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journal homepage: www.elsevier.com/locate/sscStructure, magnetic and electrical properties of disordered double perovskite $\text{Pb}_2\text{CrMoO}_6$ H.F. Zhao^a, L.P. Cao^a, Y.J. Song^{a,b}, S.M. Feng^a, X. Shen^a, X.D. Ni^b, Y. Yao^a, Y.G. Wang^a, C.Q. Jin^a, R.C. Yu^{a,*}^a Beijing National Laboratory for Condensed Matter Physics, Institute of Physics, Chinese Academy of Sciences, Beijing 100190, China^b Department of Physics, University of Science and Technology Beijing, Beijing 100083, China

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ABSTRACT

We prepared $\text{Pb}_2\text{CrMoO}_6$ under high pressure and high temperature. The sample has a heavily or fully disordered double perovskite structure ($Pm-3m$, $a=3.9472 \text{ \AA}$). It shows a weak ferromagnetic behavior ($T_C \sim 33 \text{ K}$) and a significant low magnetization ($\sim 0.095 \mu_B/\text{f.u.}$ at 7 T and 5 K), which is discussed based on the cation disorder. The temperature dependence of the electrical resistivity of $\text{Pb}_2\text{CrMoO}_6$ exhibits a semiconducting behavior, which could be well understood by the 3D variable range hopping model. No significant magnetoresistance was observed in this sample.

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1. Introduction

Double perovskite oxides $\text{A}_2\text{BB}'\text{O}_6$ have attracted considerable attention since the discovery of colossal magnetoresistance in $\text{Sr}_2\text{FeMoO}_6$ [1–3]. As a most studied member of $\text{A}_2\text{BB}'\text{O}_6$ family, $\text{Sr}_2\text{FeMoO}_6$ is a half-metallic ferrimagnet with a high Curie temperature (T_C) of $\sim 415 \text{ K}$, and low field magnetoresistance of $\text{Sr}_2\text{FeMoO}_6$ is attributed to the spin-polarized electron scattering at grain or magnetic domain boundaries. The large room-temperature magnetoresistance makes such materials a good candidate for the applications including nonvolatile magnetic random access memory, magnetic read heads for hard drives and magnetic sensor, etc.

Nowadays, experimental and theoretical studies are still being carried out to seek $\text{A}_2\text{BB}'\text{O}_6$ materials with optimized properties. Up to now, the $\text{A}_2\text{BB}'\text{O}_6$ ($A=\text{Ca}, \text{Sr}, \text{Ba}$; $B=\text{Cr}, \text{Fe}$; $B'=\text{Mo}, \text{W}, \text{Re}$) analogues of $\text{Sr}_2\text{FeMoO}_6$ have been systematically investigated [4–6], while the Pb-based analogues are less studied due to the toxicity of lead, although the ionic radius of Pb^{2+} is very close to that of Sr^{2+} . Meanwhile, unlike the alkaline earth elements, Pb ion has additional 6 s lone pair electrons, which is usually related to the ferroelectricity of perovskite materials [7]. Recently, first-

principle calculations performed by Gong et al. [8] predict that the ordered double perovskite $\text{Pb}_2\text{CrMoO}_6$ is a half-metallic ferrimagnet with a $T_C \geq 480 \text{ K}$. However, no experimental study of $\text{Pb}_2\text{CrMoO}_6$ has been reported up to now. So, we attempted to synthesize $\text{Pb}_2\text{CrMoO}_6$, and finally got a single phase with the nominal formula, then investigated its structural, magnetic and transport properties.

2. Experimental

Our attempts to synthesize $\text{Pb}_2\text{CrMoO}_6$ at ambient pressure (sintering of stoichiometric mixture of raw materials in vacuum, sintering in a flowing Ar/H_2 gas) were unsuccessful. However, single-phase $\text{Pb}_2\text{CrMoO}_6$ could be fabricated via a high pressure-high temperature synthesis route. Stoichiometric mixture of PbO , Cr_2O_3 , Mo and MoO_3 was ground within an agate mortar for 1 h, pressed into pellets of $\Phi 6 \text{ mm}$, and then enveloped in Au foil. The ultimate synthesis was conducted in a cubic anvil-type apparatus at about $880 \text{ }^\circ\text{C}$ and 5.5 GPa for 30 min. Pressure was released slowly after quenching the specimen to room temperature. The cation stoichiometry of the synthesized sample was examined with inductively coupled plasma-atomic emission spectrometry (ICP-AES) (IRIS Advantage, Thermo Elemental).

The crystal structure of the ceramics were characterized by powder X-ray diffraction (XRD) (X'Pert Pro, Philips) with $\text{Cu K}\alpha$ radiation and transmission electron microscopy (TEM) (Tecnai F20 FEG, Gatan) analysis. For the $\text{Pb}_2\text{CrMoO}_6$ slice is fragile, the sample

* Corresponding author at: Beijing National Laboratory for Condensed Matter Physics, Room 1025, Building M, Zhongguancun South 3rd Street, Beijing 100190, China. Tel.: +86 10 82649159.

E-mail address: rcyu@aphy.iphy.ac.cn (R.C. Yu).

for TEM observation was prepared as a cross-sectional TEM sample with single crystal Si as the supporting material.

The magnetic property measurements were performed on Quantum Design Physical Property Measurement System (PPMS). In the electrical property measurements, temperature control and measurement were performed on an Oxford Maglab measuring system, the resistivity data were measured with an external Keithley 2182A nanovoltmeter.

3. Results and discussion

The normalized atomic ratio of Pb:Cr:Mo is about 51.80:23.63:24.57 measured by ICP-AES and agrees well with the nominal value of 50:25:25. The XRD pattern of the synthesized $\text{Pb}_2\text{CrMoO}_6$ can be indexed with a simple cubic unit cell with the parameter $a=3.9472 \text{ \AA}$ using WinPLOTR and DICVOL [9] as shown in Fig. 1. Since no systematic absence is observed on the XRD pattern, there are only 5 possible space groups and there is one-to-one correspondence between these space groups and the 5 point groups of cubic lattice. For a certain point group, the exhibited symmetries at different zone axes, i.e. the diffraction group (DG), are different. For different diffraction groups, the exhibited symmetries in the convergent beam patterns (such as whole pattern, dark field, bright field and $\pm G$) are different. Buxton et al. [10]

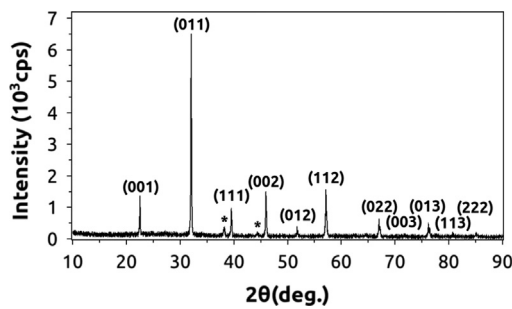


Fig. 1. XRD pattern of $\text{Pb}_2\text{CrMoO}_6$. The peaks marked with * are from Au.

established the relationships among point groups, diffraction groups along different directions and the exhibited symmetry in CBED patterns. Thus, by observing the symmetry in CBED patterns along different orientations of the sample, the diffraction group could be determined and then the point group is concluded. At last, the space group can be uniquely determined with regard to the one-to-one correspondence mentioned before. The correspondence among the 5 space groups, point groups, diffraction groups along $[111]$ and $[001]$ orientations, and whole pattern symmetry (WPS) is summarized in Fig. 2(a). For there are mirror planes on the whole pattern of the $[111]$ orientation, the possible space groups are reduced to $P-43m$ and $Pm-3m$. Since the whole pattern of the $[001]$ orientation exhibits 4-fold symmetry rather than 2-fold symmetry, the space group $P-43m$ is also excluded. As a result, the space group of the sample is uniquely determined to be $Pm-3m$ (221). To make sure our result is representative, we acquired the CBED patterns from many particles and all of them give the same information.

In the case of $\text{Sr}_2\text{FeMoO}_6$ [11], although the order-related peaks are totally absent, Rietveld analysis of the XRD pattern indicates the presence of a significant extent of ordering at the Fe/Mo site. However, we didn't find the evidence of the presence of the Cr/Mo ordering in our samples during our TEM work. In a word, our samples crystallize in a heavily or completely disordered double perovskite phase, different from A_2BMoO_6 ($A=\text{Ca, Sr, Ba}$; $B=\text{Cr, Fe}$); [5,12,13], but same as $\text{Ca}_2\text{CrMoO}_6$ [14], $\text{Pb}_2\text{FeMoO}_6$ [15].

Fig. 3 displays the temperature dependence of zero-field-cooled (ZFC) and field-cooled (FC) magnetization of $\text{Pb}_2\text{CrMoO}_6$ under 1000 Oe. The magnetization behavior of $\text{Pb}_2\text{CrMoO}_6$ is almost the same as that of $\text{Ba}_2\text{CrMoO}_6$ [13]. With decreasing temperature, the ZFC and FC curves start splitting below $\sim 30 \text{ K}$, and the ZFC curve shows a narrow cusp at $\sim 24 \text{ K}$, while the FC curve increases monotonically. The inverse magnetization is not Curie-Weiss like up to 300 K, and the curvature is consistent with short range ferromagnetic interactions. And the lack of magnetic saturation below the transition temperature also reveals that the ferromagnetism cannot be long-range order. The transition temperature ($T_c \sim 33 \text{ K}$) is obtained from the inflection point of the first

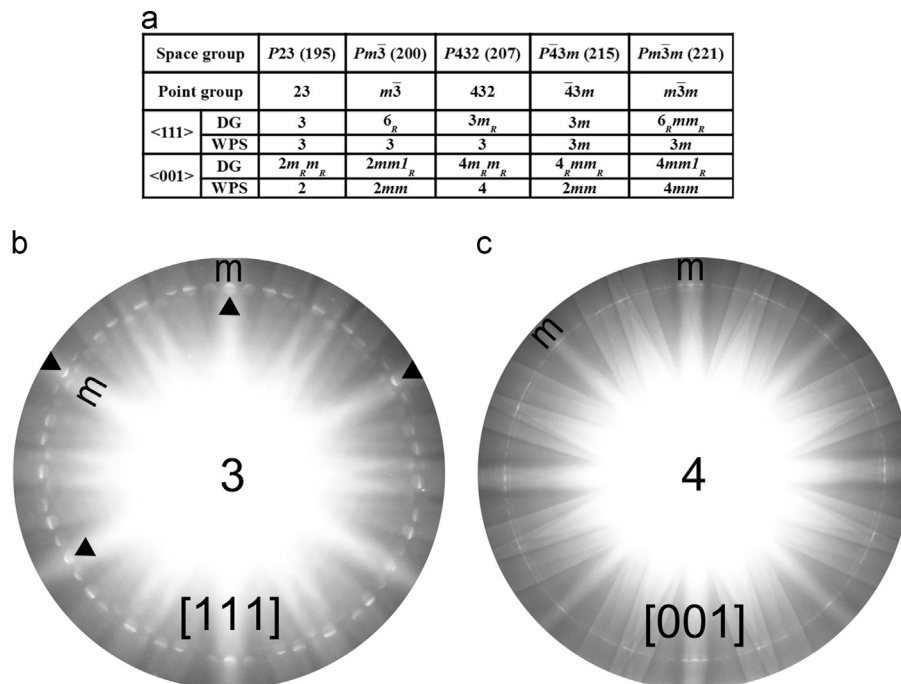


Fig. 2. (a) The correspondence among the 5 space groups with no systematic absence, point groups, diffraction groups (DG) along $[111]$ and $[001]$ orientations, and whole pattern symmetry (WPS). The experimental whole CBED patterns along (b) $[111]$ and (c) $[001]$ orientations were obtained at 200 kV and 160 kV, respectively.

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