LaTe_{1-x}Mn_xO₃^[28]; 3) unlike Figure 3(b) and 4(b), the ρ -*T* curve in Figure 5(b) still keeps a single peak when a magnetic field of 4 T was applied.



Figure 5 The resistivity curves vs. temperature of $LaMn_{0.7}Te_{0.3}O_{3.03}$. (a) 0 T; (b) 4 T.

For the manganites of Mn-site doped LaMnO₃, it is common that the samples have the ρ -*T* curves as shown in Figures 3(a) and 4(a). However, why do the curves of Figure 5 exhibit the metal-insulator transition behaviors when the temperature increases? Why is the curve of the single peak of Figure 5(b) while the curve of Figure 3(b) or Figure 4(b) is double peak under the same applied field of 4 T?

To answer the above questions, it is necessary to ana-

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lyze the variation of Mn ions in $LaMn_{1-x}Te_xO_{3+\delta}$. As we know, Mn ions of the samples are composed of Mn²⁺, Mn³⁺ and Mn⁴⁺. One Mn site is occupied by Te⁴⁺, meaning leading to that the valence of one Mn³⁺ decreases and another Mn³⁺ converts into Mn²⁺, i.e. substitution of Mn by Te⁴⁺ equals decreasing the valence of two Mn³⁺ and adding one Mn²⁺. Besides, excess oxygen also causes some Mn³⁺ to change into Mn⁴⁺. Therefore, the formula of $LaMn_{1-x}Te_xO_{3+\delta}$ should be rewritten as $LaMn_{1-2x-2\delta}^{3+}Mn_{2\delta}^{4+}Mn_x^{2+}Te_xO_{3+\delta}$ according to the requirement of charge equilibrium and stable perovskite structure. Therefore, the amount ratio of Mn²⁺, Mn³⁺ and Mn⁴⁺ are obtained as the following:

 $[Mn^{3+}]:[Mn^{4+}]:[Mn^{2+}]=(1-2x-2\delta):2\delta:x.$

Now, substituting the number of δ and x into the above formula, the amount ratio of Mn²⁺, Mn³⁺ and Mn^{4+} of all samples are shown in Table 1. $[Mn^{3+}]$ is maximum in LaMn_{0.9}Te_{0.1}O_{3.09}, larger than two times of the sum of Mn²⁺ and Mn⁴⁺. Thus, the probability of the formation of the Mn³⁺-O-Mn³⁺ is also highest in the network of M-O-M (M=Te⁴⁺, Mn²⁺, Mn³⁺, Mn⁴⁺) which provide the transport channels for the carriers. Consequently, the superexchange of the predominant Mn³⁺-O- Mn^{3+} gives rise to the antiferromagnetism of LaMn_{0.9}-Te_{0.1}O_{3.09} at the low temperature, as shown in Figure 2(a). At the same time, the transports of the carriers through M-O-M would be destroyed when Mn³⁺ are partially replaced by Te⁴⁺. It makes the resistivity of the compound increase, leading to that the compound converts into insulator as shown in Figure 3(a). However, in LaMn_{0.9}Te_{0.1}O_{3.09} there exists also a part of the network of Mn⁴⁺-O-Mn³⁺ and Mn²⁺-O-Mn³⁺. The DE interaction of the above network would be strengthened under the applied magnetic field, resulting in the decrease of the resistivity of the compound (Figure 3(b)). Similar to the case of LaMn_{0.9}Te_{0.1}O_{3.09}, LaMn_{0.7}Te_{0.3}O_{3.03} has more Mn^{2+} and Mn^{4+} . The sum of Mn^{2+} and Mn^{4+} is almost equal to that of Mn³⁺, indicating that the network of Mn⁴⁺-O-Mn³⁺ and Mn²⁺-O-Mn³⁺ become predominant in the network of M-O-M. As the aforementioned, the DE interaction of Mn⁴⁺-O-Mn³⁺ and Mn²⁺-O-Mn³⁺ would make the manganites tend to ferromagnetism (Figure 2(b)) and MIT. When a magnetic field is applied, the DE interaction further decreases the resistivity of the material and leads to CMR, just as shown in Figure 5.

As for $LaMn_{0.8}Te_{0.2}O_{3.14}$, the ratio of $[Mn^{3+}]$ to the

Table 1 The amount of Mn^{2+} , Mn^{3+} and Mn^{4+} in $LaMn_{1-x}Te_xO_{3+\delta}$ for different δ and x

LaMn _{1-x} Te _x O _{3+δ}	$[Mn^{3+}]$	$[Mn^{4+}]$	$[Mn^{2+}]$	$[Mn^{3+}]:([Mn^{4+}]+[Mn^{2+}])$
$x = 0.1, \delta = 0.09$	0.62	0.18	0.1	31:14 ~ 2:1
$x = 0.1, \delta = 0.14$	0.32	0.28	0.2	32:48=2:3
$x = 0.3, \delta = 0.03$	0.34	0.06	0.3	34:36≈1:1

sum of Mn^{2+} and Mn^{4+} is about 2:3. In other words, the ions of Mn^{2+} and Mn^{4+} become the predominant ions in the samples, indicating that the network of Mn^{4+} -O- Mn^{2+} play a leading role in the network of M-O-M. Obviously, there could be existed the ferromagnetic superexchange interaction in Mn^{4+} -O- Mn^{2+} but no DE, which makes the material tend to insulator at 0 T. Furthermore, the resistivity of $LaMn_{0.8}Te_{0.2}O_{3.14}$ should lower than that of $LaMn_{0.9}Te_{0.1}O_{3.09}$ because the former have more the network of Mn^{4+} -O- Mn^{3+} and Mn^{2+} -O- Mn^{3+} , as shown in Figure 4(b).

By now, Most results shown in Figures 3-5 have been qualitatively explained by the traditionally DE mechanism. However, it is difficult to explain by using only DE that the resistivity drops dramatically with the increasing temperature in the low temperature region (Figures 3(b) and 4(b)). This complex magnetotransport behavior, maybe originating from John-Teller effect^[29]or other unknown magnetoresistance mechanism, need further study in the following aspects: It is worthwhile to analyze whether La2TeO6 affects the transport of LaMn_{0.7}Te_{0.3}O_{3.03}. Note that La₂TeO₆ is lack of magnetic ions analogous to Mn, indicating no DE interaction occur in La₂TeO₆ with or without the applied magnetic field. Besides, the amount of La₂TeO₆ is very low in the samples of x=0.3, so that the influence induced by La₂TeO₆ is limited on LaMn_{0.7}Te_{0.3}O_{3.03}. Considering the above two factors, it is reasonable to ignore the influence of La2TeO6 when discussing the magnetic and transport of LaMn_{0.7}Te_{0.3}O_{3.03}.

The double peak structure shown in the Figures 3(b) and 4(b) can be attributed to the inhomogeneous distribution of oxygen content in the samples. According to Sun et al.'s^[30] work, when fabricating the sample of La_{0.72-x}Gd_xCa_{0.3}Mn_yO_{3+ δ} by solid-state reaction, the different activities of La₂O₃ and La(OH)₃ leaded to inhomogeneous distribution of oxygen content in the compound, which caused two peak of the ρ -*T* curves. Wu et al.^[19] also confirmed that the double peak shown in ρ -*T* curves of La_{0.9}MnO_{3+ δ} were closely related to the oxygen content. The peak at low temperature shifted to high

temperature and was weaken with the increasing oxygen content when $La_{0.9}MnO_{3+\delta}$ was treated with different post-annealing and oxygenation processes.

Because both LaMn_{0.9}Te_{0.1}O_{3.09} and LaMn_{0.8}Te_{0.2}O_{3.14} are prepared by solid-state reaction and have excess oxygen content of 0.09 and 0.14, respectively, it is possible that there are inhomogeneous distribution of oxygen content in the samples, leading to the double peak curves in Figures 3(b) and 4(b). Moreover, the more the δ is, the larger the distance between the two peaks is. As LaMn_{0.7}Te_{0.3}O_{3.03} has a little excess oxygen, and its inhomogeneous oxygen distribution has no influence on the magnetoresistance of the material, the ρ -*T* curves of LaMn_{0.7}Te_{0.3}O_{3.03} exhibits a single peak.

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3 Conclusions

LaMn_{1-x}Te_xO_{3+ δ} was synthesized by the solid-state reaction and its magneto transport properties were investigated. In this compound, Mn ions have three kinds of valence: Mn²⁺, Mn³⁺ and Mn⁴⁺, and are closely associated with the magnetotransport. It is found that the different the ratio between Mn³⁺ and the sum of Mn²⁺ and Mn⁴⁺ result in the different transport behavior. The samples with the ratio of about 2:1 or 2:3 exhibit the insulating state, while the samples with the ratio of about 1:1 display MIT. These anomalous magnetotransport behaviors are analyzed in the frame of the DE mechanism

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