

Contents lists available at [ScienceDirect](www.sciencedirect.com/science/journal/00220248)

Journal of Crystal Growth

journal homepage: www.elsevier.com/locate/jcrysgro

Cationic order-related magnetoresistivity and half-metallicity in bulk $Pb_2FeMoO₆$ grown by high pressure synthesis

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ARTICLE INFO

Communicated by Rosalba Fittipaldi

Keywords: A1. Solid-state reaction A2. High pressure/high temperature crystal growth B1. Perovskites B2. Magnetoresistive materials B2. Half-metallic materials B2. Magnetic materials

ABSTRACT

We investigate the electric properties of Pb_2FeMoO_6 (PFMO), a double perovskite that can be grown in a bulk pure phase only via High Pressure/High Temperature solid-state reaction. The as-obtained PFMO is characterized by a high degree of cationic ordering at the B site between Fe^{3+} and Mo⁵⁺ and a ferrimagnetic transition with T_N around 275 K. A semi-metallic to half-metallic transition seems to occur when the ferrimagnetic order arises thanks to the localization of a minority fraction of electrons on the Mo site. In this thermal regime, the system displays asymmetric magneto-resistivity, with selective-polarized carriers promoted by intense negative magnetic fields applied across the N´eel transition during the cooling ramp. This cation-ordering related feature vanishes when the Mo-O bond length stabilizes, indicating a strong connection between the magnetic and electrical properties of this compound.

1. Introduction

Double perovskites with general formula $A_2BB'O_6$ are considered interesting multifunctional materials in the material science community. The large stability and tolerance to a wide range of chemical substitutions, typical of the perovskite structure, allow to explore further degrees of freedom represented by the modification on the A and B sites' periodic arrangement: for instance, cation ordering, superimposed magnetic exchange interactions and unusual electrical behaviors are possible consequences. Double perovskites having the B and B' sites occupied by ions with different radii and/or oxidation states may show half-metallic properties [\[1,2\]](#page-3-0) due to the coexistence of bound and delocalized electrons, determining in some cases the presence of spinpolarized carriers [\[3,4\];](#page-3-0) same structures can display also multiferroism [5–[7\]](#page-3-0), magnetoresistance [\[8,9\]](#page-3-0) or catalytic properties [\[10,11\].](#page-3-0) Specifically, among the perovskite oxides, a wide range of magnetoresistive compounds has been reported in the literature, such as for SrRuO₃ single-crystal films $[12]$ or La_{0.7}Ca_{0.3}MnO₃ films $[13]$. By properly tuning the stoichiometric amount of the cationic species, it has been also demonstrated that colossal or even giant magnetoresistive effects can be achieved, as in the case of $La_{0.3}Pr_{0.4}Ca_{0.3}MnO₃ films [14]$ $La_{0.3}Pr_{0.4}Ca_{0.3}MnO₃ films [14]$. It is important

to underline that the magnetoresistance has been reported to vary strongly in polycrystalline systems, such as for $La_{2-2x}Ca_{1+2x}Mn_2O_7$ layered-perovskite [\[15\]](#page-4-0), since this physical property is dependent on the grains size, surface defects, impurities, and nature of grain boundaries [\[16\]](#page-4-0). Beyond the fundamental scientific interest in investigating these compounds, large magnetoresistive materials are industrially employed for angle, speed, and position sensing, in automotive as well as magnetic field and electrical current sensing, making them intriguing functional compounds in effective technological applications [\[17](#page-4-0)–19].

The double perovskite $Sr_2FeMoO₆$ is an important member among these magnetoresistive compounds families, and it has been accurately studied due to its high ferrimagnetic transition temperature ($T_N = 420$ K) together with room-temperature magnetoresistance, which makes it an interesting candidate for spintronic applications [20–[22\].](#page-4-0) Specifically, the presence of both the Fe^{3+} (3d⁵) and Mo⁵⁺ (4d¹) ions confers to the material half metallicity $[23]$, which results strongly affected by the possible presence of antisite defects [\[24\].](#page-4-0)

The synthesis conditions are known to play a central role in determining the degree of cation order, and consequently the properties of the system. Particularly, the application of high pressure during the solid-state reaction process is reported to decrease the amount of antisite

<https://doi.org/10.1016/j.jcrysgro.2024.127629>

Available online 23 February 2024 Received 25 October 2023; Received in revised form 26 January 2024; Accepted 18 February 2024

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defects $[25,26]$. Pb₂FeMoO₆ (PFMO) is a double perovskite with the substitution of Sr^{2+} for the isovalent ion Pb²⁺ which can be stabilized in a pure bulk phase under high pressure/high temperature (HP/HT) reaction conditions [\[27\]](#page-4-0). This compound is characterized by low room temperature (RT) resistivity and by the retention of a low field magnetoresistive character, while the electronic conduction process has been associated with the variable-range hopping of the carriers in samples with a high degree disorder between iron and molybdenum at the B site [\[28\]](#page-4-0). In addition, the high symmetry of the structure excludes the possible existence of spontaneous electrical polarization, avoiding a possible coexistence of magnetism and ferroelectricity, differently than other similar double perovskites [\[29](#page-4-0)–32]. Previous works describe the magnetism of the compound in terms of a high temperature ferrimagnetic ordering process (T_N = 272 K) arising from the Fe³⁺-O-Mo⁵⁺ superexchange interaction, resulting in a significant saturation magnetization (1.75 μ_B) and in a very soft (quasi superparamagnetic) character [\[27\]](#page-4-0). Additionally, PFMO has been addressed to show low-field magnetoresistance, ascribed to field-induced suppression of spin fluctuations.

In this work, we have investigated the electric properties of $Pb₂Fe MO₆$ samples, synthetized in HP/HT conditions as reported in ref. [\[27\]](#page-4-0), which crystallize in the Fm-3m space group with lattice parameter $a =$ 7.9609 Å, characterized by a rock-salt ordering of the transition metal cations at the B site. Herein, the asymmetric trend of the magnetoresistivity together with a peculiar evolution of the transport mechanisms is investigated and motivated by the occurrence of the ordered ferrimagnetic state of the compound, as a magnetic field-dependent phenomenology.

2. Materials and methods

High purity (*>*95 %) polycrystalline bulk sample of PFMO was crystallized under HP/HT conditions (5 GPa, 900 ◦C for 1.5 h) in a Walker-type multi-anvil press by solid-state reaction, starting from stoichiometric amounts of PbO, FeO, and MoO₃ powders. The crystalline structure displays partial cation ordering at the B site involving iron and molybdenum, demonstrated by selected-area electron diffraction (SAED), synchrotron and neutron diffraction experiments [\[27\].](#page-4-0)

Transport measurements were performed on an as-synthesized pellet disk (with radius $r = 2$ mm, thinned down to thickness $t = 0.34$ mm), double-side polished to reduce the surface roughness below a few μ m.

Electrical transport was measured in a four-probe (van der Pauw) configuration with an injected DC current of 2 mA using a Keithley 2182A nanovoltmeter and a Keithley 2400 sourcemeter. To investigate the magnetoresistance, we exploited the magnet and the cryostat of a commercial Quantum Design superconducting quantum interference device (SQUID) magnetometer. The sample was brought to 200 K, 150 K and 20 K applying both ZFC and FC protocols starting from RT, the latter in applied field of ± 40 kOe. Once the set-point temperature had been reached, the electrical resistance was measured as a function of the external magnetic field spanning from -40 kOe to $+40$ kOe.

Magnetization vs. temperature has been investigated by using a MPMS-XL SQUID magnetometer on bulk fragments of PFMO in the 80–350 K range with external DC field of 10 Oe. In order to avoid possible spurious signals from Hall effect, the magnetic field was applied in the plane of the sample and all the permutations of contact pairs and current sign have been measured and averaged.

3. Results and discussion

3.1. Electrical transport

The resistivity behavior of PFMO measured as a function of temperature in both the cases of no external magnetic field and under a static field of $H = 2$ T is reported in Fig. 1. The two curves appear very similar and show a continuous enhancement of ρ by decreasing temperature. Noteworthy, such a thermal decrease of the resistivity is not

Fig. 1. Electrical resistivity measured via van der Pauw method on a polycrystalline sample of PFMO between 320 K and 5 K with an applied magnetic field of 2 T (green squares) and with no magnetic field (blue dots). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

describable as a semiconductor thermal activated transport or by any hopping model (see Supporting Information file, Fig. S1-S2-S3). The ρ ranges between 10^{-3} Ω⋅m above RT and 10^{-1} Ω⋅m at around 5 K, highlighting the overall good conductivity of the material.

In order to further investigate the electrical behavior of PFMO, the incremental ratio of the resistivity $\Delta\rho/\Delta T$ vs. temperature has been

Fig. 2. a) Incremental ratio Δρ/ΔT as a function of temperature under the application of 0 T (blue dots) and 2 T (green squares); b) Magnetization as a function of temperature measured with an applied field of 10 Oe (Field-Cooling mode). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

calculated ([Fig. 2a](#page-1-0)). Both the curves, collected with and without the magnetic field applied, display an almost constant and superimposed trend of $\Delta\rho/\Delta T$ in the high-temperature regime, above the Néel tem-perature of PFMO T_N around 275 K [\(Fig. 2b](#page-1-0)). Constant $\Delta \rho / \Delta T$ values (close to zero) imply linear trend (almost flat) of the main function and the observed mismatch with all the investigated transport mechanisms suggest a possible semi-metallic behavior of the material [\[33,34\].](#page-4-0)

This hypothesis is supported by the anomaly recorded in the resistance trend of PFMO when the sample is heated over the long-range magnetic ordering transition T_N , as reported in Fig. 3. This change in the transport property at the onset of the magnetic order has been already reported in Heusler compounds as a fingerprint of the possible semi-metallicity [\[35\]](#page-4-0).

Noteworthy, it should be underlined that when the system transforms into a ferrimagnetic material, the magnetization gradually starts to increase displaying a flex towards positive second derivative when temperature is lowered below 200 K [\(Fig. 2b](#page-1-0)). Concomitantly, when this flex in magnetization is detected, the $\Delta \rho / \Delta T$ starts to continuously increase with comparable T dependence for the measurement with and without positive applied magnetic field, even though with a mild trend not ascribable to any semiconductor transport models (Fig. S1-S2-S3). Hence, it seems that the magnetic order can arise only after a partial localization of a minority of electrons on the valence band of the cations at the B site, probably mainly at the Mo site, conferring to the ion a magnetic character (from Mo^{6+} (d0) to $Mo^{5+}(d1)$) and consequently activating the super-exchange interaction with Fe^{3+} along the Fe^{3+} -O- $Mo⁵⁺$ straight bond. This consideration is in agreement with previous neutron diffraction analyses [\[27\],](#page-4-0) in which the Mo-O bond was reported to continuously evolve until the saturation of the ferrimagnetic resultant. Hence, it can be pointed out that magnetic order is the main responsible for the increase of the resistivity below T_N , leaving thermal effects on the resistivity as a second-order phenomenon.

3.2. Magnetoresistance

In order to investigate in depth the correlation between magnetic order and transport properties in the material, magnetoresistance measurements were performed on the same sample keeping the same orientation of the pellet with respect to the external field.

The magnetoresistance MR%, reported in Fig. 4, has been calculated

Fig. 3. Zoom of the resistance values in the high temperature regime, displaying an anomaly in its trend crossing the ferrimagnetic ordering temperature.

Fig. 4. Magnetoresistance measurements collected between –40 kOe and 40 kOe in different thermal conditions and different field cooling protocols, specifically: a) at 200 K after ZFC (empty dots) and –40 kOe after FC (filled dots); b) at 150 K after ZFC (empty dots) and –40 kOe FC (filled dots); c) at 20 K after –40 kOe FC.

in percentage values as follows:

$$
MR\%(H,T) = 100 \bullet \frac{\rho(H,T) - \rho(0,T)}{\rho(0,T)}
$$

where $\rho(H,T)$ is the resistivity measured at temperature T in the applied magnetic field H, while $p(0,T)$ is the value recorded without the applied field at the same T.

Magnetoresistance measurements have been performed by reaching the temperature setpoints both through ZFC and FC protocols starting from RT, carrying out the FC branch with both positive and negative magnetic field \pm 40 kOe on cooling, then scanning the applied field from − 40 kOe to +40 kOe during the measurement. The results are reported in Fig. 4 and S4. While no effect is detected at RT (not shown in Fig.), magnetoresistivity develops at temperatures below T_N . It is remarkable that the results also depend on the *sign* of the field applied on cooling. Specifically, at 200 K, ZFC-MR shows small MR effects with values around 1.5 % with almost symmetric branches at negative and positive scan fields; on the contrary, the FC-MR with –4T displays a large asymmetric MR effect with an increased loss of around 4.2 % in magnitude at –40 kOe, around three times more intense than the ZFC-MR and, vice versa a quench of the MR for positive fields (Fig. 4a). On the contrary, no effect of FC-MR at 4 T with respect to the ZFC MR is detected (see Fig. S5). Same effects, even if much less intense, are detected also at 150 K (Fig. 4b). Below the completion of the magnetic order process, no asymmetry is observed in the curves while the intensity of the MR effect is maximized, reaching $14%$ at ± 40 kOe after FC at –40 kOe at 20 K (Fig. 4c), a considerably high value respect with other polycrystalline magnetoresistive perovskites [\[36\].](#page-4-0)

These measurements pointed out that in isothermal conditions, across the stabilization of the magnetic order of the material between T_N and 150 K, the system responds differently with respect to the applied field sign, promoting carriers in the conduction band for negative fields and vice versa confining a minority portion of them in the valence band for positive fields.

Such asymmetric behavior could be explained by the presence of exchange bias, feature reported also in $Sr_2FeMoO₆$ ceramics characterized by B/B' antisite disorder or spin glass states [\[37,38\]](#page-4-0). Specifically, magnetoresistance effects have been associated to the grain size of polycrystalline samples [\[39\],](#page-4-0) thus effectively representing an inter-grain tunnelling magnetoresistivity [\[40\].](#page-4-0) However, the studied PFMO samples are characterized by a superparamagnetic-like hysteresis loop, with a negligible (quasi zero) coercive field, as reported in previous literature [\[27\]](#page-4-0), which prevents any possible exchange bias mechanism, consequently excluding this effect from being responsible for the detected asymmetric magnetoresistive trend.

Having said that, the asymmetric trend of the MR suggests a halfmetallic nature of the compound with transport mechanism driven by polarized carriers as it is observed for similar materials [\[41\].](#page-4-0) Consequently, it can be argued that these polarized carriers may be characterized by a spin resultant prevalently oriented in the negative field direction. Moreover, because of this effect vanishes lowering T, the partial localization driven by the magnetic order and connected to the gradual stabilization of Mo-O bond length, acts in re-symmetrizing the conduction band by trapping the negatively polarized electrons at the Bsite. As a result, in the very low field regime, below 150 K, the MR effect has a conventional behavior but with an increased intensity due to a larger confinement of the carriers, which is very sensitive to the application of a proper magnetic field which acts in promoting conductivity with enhanced yield, independently from its sign.

Since magnetoresistance has already been investigated in PFMO samples with disorder on B site, but in that case no asymmetric response with the sign of the magnetic field has been observed [\[28\],](#page-4-0) the current observations point out the role of the cation order at the B site of the double perovskite (which means ordered distribution of Fe-O-Mo chains) as the driving force of the spin-polarized carriers halfmetallicity of the material. In addition, even the intensity of the magnetoresistance seems to depend on the cation order too as, particularly, at 20 K in B-ordered PFMO the absolute MR% is around 50 % larger than B-disordered PFMO [\[28\]](#page-4-0).

4. Conclusions

The electrical properties of the double perovskite $Pb_2FeMoO₆$ (PFMO) have been investigated as a function of temperature and external magnetic field. This Pb-based double perovskite can be only obtained in HP/HT environments due to the difficulty of stabilizing the $Mo⁵⁺$ in octahedral coordination and it is characterized by the predominant Fe^{3+} and Mo^{5+} ordering on the B-site (80 % of the ions), leading to a long-range ferrimagnetic structure close to RT.

At RT PFMO is possibly semi-metallic, with very low resistivity and an anomalous resistive trend located around the T_N . Below T_N , the localization of a minority fraction of electrons at the Mo site is mainly driven by the long-range magnetic order process and determines an increase of the resistivity between T_N and 150 K. In this thermal regime, the system behaves as a half-metal, displaying asymmetric magnetoresistivity with polarized carriers promoted by negative magnetic fields and, vice versa, quenched MR for positive magnetic fields. Asymmetric magnetoresistance and half-metallicity are in PFMO cationorder related phenomena as they disappear once the Mo-O bond length stabilizes at lower temperatures, determining the completion of the ferrimagnetic resultant and the conclusion of the partial localization mechanism. Below this threshold, asymmetric magnetoresistivity is suppressed but record values of MR have been collected in the cationordered phase of PFMO with MR loss of 14 % at 20 K, about 50 % larger than in the disordered phase.

The interconnection between its magnetic and electrical properties allows us to define PFMO as a proper multifunctional material while its spin-polarized carriers-half metallicity makes it a possible candidate for spintronic application.

CRediT authorship contribution statement

Chiara Coppi: Conceptualization, Data curation, Formal analysis, Investigation, Validation, Visualization, Writing – original draft, Writing – review & editing. **Riccardo Cabassi:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology. **Francesco Mezzadri:** Conceptualization, Data curation. **Massimo Solzi:** Conceptualization, Data curation, Writing – original draft, Writing – review & editing. **Francesco Cugini:** Conceptualization, Data curation. **Edmondo Gilioli:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Supervision, Writing – original draft, Writing – review & editing. **Davide Delmonte:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Project administration, Supervision, Validation, Visualization, Writing – original draft, Writing – review $&$ editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Appendix A. Supplementary data

Supplementary data to this article can be found online at [https://doi.](https://doi.org/10.1016/j.jcrysgro.2024.127629) [org/10.1016/j.jcrysgro.2024.127629](https://doi.org/10.1016/j.jcrysgro.2024.127629).

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