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Research Article

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Preparation and characterization of double perovskit $LaKFe_{2-x}Mo_xO_6$

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ABSTRACT

The micro-powder of $LaKFe_{2-x}MO_xO_6$ (x=0.45, 0.55, 0.65) was prepared by the sol-gel technique. After annealing, the the XRD spectrum of the samples which annealed at different temperature was tested and the influence on structure of the samples was studied. The samples belong to monoclinic system and the space group is P21/n by crystallographic plane index of diffraction peak of the main phase. The diffraction peaks of $LaKFe_{2-x}MO_xO_6$ move to the right with the increased Mo doped in. The lattice parameters and volume decrease as x rise. The results show that the optimal doping ratio is obtained as x is 0.45 and the optimal samples of the series is $LaKFe_{2-x}MO_xO_6$.The diffraction peaks of$LaKFe_{2-x}MO_xO_6$move to the right with the rise of temperature and the optimal annealing temperature of it is 1473K.

Key words: Double Perovskite, Magnetic properties, colossal magnetoresistance, sol-gel

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INTRODUCTION

Crystal structure and physical properties of the perovskite-type manganese oxide is extremely sensitive to their stoichiometric ratio of the constituent elements so minor modulation in composition may change a lot the nature of the compound. So the doping method become an important method in the study of such compounds. The concentration and mobility of the carriers can be improved by doping. Colossal magnetoresistance materials gets great deal of attention because it has a good prospect in the field of electronics, especially in magnetic sensors and high-density magnetic memory. Rare earth manganese oxide become a research focus because they have a wealthy physical meaning due to the colossal magnetoresistance (CMR) [1]. The nature of the substance is directly determined by the structure of matter, so the rich physical properties of perovskite-type oxides is closely related to its own special structure[2-5] and double perovskite oxides is an important part of it. double perovskite oxides have significant applications in superconductivity[6], colossal magnetoresistance room temperature magnetoresistance materials and etc. Previous research has shown that $Sr_2FeMoO_6$and Sr_2FeReO_6 have CMR at room temperature.[7-10] Pradheesh R researched spin glass state in weakly ferromagnetic Sr_2FeCoO_6double perovskite[11],

The double perovskite oxides can expressed as $A_2B'B''O_6$ and the schematic picture is shown in Figure 1, wherein A represents an element of the larger ionic radius such as divalent alkaline earth metals Ca, Ba, Sr and Pb and B' or B'' are transition elements such as Fe, Co, Ni and Mn with a smaller ion radius[12].

In the doped oxide the radius of A and B was difference which resulted in crystal structure distortion. It can be measure by the tolerance factor t[13].

$$R_A+R_O = t\ (R_B+R_O) \qquad (1)$$

The radius of ion A, B and O can be expressed as R_A, R_B and R_O. Usually if t is in the range of 0.89 to 1.02 they can form a stable crystal structure. When t is 1, it corresponds to a full cubic dense structure. With the doping on A or B site, the t-factor and symmetry of the system will change. Meanwhile, the tolerance factor t is closely related to temperature and pressure. Usually with the increasing of temperature t becomes larger and with the decreasing of pressure t becomes larger.[14]

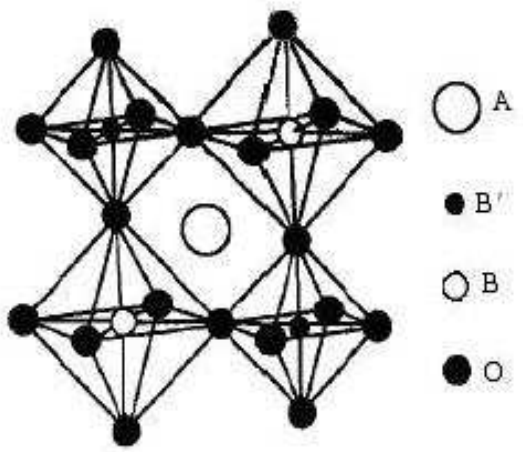

Figure 1 Schematic picture of double perovskite structure $A_2B'B''O_6$

Experiment

The micro-powder of $LaKFe_{2-x}Mo_xO_6$ (x=0.45,0.55,0.65) were prepared by the sol-gel technique[15]. The crude materials included La_2O_3(99.99%), KNO_3(99.99%), Fe $(NO_3)_3 \cdot 9H_2O$(AR), $(NH_4)_2MoO_7$ etc. The experimental procedure is as follows:

(1) to weigh materials with an electronic balance;
(2) to configure raw material into a solution;
(3) to stir and heat the solution and to get black powder sample;
(4)to calcine the powder in the cased furnace at 1073 K for 2h
(4) to tablet the calcined the powder with a size of Φ10 mm×2 mm in the squeezer;
(5) to anneal the samples at 1473K, 1573K and 1673K for 24 h in the cased furnace and then naturally cool to room temperature in air.

The structure of the samples was determined by X-ray diffractionon a diffractometer of RK D/Max-γA type (Cu Kα) with rotating target.The diffraction peaks were calibrated.

RESULTS AND DISCUSSION

Figure2-4 show the XRD patterns of $LaKFe_{2-x}Mo_xO_6$ annealed at 1473K, 1573K and 1673K respectively. Figure 5 shows the XRD patterns of $LaKFe_{1.55}Mo_{0.45}O_6$ annealed at 1473K, 1573K and 1673K.

The samples belong to monoclinic system and the space group is $P2_1/n$ by crystallographic plane index of diffraction peak of the main phase[16]. The results show that the diffraction peaks move to the right with the increase Mo doped in. As mentioned earlier , the double perovskite oxides is expressed as $A_2B'B''O_6$, Fe and Mo is in the position of B' and B" in this series of experiments. By doping different content of Mo, the valence of it is change as the increase of x. Gradually Mo^{+6}became more than Mo^{+5}, and the ionic radius of Mo^{+6} is smaller than Mo^{+5}, so the total ionic radius of B' and B" become smaller. This leads to the lattice parameters and volume decrease, thus the diffraction peaks move to the right.

In Figure 2, the characteristic peaks in the XRD spectrum of $LaKFe_{2-x}Mo_xO_6$ is very obvious and the spectrum has almost no impurity one so the crystallinity of the samples is full. In Figure 3, impurity phase appears in the spectrum of $LaKFe_{2-x}Mo_xO_6$ when x is 0.55 and 0.45 which was annealed at 1573K. The spectrum of $LaKFe_{1.55}Mo_{0.45}O_6$ has no impurity one. In Figure 4, the spectrum of three samples have impurity phase all when they were annealed at 1673K. If the annealing temperature is too high, it is not conducive to the crystallization of the samples. Finally, the optimal doping ratio is obtained as x is 0.45 and the optimal samples of the series is $LaKFe_{2-x}Mo_xO_6$.

Through the research above, sample $LaKFe_{1.55}Mo_{0.45}O_6$ is relatively stable in the high temperature process of annealing. Figure 5 shows the XRD spectrum of $LaKFe_{1.55}Mo_{0.45}O_6$ annealed at 1473K, 1573K and 1673K. It can be

seen that the crystallinity of $LaKFe_{1.55}Mo_{0.45}O_6$ is full when the annealing temperature is 1473K and the impurity phase appears when the annealing temperature rise to 1573K and 1673K. The diffraction peaks move to the right with the rise of temperature, this because the higher annealing temperature leads to more fully crystallization so the lattice parameters and volume decrease so the diffraction peaks move to the right. So the optimal annealing temperature of $LaKFe_{1.55}Mo_{0.45}O_6$ is 1473K.

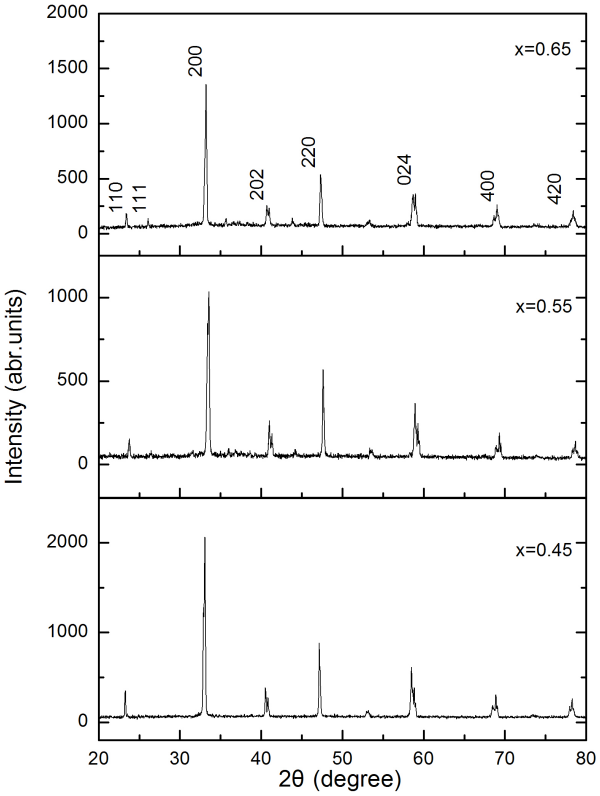

Figure 2 the XRD spectrum of $LaKFe_{2-x}Mo_xO_6$ annealed at 1473K

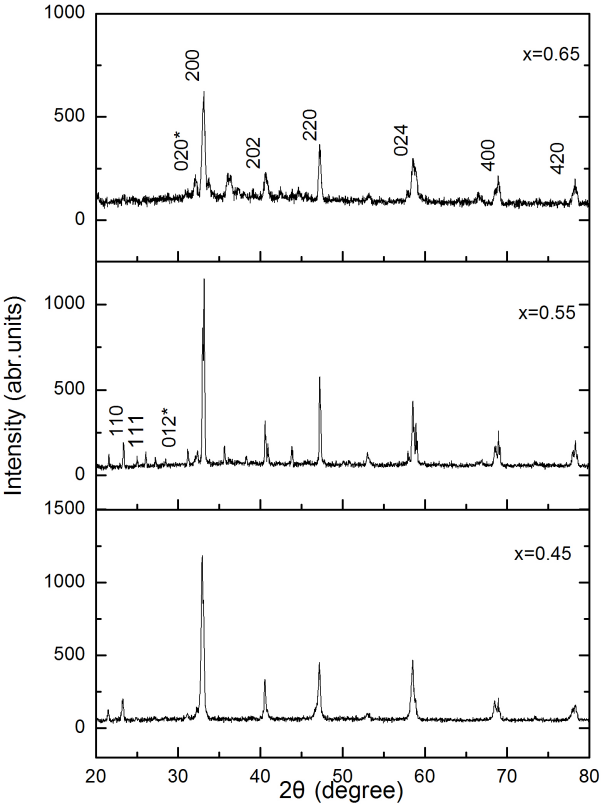

Figure 3 the XRD spectrum of $LaKFe_{2-x}Mo_xO_6$ annealed at 1573K

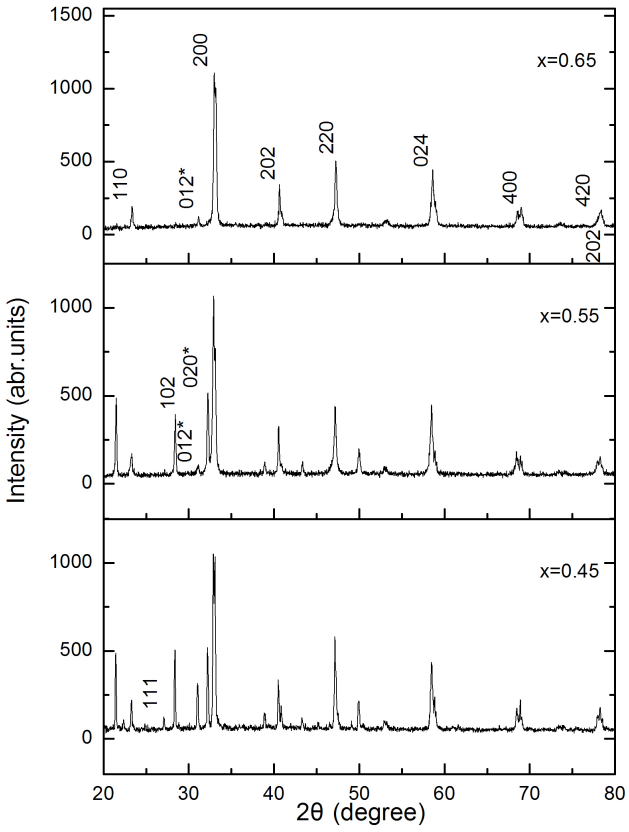

Figure 4 the XRD spectrum of $LaKFe_{2-x}Mo_xO_6$ annealed at 1573K

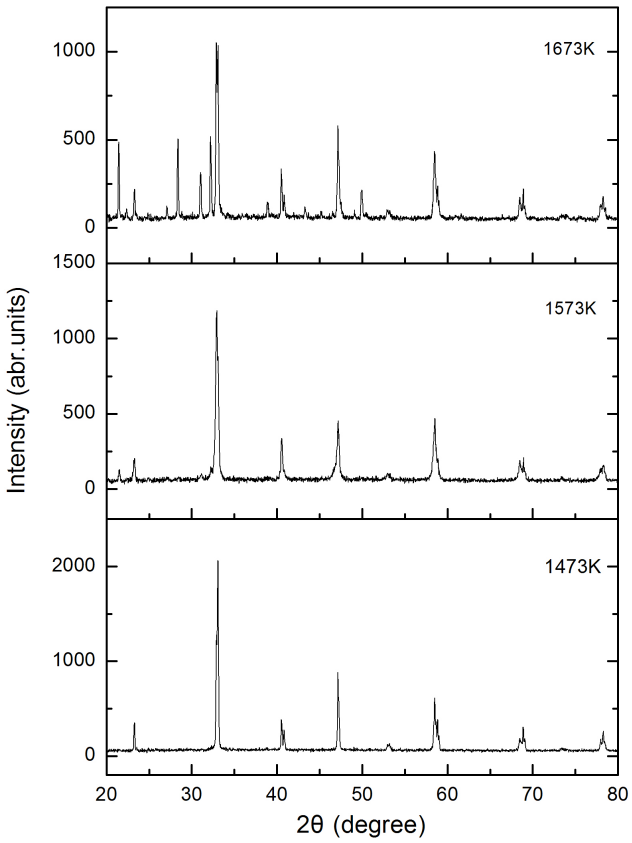

Figure 5 the XRD spectrum of $LaKFe_{1.55}Mo_{0.45}O_6$ annealed at 1473K, 1573K and 1673K

CONCLUSION

The micro-powder of $LaKFe_{2-x}M_{Ox}O_6$ (x=0.45,0.55,0.65) were prepared by the sol-gel technique. After annealing treatment, the X ray diffraction tests of different annealing temperature on the influence of the structure of the samples was studied.The optimal doping ratio is obtained as x=0.45 and the optimal samples of the series is obtained as $LaKFe_{2-x}Mo_xO_6$ and the optimal annealing temperature of it is 1473K.

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