Suppressed antiferromagnetic ordering and enhanced electrical conduction in doped iridate \( \text{Sr}_2\text{Pr}_x\text{IrO}_4 \)

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\( \text{Sr}_2\text{IrO}_4 \) has been shown to exhibit a novel \( J_{\text{eff}} = \frac{1}{2} \) Mott spin–orbit insulating state with antiferromagnetic ordering. In the present investigation, the effects of Pr substitution at Sr-site on structural, magnetic and electrical properties are studied in \( \text{Sr}_2\text{Pr}_x\text{IrO}_4 \). Pr-doped samples still retain the insulating behavior, but with the increase of Pr doping concentration, the resistivity decreases. The magnetic properties show that long-range antiferromagnetic order is collapsed when the Pr doping content increases up to 20\%, which may results from a competition between the AFM (Ir 5d electrons) and ferromagnetic (Pr 4f electrons) coupling. In addition, Pr doping induces a spin glasslike state at low temperatures.

1. Introduction

In recent years, Ir-based oxides have been widely studied because of their numerous interesting properties. In these materials, the energies of spin–orbit coupling (SOC), Coulomb repulsive interactions (\( U \)), crystalline electric field and the Hund’s coupling are comparable so that they compete and cooperate with each other, resulting in exotic phases [1]. The layered \( \text{Sr}_2\text{IrO}_4 \) is of typical interest due to its exotic \( J_{\text{eff}} = \frac{1}{2} \) ground state. The Ir in \( \text{Sr}_2\text{IrO}_4 \) has a 5d\(^5\) electronic configuration and an ionic state of Ir\(^{4+}\). The crystal-field effect in the octahedral IrO\(_6\) splits the 5d orbital into the low-lying \( t_{2g} \) and high-lying \( e_{g} \) levels; then, in the presence of a strong SOC effect, the \( t_{2g} \) level is further split into a fully filled \( J_{\text{eff}} = \frac{3}{2} \) quartet and a partially filled \( J_{\text{eff}} = \frac{1}{2} \) doublet state near the Fermi level, where the \( J_{\text{eff}} = \frac{1}{2} \) band is split into an upper Hubbard band (UHB) and a lower Hubbard band (LHB) due to on-site Coulomb interactions [2]. The \( J_{\text{eff}} = \frac{1}{2} \) band is so narrow that even a modest on-site Coulomb repulsion \( U \sim 0.5 \) eV owing to the extended nature of 5d-electron orbitals is sufficient to open a small Mott gap \( \sim 0.1 \) eV, leading to an insulating state [2, 3]. Besides, \( \text{Sr}_2\text{IrO}_4 \) exhibits antiferromagnetic (AFM) ordering below \( T_{\text{C}} = 240 \) K. However, the moments are significantly canted, resulting in weak ferromagnetism with a saturation moment of \( \sim 0.1 \mu_B/\text{Ir} \) [4–6].

Mott insulating state in \( \text{Sr}_2\text{IrO}_4 \) is due to a cooperative interplay of spin–orbit coupling and on-site Coulomb interactions. The change of the two factors probably tunes the ground state, leading to new exotic phases. In addition, the similarity of the crystal structure and the magnetic insulating state with the layered cuprate \( \text{La}_2\text{CuO}_4 \) has excited the great interest in realizing superconductivity via doping [7, 8]. Therefore, many investigations have been carried out to discover the possible superconductivity and other exotic properties by using electron or hole doping. For example, the suppression of the long-range antiferromagnetic order and the occurrence of metallic behavior have been observed in electron-doped \( \text{Sr}_1-x\text{La}_x\text{IrO}_4 \) [9–11]. Progressive substitution of Ca\(^{2+}\) onto Sr\(^{2+}\) sites in \( \text{Sr}_1-x\text{Ca}_x\text{IrO}_4 \) samples has driven the structure to evolve across a series of structure types, each with insulating ground states [12]. It has been found that Sm-doped \( \text{Sr}_2-x\text{Sm}_x\text{IrO}_4 \) samples still retain the insulating behavior, but with the increase of Sm doping, the resistivity presents a non-monotonic electronic transport, as well as magnetic properties [13]. The Rh substitution in \( \text{Sr}_2\text{Ir}_1-x\text{Rh}_x\text{O}_4 \) has brought about a paramagnetic (PM) and metallic state when the concentration of Rh increases up to a critical
value [14, 15]. The Ru substitution in Sr$_2$Ir$_{1-x}$Ru$_x$O$_4$ has caused suppression of magnetic ordered and the insulating state [16, 17]. Substitution of Tb in Sr$_2$Ir$_{1-x}$Tb$_x$O$_4$ has shown that 3% Tb doping effectively suppresses the long-range collinear AFM transition from $T_N = 240$ K to zero but retains the insulating behavior [18]. In Cu doped Sr$_2$Ir$_{1-x}$Cu$_x$O$_4$ system, it has been found that the long-range magnetic ordering is weakened and the $x = 0.2$ sample shows almost PM-like behavior, while the electric transport property retains the insulating state in all the doped samples [19].

In spite of the aforementioned efforts, the investigation on the evolution of magnetic and electric transport properties with doping is still on the threshold. It is necessary to learn more about the effect of doping on the ground state, and it is yet to explore how robust the gapped $J_{el} = 1/2$ state is to perturbations (such as doping), which may induce novel phases. In this paper, we have investigated the magnetic and electric properties in Pr doped Sr$_2$Ir$_{1-x}$Pr$_x$O$_4$ system. The long range AFM order is suppressed with increasing doping. The magnetism may result from a competition between the AFM (Ir 5$d$ electrons) and ferromagnetic (FM) (Pr 4$f$ electrons) coupling. The transport properties can be explained by dividing the measurement temperature into three ranges. With the increase of doping content, the resistivity decreases significantly, but still keep the insulating state.

2. Experiment

Polycrystalline of Sr$_2$Ir$_{1-x}$Pr$_x$O$_4$ samples were synthesized by conventional solid state method. Stoichiometric high purity powders of SrCO$_3$, IrO$_2$, and Pr$_2$O$_3$ were mixed, ground, and heated in a furnace at 1000 °C for 24 h, followed by calcining at 1250 °C for about two days with several intermediate grindings. The x-ray diffraction patterns were recorded in a PANalytical x-ray diffractometer using the Cu Kα radiation at room temperature. Magnetization measurements were performed using Quantum Design 7 T SQUID magnetometer. The electrical resistivity and heat capacity were performed using Quantum Design physical property measurement system (PPMS).

3. Results and discussion

Figure 1 (a) shows the x-ray diffraction patterns of the sintered samples. The XRD patterns show that the materials have a pure phase of reduced tetragonal structure with $I4_1/acd$ space group. Rietveld refinements of the
structure were performed with Fullprof program. The representative refinement for parent material is given in figure 1(b). The corresponding parameters obtained from the Rietveld refinements are shown in figures 1(c) and (d). We find from figure 1(c) that lattice parameter a does not change much, while c decreases with x. The decreased c/a ratio in figure 1(d) imply that tetragonal distortion is reduced with progressive doping of Pr. In addition, the Ir–O–Ir bond angle increases with increasing doping (see figure 1(d)), indicating less distorted IrO$_6$ octahedra. It has been established that Ir–O–Ir bond angle is of prime importance to the electronic and magnetic structure of Sr$_2$IrO$_4$ [3].

Figures 2 and 3 show the temperature dependence of zero-field-cooling (ZFC) and field-cooling (FC) susceptibility ($\chi$-T) curves of parent material Sr$_2$IrO$_4$ and doped Sr$_{2-x}$Pr$_x$IrO$_4$ series, respectively. For the undoped sample, a sharp increase around 240 K has been observed as the temperature decreases, which indicates a FM-like transition occurs around $T_N = 240$ K. Below this temperature, the magnetic state has been
determined to be canted AFM [2, 20]. Interestingly, with Pr substitution, the magnetic transition temperature $T_N$ is decreased. When the doped content increased up to 20%, the $T_N$ disappears and the $\chi$-$T$ curve almost presents a PM behavior. That is to say, 20% Pr doping effectively suppresses the long-range AFM transition temperature. The suppression of long-range magnetic ordering in Sr$_2$IrO$_4$ has also been observed when doping with other elements, such as Sr$_2$Ir$_{1-x}$Rh$_x$O$_4$ [15], Sr$_2$Ir$_{1-x}$Ru$_x$O$_4$ [16, 17] Sr$_2$Ir$_{1-x}$Tb$_x$O$_4$ [18] Sr$_2$Ir$_{1-x}$Cu$_x$O$_4$ [19] and (Sr$_{1-x}$La$_x$)$_2$IrO$_4$ [10]. However, there is a cusp at $T_F$ in the ZFC magnetization curve for the doped samples at low temperatures. The observed $T_F$ cusp and inflection in irreversibility support the appearance of a spin glass state at low temperature in doped samples [10]. The spin glass transition shifts to low temperature with increased Pr content, from $T_F = 15$ K for $x = 0.05$ to $T_F = 10$ K for $x = 0.15$, and at last diminishes with continued doping for $x = 0.2$. The suppressed $T_N$, $T_F$, and the irreversibility are due to the weakening of AFM order, which can be determined by the Curie-Weiss fitting below. The rapid suppression of the AFM state is due to a different valence state of Pr and Sr ions and a change in the electron correlation, crystalline electric field effects, and Hund’s rule coupling.

In order to investigate the magnetic state in all the samples, we have plotted inverse susceptibility as a function of temperature for this series in figure 4. The change in slope in $\chi^{-1}$-$T$ curves within the PM state is similar with that of Sr$_2$Ir$_{1-x}$Cu$_x$O$_4$, which should be related to anisotropic magnetic interaction [19]. The susceptibility $\chi$.

Figure 4. Inverse susceptibility $\chi^{-1}$ as a function of temperature for Sr$_{2-x}$Pr$_x$IrO$_4$ series. The solid line is the fitting using Curie-Weiss law.
follows a Curie–Weiss law as expressed by the function $\chi = C / (T - \theta_{cw})$, where $\theta_{cw}$ the Curie-Weiss temperature, and $C$ the Curie constant. The straight lines in figure 4 are due to fitting with the Curie-Weiss law. The calculated $\theta_{cw}$ and the effective magnetic moment $\mu_{eff}$ calculated from $C$ are displayed in table 1. A successive decrease in $\theta_{cw}$ with $x$ is consistent with the decreasing $T_N$, indicating the ferromagnetism associated with the AF spin canting is suppressed. Such relatively large value of $\theta_{cw}$ in the highest doped sample without magnetic ordering implies strong suppression of magnetic ordering or spin frustration, which may stem from a competition between the AFM (Ir 5$d$ electrons) and FM (Pr 4$f$ electrons) interactions. The frustration indexes are shown in table 2.

Considering there are two kinds of magnetic ions in the doped samples, we can describe the effective magnetic moment $\mu_{eff}$ / f. u. as

$$\mu_{eff}^2 = \mu_{Ir}^2 + x\mu_{Pr}^2.$$  

Table 1. Fitting parameters of resistivity in different temperature regions.

| $x$ | $\alpha$ (K$^{-1}$) | $\rho_0$ (Ω cm) | $\Delta$ (mev) | $\rho_0$ (Ω cm) | $T_N$ (K) | $\rho_0$ (Ω cm) |
|----|------------------|------------------|----------------|----------------|----------|----------------|
| 0  | $1.03 \times 10^{-2}$ | 140.22           | 80.43          | 1.76           | $3.27 \times 10^7$ | 6.11 $\times 10^{-9}$ |
| 0.05 | $7.80 \times 10^{-3}$ | 8.19             | 55.96          | 0.38           | $2.10 \times 10^6$ | 5.98 $\times 10^{-5}$ |
| 0.1 | $7.55 \times 10^{-3}$ | 1.03             | 56.94          | 0.05           | $1.85 \times 10^6$ | 1.03 $\times 10^{-5}$ |
| 0.15 | $7.13 \times 10^{-3}$ | 0.57             | 57.01          | 0.03           | $9.02 \times 10^3$ | 4.25 $\times 10^{-5}$ |
| 0.2 | $3.26 \times 10^{-3}$ | 0.09             | 18.87          | 0.03           | $2.47 \times 10^3$ | 9.17 $\times 10^{-3}$ |

Figure 5. Field dependence of magnetization for Sr$_{2-x}$Pr$_x$IrO$_4$ samples at 10 K.
Using the value $\mu_{\text{Ir}} = 0.326 \, \mu_B$ of Ir$^{4+}$ in Sr$_2$IrO$_4$, we estimate the $\mu_{\text{Pr}}$ for the doped samples, and obtain that these values are 1.20 $\mu_B$, 1.72 $\mu_B$, 2.69 $\mu_B$ and 3.09 $\mu_B$ for $x = 0.05$, 0.1, 0.15 and 0.2 respectively. With $J = 4$, the trivalent Pr$^{3+}$($4f^2$) ion has an effective moment of 3.2 $\mu_B$/Pr. The weakened $\mu_{\text{Pr}}$ suggests a significant interaction between Ir 5d and Pr 4f electrons.

Field dependence of magnetization at 10 K is given in figure 5. For parent material, $M$-$H$ curve presents a hysteresis, reflecting some FM interactions in the compound, which should be originated from canted AFM order. With Pr substitution increasing, the S-shaped canted AFM like behavior and $H_c$ in the $M$-$H$ plot weaken gradually, and the $x = 0.2$ sample shows an almost linear variation with the magnetic field. This means that ferromagnetism associated with the canted AF state is weakened. The hysteresis with enhanced linear behavior for the Pr doped samples demonstrates the existence of magnetic frustration.

The temperature dependence of the resistance is shown in figure 6. The room-temperature resistance of our parent sample is 6.49 $\Omega$ cm, which is of the same order of magnitude as that reported previously [19]. All the samples exhibit an insulating behavior in the whole temperature measured. With the increase of Pr doping, the electrical resistivity is decreased, indicating enhanced conductivity with doping. The increasing Ir–O–Ir bond angle with doping induces reduced tetragonal distortion, thus the overlap of d-orbital electrons is enhanced and electron hopping is more favorable [21, 22]. Besides, the Pr atoms in Sr$_2$IrO$_4$ result in the high electron concentration compared with the parent material, which will lead to the enhancement of density of states at

| Sample $x$ | $\theta_{\text{CW}}$ (K) | $C$ (emu K/Oe mol) | $\mu_{\text{eff}}$ (\mu_B/\text{f.u.}) | $f$ | $H_c$ (Oe) |
|------------|----------------|-----------------|----------------|------|-----------|
| $x = 0$    | 247            | 0.013           | 0.326          | 1.03 | 3065      |
| $x = 0.05$ | 208            | 0.022           | 0.422          | 0.92 | 1582      |
| $x = 0.1$  | 159            | 0.050           | 0.635          | 0.76 | 831       |
| $x = 0.15$ | 92             | 0.149           | 1.093          | 0.53 | 627       |
| $x = 0.2$  | 41             | 0.252           | 1.419          | —    | 238       |

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Fermi level. Both the two factors play an important role in the enhanced electrical conduction \[21\]. The persisting insulating state at the highest doped level suggests that the band gap is not fully closed.

In order to understand the transport nature, we have further fitted the resistivity data with different transport models. This fitting is similar to previous reports \[13, 23, 24\]. In the high-temperature region (210–290 K), the resistivity is found to be described by the equation:

\[
\rho(T) = \rho_0 \exp \left( -\alpha T \right)
\]

The linear fit for \(\ln \rho\) versus \(T\) curves are shown in figure 6(b). The \(\rho_0\) and \(\alpha\) constants obtained by fitting are listed in table 1. The linear dependence of \(\ln \rho\) on \(T\) indicates a weak electronic localization in quasi-2D materials. The decreased \(\alpha\) suggests that the strength of electron interactions becomes weak with doping.

In the middle-temperature region (100–200 K), the resistivity exhibit an Arrhenius-type behavior:

\[
\rho(T) = \rho_0 \exp \left( \frac{\Delta}{2k_B T} \right)
\]

The linear dependence of \(\ln \rho\) on \(T^{-1}\) is plotted in figure 6(c). The obtained results \(\rho_0\) and \(\Delta\) are listed in table 1. The calculated gap (80.43 meV) for \(\text{Sr}_2\text{IrO}_4\) is slightly larger than those of previous reports \[23–25\]. The small value of gap implies that a pseudogap arising from localization of electronic states. With the increase of the Pr doping level, the gap exhibits a systematic decrease, confirming that the Pr-doping changes the electrical structure and thus enhances conductivity.

In the low-temperature region (35–80 K), the resistivity can be fitted to a VRH-type behavior expressed by the equation:

\[
\rho(T) = \rho_0 \exp \left( \frac{T_0}{T} \right)^{1/4}
\]

\(\ln \rho\) versus \(T^{-1/4}\) plot in this region is given in figure 6(d). The calculated parameters are listed in table 1. VRH model means that the localization of carriers by disorder and defects is responsible for the conductivity behavior in the low temperature region.

The characterization temperature \(T_0\) of the VRH model can be given by \[26\]:

\[
T_0 = \frac{18}{k_B l N(E_F)}
\]

where \(k_B\) is Boltzmann constant, \(l\) denotes the localization length, and \(N(E_F)\) represents the density of states at the Fermi level. Based on previously estimated value of \(N(E_F)\) at \(10^{26} \text{eV}^{-1} \text{m}^{-3}\) in \(\text{Sr}_2\text{IrO}_4\) \[26\], we calculated localization length \(l\) of the present materials ranging from 0.74 Å to 17.4 Å. The values are similar to the order of the Ir–O bond length (~2 Å) of \(\text{Sr}_2\text{IrO}_4\) \[13\], confirming the validity of the VRH mechanism. We find that \(T_0\) decreases with increasing the Pr concentration. Equation (5) shows that both \(N(E_F)\) and \(l\) will contribute to the
variation of $T_\varphi$. However, a large change in resistivity in the $\mathrm{Sr}_2-x\mathrm{Pr}_x\mathrm{IrO}_4$ series reveals that $N(E_F)$ has a main contribution for change of $T_\varphi$.

In order to investigate more physical properties of the samples, we measured the low-temperature specific heat. From previous reports, we have known that the heat capacity of $\mathrm{Sr}_2\mathrm{IrO}_4$ shows no corresponding anomaly at the magnetic ordering temperature 240 K, which is one of the characters of the $I_{\text{eff}} = 1/2$ insulator [27, 28]. Thus we only measured the heat capacity of a selective sample, i.e., $x = 0.1$ sample in the range of 2–20 K, which is shown in figure 7(a). A plot of $C(T)/T$ versus $T^2$ indicates that the lattice contribution is not well-described by a simple Debye model (see figure 7(b)). The specific heat between 10 K and 20 K can be fitted to the following equation [29, 30]:

$$C/T = \gamma + \beta_1 T^2 + \beta_2 T^4$$  \hspace{1cm} (6)

where the first term is the electronic contribution, the second term is the phonon contribution according to the Debye approximation, and the third term is the deviation from the linear dispersion of the acoustic modes. The corresponding fitting is shown in the solid line in figure 7(b). The fitted parameters are $\gamma = 3.47 \text{ mJ mol}^{-1} \text{ K}^{-2}$, $\beta_1 = 7.39 \times 10^{-4} \text{ mJ} \text{ mol}^{-1} \text{ K}^{-4}$, and $\beta_2 = 2.27(3) \times 10^{-9} \text{ mJ} \text{ mol}^{-1} \text{ K}^{-6}$. The Sommerfeld constant $\gamma$ here is slightly larger than that of the parent material $\mathrm{Sr}_2\mathrm{IrO}_4$ in previous reports ($\gamma = 1.8 \text{ mJ} \text{ mol}^{-1} \text{ K}^{-2}$ in [31]) and 2 mJ mol$^{-1}$ K$^{-2}$ in [23]). The increased $\gamma$ value is due to the enhancement in the density of electronic states at Fermi level, suggesting enhanced conductivity, which is consistent with the results of electric resistivity. We also note that there is an upturn in $C(T)/T$ for $x = 0.1$ below 10 K, which may be due to spin degrees of freedom of the Pr$^{3+}$ ($J = 4$) states. This is similar to the heat capacity of the $\mathrm{Sr}_2\mathrm{Ir}_{1-x}\mathrm{Tb}_x\mathrm{O}_4$ samples [18].

4. Conclusion

In conclusion, we have studied the magnetism and transport properties in $\mathrm{Sr}_2-x\mathrm{Pr}_x\mathrm{IrO}_4$ polycrystalline samples in detail. With the Pr$^{3+}$ doping content increasing, the lattice parameter decreases, and tetragonal distortion is reduced along with the Ir–O–Ir bond angle increasing. A systematic decrease in $\theta_{\text{cw}}$ and $T_N$ indicates the ferromagnetism associated with the canted AF state is weakened. Furthermore, long-range AF order is completely suppressed in $x = 0.2$ sample. In addition, electron doping induces a low-temperature phase transition consistent with a spin glasslike state. The magnetism may result from a competition between the AFM Ir–Ir and FM Pr–Pr interactions. The transport properties can be explained by dividing the measurement temperature into three ranges. With the increase of doping concentration, the resistivity decreases significantly. This can be further verified by the heat capacity result.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (Grant No. 11604071), Natural Science Foundation of the Education Department of Anhui Province (Grant No. 2018jxm0377), the Fostering Master’s Degree Empowerment Point Project of Hefei University (Grant No. 2018xs03), the Natural Science Foundation of Hefei University (Grant Nos. 2017jxm013, 18ZR13ZDA and 2016dr02) and the National Undergraduate Training Programs for Innovation and Entrepreneurship (201811059098).

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