



New oxypnictide superconductors: $\text{PrOFe}_{1-x}\text{Co}_x\text{As}$

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ABSTRACT

Oxypnictides of the type $\text{PrOFe}_{1-x}\text{Co}_x\text{As}$ ($x \leq 0.3$) were synthesized for the first time by the sealed tube method. All the compounds were found to be monophasic and crystallize in the tetragonal ZrCuSiAs type structure (space group= $P4/nmm$) and the lattice parameters (**a** and **c**) decrease with increase in cobalt content. Mössbauer measurements of the compounds indicate low spin Fe^{2+} in tetrahedral coordination. Resistivity and magnetization studies reveal superconducting transitions in compounds with $x=0.05, 0.10$ and 0.15 , with maximum transition temperature (T_c) at ~ 14 K in the compound with $x=0.1$. The variation of resistivity with temperature under different magnetic field has been studied to estimate the upper critical field (H_{c2}) (~ 50.2 T for the $x=0.1$ composition). The Seebeck and Hall coefficient (R_H) suggests electron type charge carriers in these compound and the charge carrier density increases with increase in Co-doping.

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1. Introduction

The discovery of superconductivity at 26 K in an unexpected FeAs-based oxypnictide compound ($\text{La}(\text{O}/\text{F})\text{FeAs}$) [1] has attracted a great deal of attention among the scientific community. The family of the FeAs-based superconductors can be divided into three classes. The first class of these superconductors has the general formula LnOFeAs (Ln =the rare-earth metals), which is abbreviated as FeAs-1111 family. The second class has a general formula AFe_2As_2 ($A=\text{K}, \text{Ca}, \text{K}/\text{Sr}$ and Ca/Sr) and is denoted as A122 systems [2–3]. The third type MFeAs (M =alkali metals) has an infinite layered structure and denoted as M111 system [4–5]. For FeAs-1111 phase, the transition temperature (T_c) has been found to increase with the substitution of smaller rare-earth metal ions like Ce [6–7], Pr [8], Nd [9], Sm [10] and Gd [11] with the highest T_c reported for $\text{Sm}(\text{O}/\text{F})\text{FeAs}$ at 55 K [12]. These compounds with the general formula LnOFeAs (Ln =rare-earth metal) crystallize in the tetragonal ZrCuSiAs type structure (space group= $P4/nmm$) [13], which is a filled variant of the well-known PbFCl type structure consisting of alternate layers of Ln-O and Fe-As layers where the former acts as a charge reservoir and the latter acts as a charge carrier [14]. The parent LnOFeAs compounds are semi-metallic in nature and show an anomaly around 130–150 K (depending on the rare-earth metal) in the resistivity and dc magnetization measurements due the structural distortion from

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tetragonal to orthorhombic/monoclinic symmetry followed by a magnetic transition leading to a spin density wave (SDW) with antiferromagnetically coupled 'Fe' spins. There have been some reports that the magnetic rare-earth spins like Pr^{3+} in PrOFeAs order antiferromagnetically at much lower temperature (~ 14 K) with the Pr moments aligned along the c -axis [15].

The structural distortion and SDW in LnOFeAs compounds can be suppressed with the evolution of superconductivity on doping with electrons or holes. Electrons are injected in the FeAs layers in the oxypnictides by replacing O^{2-} ion with F^- ion [1] or Ln^{3+} with Th^{4+} [16] ions and also by introducing oxygen vacancies [17]. Relatively fewer examples of hole doping (substitution of Ln^{3+} ions by Sr^{2+}) are known in the literature in 1111-system. The highest transition temperature achieved by hole doping is 26 K [18] as compared to 55 K in electron-doped superconductors. We have earlier shown that superconductivity can be realized by doping KF and NaF in LaOFeAs [19–20]. We have also reported the enhancement in transition temperature (T_c) and upper critical field (H_{c2}) in Sb-doped $\text{La}(\text{O}/\text{F})\text{FeAs}$ [21]. It is interesting to note that superconductivity can also be induced by cobalt doping at the iron site [22–24] in LnOFeAs and AFe_2As_2 , which is a surprising fact since the transition temperature is lowered in the well-known cuprate superconductors on substitution of magnetic ions at the Cu sites. The structural distortion and SDW in LnOFeAs and AFe_2As_2 type compounds can be suppressed by the substitution of cobalt ions at the iron sites. The maximum T_c in Co-doped LnOFeAs compound was observed at ~ 15 K [24] in $\text{SmO}(\text{Fe}/\text{Co})\text{As}$ superconductor, which is quite low as compared to T_c observed in $\text{Sm}(\text{O}/\text{F})\text{FeAs}$ (max $T_c=55$ K)[12] compound. On the other hand the maximum T_c was observed at 22 K [25] in

cobalt-substituted $\text{Ba}(\text{Fe}/\text{Co})_2\text{As}_2$ (FeAs-122). The substitution of cobalt at iron sites in AFe_2As_2 compounds leads to decrease in the *c*-lattice parameter while the *a*-lattice parameter remains nearly constant [26]. A similar trend was observed in case of cobalt-doped LnOFeAs [22,24].

In this paper, we have investigated the effect of cobalt doping at the iron sites of semimetallic PrOFeAs on the structural and transport properties. The transition temperature (T_c) varies with Co-content with a maximum T_c of ~ 14.2 K in $\text{PrOFe}_{0.9}\text{Co}_{0.1}\text{As}$. We present temperature-dependent thermoelectric power (TEP) and Mössbauer spectroscopic data complemented by Hall coefficient data on the above oxypnictides. The upper critical field has also been estimated of these novel superconductors.

2. Experimental

Polycrystalline samples with nominal compositions of $\text{PrOFe}_{1-x}\text{Co}_x\text{As}$ were synthesized by sealed tube method using high-purity Pr, Pr_6O_{11} , Co_3O_4 , FeAs and As. FeAs was obtained by heating Fe and As chips in evacuated sealed silica tubes at 800°C for 24 h. The reactants were weighed in stoichiometric amounts and sealed in evacuated silica (10^{-4} Torr) tubes and heated at 950°C for 24 h. The resulting powder was ground and compacted into disks. The disks were wrapped in Ta foil and sealed in evacuated silica ampoules and sintered at 1100°C for 48 h and then cooled to room temperature. All the chemical manipulations were performed in a nitrogen-filled glove box. Powder X-ray diffraction (PXRD) studies were carried out on a Bruker D8 Advance diffractometer with Ni-filtered $\text{CuK}\alpha$ radiation using a scan speed of 1 s and scan step of 0.05° . The lattice parameters were obtained from a least squares fit to the observed *d* values using the PowderCell software [27].

Mössbauer spectra were recorded in transmission geometry using a constant acceleration-type Mössbauer spectrometer with a 10-mCi ^{57}Co source in Rh matrix for ^{57}Fe spectra. A Xe-filled proportional counter was used as the detector. The data were acquired in the MCS mode in a multichannel analyzer containing 1024 channels. The velocity scale was calibrated using a ^{57}Co source and a metallic iron foil absorber. Mössbauer spectra of all the samples were recorded at 298 and 20 K. The low temperature measurements were carried out using a Janis, closed cycle refrigerator fitted with a special anti-vibration stand. The spectra were fitted by a least squares fitting program with Lorentzian profiles. All the isomer shift values were calculated with respect to the center of the α -Fe spectrum.

Four probe resistivity, magnetic and Hall-effect measurements were carried out using a Cryogenic 8 T Cryogen-free magnet in conjunction with a variable temperature insert (VTI). Contacts were made using 44 gauge copper wires with air drying conducting silver paste. For magnetic measurements, the magnetic field (0–5 T) was applied perpendicular to the probe current direction and the data were recorded during the warming cycle with heating rate of 1 K/min. The inductive part of the magnetic susceptibility was measured via a tunnel diode-based rf (2.3 MHz) penetration depth technique [28]. A change in magnetic state of the sample results in a change in the inductance of the coil and is reflected as a shift in the oscillator frequency, which is measured by an Agilent 53131A counter. The thermoelectric power measurement was carried out by bridge geometry in the temperature range 13–300 K. The samples were directly attached to two Au/Fe+Chromyl thermocouple using silver paint. The voltage difference, ΔV , between hot and cold ends of the sample was measured by a Keithley 2182 Nanovoltmeter using copper wires.

In an effort to minimize errors, we have used very thin, undeformed, pieces of the sintered disk for the Hall measurements. No knowledge of the geometric dimensions of the sample is needed in this procedure to calculate the thermopower, the only requirement being an accurate measure of the temperature gradient and voltage between the same points of the sample.

3. Results and discussion

Fig. 1(i) shows the powder X-ray diffraction (PXRD) patterns of the compounds with nominal composition, $\text{PrOFe}_{1-x}\text{Co}_x\text{As}$ ($0 < x < 0.3$). All the observed reflections could be satisfactorily indexed based on the tetragonal ZrCuSiAs (space group= $P4/nmm$) type structure and hence testify the monophasic nature of these compounds. The refined *c*-lattice parameter shrinks significantly while the *a*-lattice parameter decreases slightly with an increase in Co-substitution (Fig. 1(ii)). Similar behavior has also been observed earlier in the compounds with other rare-earths ($\text{Ln}=\text{La}$, Ce and Sm) [22–23] reflecting the substitution of Co^{2+} in place of Fe^{2+} .

The ^{57}Fe Mössbauer spectra for PrOFeAs and $\text{PrOFe}_{0.9}\text{Co}_{0.1}\text{As}$ compounds as shown in Fig. 2. The corresponding hyperfine parameters are given in Table 1. The observed isomer shifts (δ) can be attributed to low spin Fe^{2+} state in tetrahedral

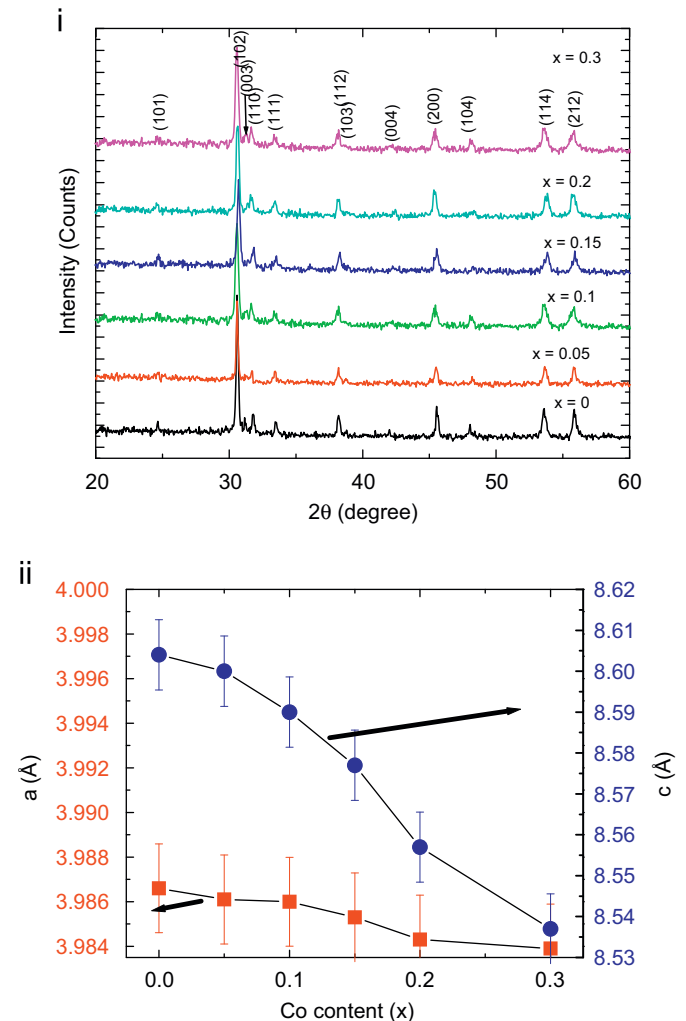


Fig. 1. (i) Powder X-ray diffraction patterns (PXRD) of $\text{PrOFe}_{1-x}\text{Co}_x\text{As}$ sintered at 1100°C and (ii) variation of lattice parameters by short bars ($\sim 0.1\%$) with doping for $\text{PrOFe}_{1-x}\text{Co}_x\text{As}$.

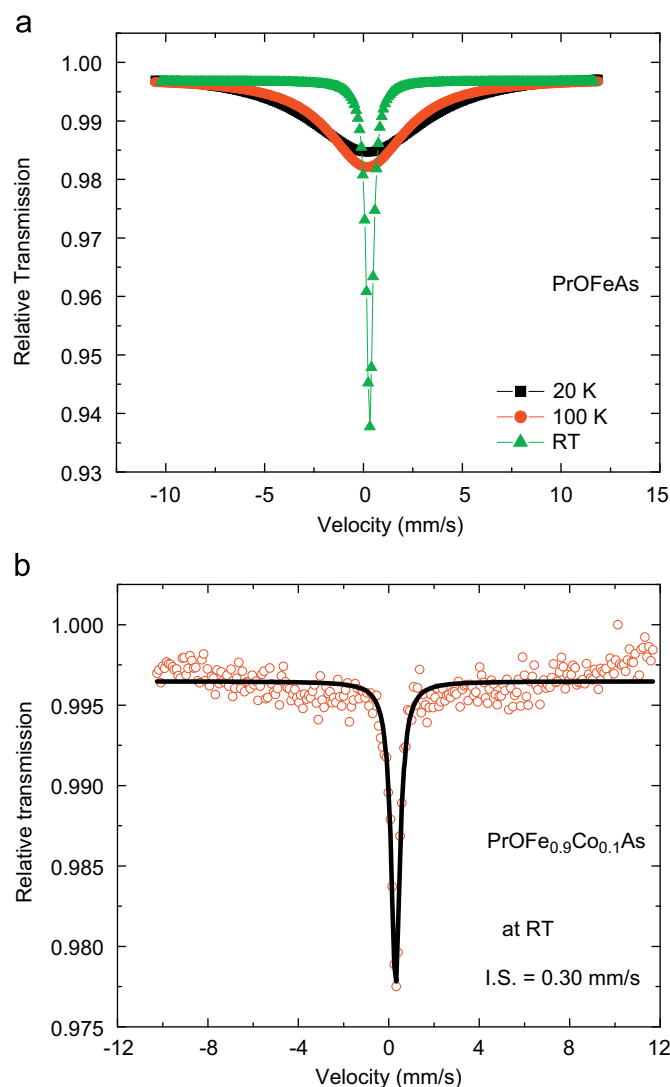


Fig. 2. ^{57}Fe Mössbauer spectra of (a) PrOFeAs and (b) PrOFe_{0.9}Co_{0.1}As.

Table 1
Hyperfine parameters for PrOFeAs and PrOFe_{0.9}Co_{0.1}As.

Sl. no.	Compound	Temperature (K)	Isomer shift (mm/s)	FWHM (mm/s)
1	PrOFeAs	298	0.32	0.40
2	PrOFeAs	100	0.18	4.7
3	PrOFeAs	20	0.23	7.2
4	PrOFe _{0.9} Co _{0.1} As	298	0.30	0.46

coordination. The significant decrease in the value of isomer shift δ for PrOFeAs below 100 K indicates the structural distortion from tetragonal to orthorhombic symmetry [29]. Both PrOFeAs and PrOFe_{0.9}Co_{0.1}As compounds show a clear sharp singlet at room temperature, which suggests that both the compounds are not magnetically ordered at room temperature. The sharp singlet also confirms the absence of any iron bearing secondary phases like FeAs and Fe₂As (impurity phase would give either a sextet pattern or a broadened single/double line absorption pattern) as observed in earlier reported LnOFeAs compounds [1,19]. In Fig. 2(a), it can be seen that as the temperature is lowered the intensity of the singlet pattern for PrOFeAs progressively reduces and the line width increases, which is a clear signature that the sample is

undergoing magnetic relaxation and undergoes magnetic ordering though it did not go to the blocked state till 20 K.

Fig. 3(i) shows the temperature dependence of resistivity for (a) PrOFeAs, (b) PrOFe_{0.95}Co_{0.05}As, (c) PrOFe_{0.9}Co_{0.1}As, (d) PrOFe_{0.85}Co_{0.15}As, (e) PrOFe_{0.8}Co_{0.2}As and (f) PrOFe_{0.7}Co_{0.3}As. For the parent compound, an anomaly characterized by a drop in resistivity was observed at ~ 155 K, which is clearly observed from the temperature dependence of the derivative of the resistivity as shown in Fig. 4, which is qualitatively similar to a previous report [30]. This anomalous resistivity drop is associated with a structural distortion from tetragonal to orthorhombic symmetry. Below the occurrence of SDW, the resistivity drops steeply and then becomes almost independent of temperature, below ~ 84 K. A down turn was observed below ~ 10 K, which is attributed to the decrease in scattering due to the ordering of the Pr moments. Upon Co-doping, the resistivity shows a drastic change and there was no observation of any anomaly due to structural distortion or SDW formation. Superconducting

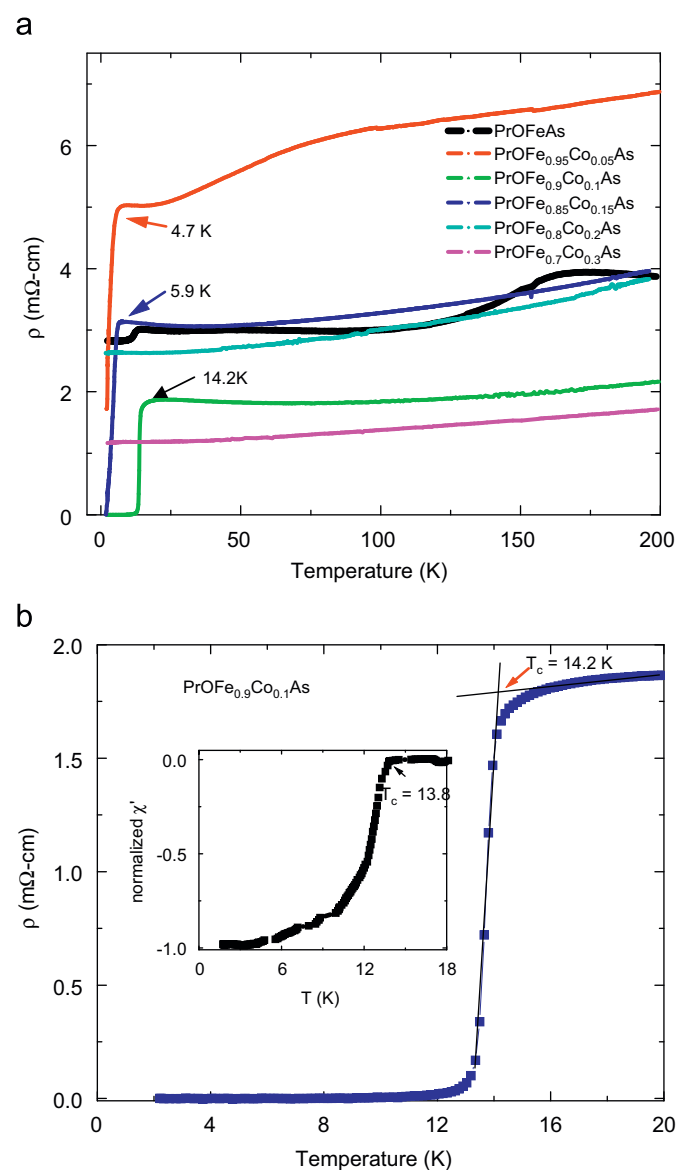


Fig. 3. (i) Temperature dependence of resistivity (ρ) for (a) PrOFeAs, (b) PrOFe_{0.95}Co_{0.05}As, (c) PrOFe_{0.9}Co_{0.1}As, (d) PrOFe_{0.85}Co_{0.15}As, (e) PrOFe_{0.8}Co_{0.2}As and (f) PrOFe_{0.7}Co_{0.3}As and (ii) temperature dependence of resistivity near the superconducting transition temperature. Inset shows the magnetic susceptibility as a function of temperature.

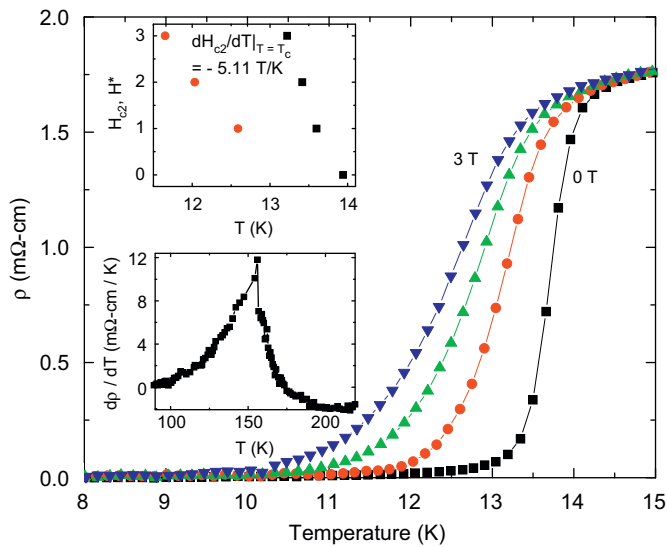


Fig. 4. Temperature dependence of the electrical resistivity of $\text{PrOFe}_{0.9}\text{Co}_{0.1}\text{As}$ under different magnetic fields. The upper inset shows the temperature dependence of estimated upper critical field (■) and irreversibility field (●) and lower inset indicates the temperature derivative of the resistivity ($d\rho/dT$).

transition is observed at 4.7 K for 'x'=0.05 (Fig. 3(i)) and increases with 'x' with highest T_c of 14.2 K for 'x'=0.1 composition. However, with higher Co-content, ($\text{PrOFe}_{0.85}\text{Co}_{0.15}\text{As}$, $\text{PrOFe}_{0.8}\text{Co}_{0.2}\text{As}$ and $\text{PrOFe}_{0.7}\text{Co}_{0.3}\text{As}$) the transition temperature (T_c) decreases. For 'x'=0.15, the resistivity drops steeply around 5.9 K and shows zero resistivity at ~ 2.5 K as shown in Fig. 3(i). However, for compositions with 'x'=0.2 and 0.3, though the resistivity (ρ) drops steeply around 4 K, suggesting a superconducting transition, zero resistivity was not observed down to lowest temperature (1.6 K). The normal state shows metallic behavior at higher temperature (Fig. 3(i)), but semiconducting behavior is always observed just above the transition temperature (T_c).

Fig. 3(ii) shows the zero field resistivity in the low temperature region (< 20 K) for $\text{PrOFe}_{0.9}\text{Co}_{0.1}\text{As}$. The resistivity decreases monotonously with decreasing temperature and a rapid drop was observed at 14.2 K showing onset of superconductivity. The criteria used for determination of transition temperature are same as that has been reported [31] and schematically shown in Fig. 3(ii). The onset of diamagnetism below the transition temperature (T_c) is observed from the plot of the real part of susceptibility for sample $\text{PrOFe}_{0.9}\text{Co}_{0.1}\text{As}$ (as shown in upper inset of Fig. 3(ii)). A slightly lower T_c of ~ 13.8 K is observed as compared to the resistivity studies. In our earlier report of $\text{CeOFe}_{0.9}\text{Co}_{0.1}\text{FeAs}$ [23] a superconducting transition was observed at 11.3 K, which indicates that the smaller praseodymium ion (as compared to cerium) plays an important role in the enhancement of T_c . It is expected that substitution of cobalt at the Fe site in the FeAs layer induces disorder which might increase with the cobalt concentration and this disorder may be responsible for the suppression of superconductivity at higher cobalt content.

In all the known cobalt-doped superconductors $\text{LnOFe}_{1-x}\text{Co}_x\text{As}$ ($\text{Ln}=\text{La}, \text{Ce}, \text{Pr}$ and Sm), the highest T_c was observed around 'x'=0.1. Increase in Co-content also leads to appreciable deviation of the Fe–As–Fe bond angle from the ideal tetrahedral angle [22]. Thus the FeAs layer plays an important role in these superconductors. However, the superconducting transition temperature was found to be less dependent on the charge reservoir (Ln-O) layer in these Co-doped superconductors as compared to fluoride-doped superconductors where substitution of La with Sm

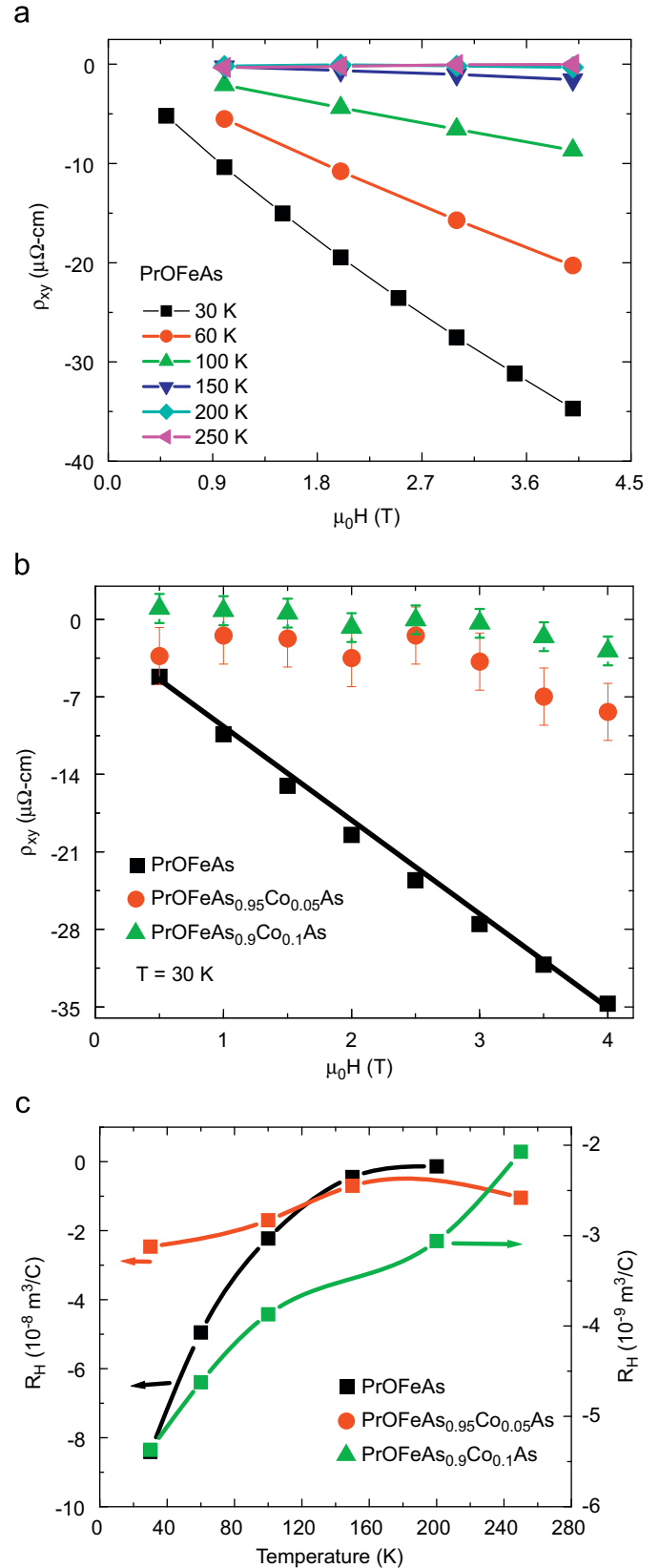


Fig. 5. (a) Hall resistivity (ρ_{xy}) versus the magnetic field (μ_0H) for PrOFeAs , (b) Hall resistivity (ρ_{xy}) versus the magnetic field (μ_0H) for PrOFeAs , $\text{PrOFe}_{0.95}\text{Co}_{0.05}\text{As}$ and $\text{PrOFe}_{0.9}\text{Co}_{0.1}\text{As}$ at 30 K (5% error bar estimated at 30 K) and (c) temperature dependence of the Hall coefficient (R_H) for PrOFeAs (■), $\text{PrOFe}_{0.95}\text{Co}_{0.05}\text{As}$ (●) and $\text{PrOFe}_{0.9}\text{Co}_{0.1}\text{As}$ (▲).

ions results in enhancement of T_c from 26 to 55 K. This suggests subtle differences in the two families of $LnOFeAs$ superconductors (fluoride- and cobalt-doped).

To obtain the upper critical field (H_{c2}), we have studied the temperature dependence of the resistivity under different magnetic fields (Fig. 4) for $PrOFe_{0.9}Co_{0.1}As$. The onset temperature shifts weakly with magnetic field, but the zero resistivity temperature shifts more rapidly to lower values. By taking a criterion of 90% and 10% of normal state resistivity (ρ_n), we calculated the upper critical field H_{c2} and the irreversibility field $H^*(T)$, respectively. The H - T phase diagram for this sample is shown in inset of Fig. 4. By using the Werthamer–Helfand–Hohenberg (WHH) formula [32], the zero field upper critical field $H_{c2}(0)$ can be calculated [23] to be 50.2 T for $PrOFe_{0.9}Co_{0.1}As$. These values are slightly higher than the reported H_{c2} value of $CeO(Fe/Co)As$ [23].

The variation of transverse resistivity (ρ_{xy}) with magnetic field for $PrOFeAs$ compound is shown in Fig. 5(a). Transverse resistivity (ρ_{xy}) remains negative at all temperatures above the critical temperature for compound $PrOFeAs$. We have also showed the transverse resistivity at 30 K for $PrOFeAs$, $PrOFe_{0.95}Co_{0.05}As$ and $PrOFe_{0.9}Co_{0.1}As$ in Fig. 5(b). This indicates that the electrical transport is dominated by the electron-like charge carriers and not holes. For a clean sample, the nonlinear Hall effect is a good sign of a multiband superconductor, and the effect is weaker in dirtier samples. From the above data, the Hall coefficient $R_H = \rho_{xy} / \mu_0 H$ was determined for compounds with 'x'=0, 0.05 and 0.1 (Fig. 5 (c)). The charge carrier density calculated from the equation $n = 1/R_H e$ is about $0.074 \times 10^{21} \text{ cm}^{-3}$, $0.27 \times 10^{21} \text{ cm}^{-3}$ and $1.16 \times 10^{21} \text{ cm}^{-3}$ for $PrOFeAs$, $PrOFe_{0.95}Co_{0.05}As$ and $PrOFe_{0.9}Co_{0.1}As$, respectively, at 30 K. It shows that charge carriers increase with Co-doping.

Fig. 6 shows the temperature dependence of thermopower (S) in $PrOFe_{0.9}Co_{0.1}As$ from room temperature to 13 K. The thermopower (S) is negative in the entire temperature range, which shows that the electron-like charge carriers dominate as seen from Hall effect. For the undoped parent compound, it is reported [33] that as the thermopower starts to increase abnormally around T_{anom} the resistivity starts to decrease. This remarkable change in the thermopower is probably caused by the change in the electronic state when the system undergoes the structural phase transition and SDW transition. This anomaly is

suppressed on cobalt doping for $x \geq 0.05$, consistent with resistivity data. The magnitude of S for the superconducting sample is smaller than that for the non-superconducting sample $PrOFeAs$ [33].

4. Conclusions

We have successfully synthesized monophasic samples of $PrOFe_{1-x}Co_xAs$ superconductors with maximum T_c at 14.2 K for 'x'=0.1 composition. Cobalt substitution at the iron site in $PrOFeAs$ suppresses the structural distortion and magnetic instability (SDW) with the evolution of superconductivity. Higher cobalt doping leads to suppression of the superconducting transition temperature. Mössbauer spectroscopy data indicates divalent state of Fe both in undoped and cobalt-doped samples. The upper critical field at 0 K is estimated to be about 50.2 T. The negative values of the thermopower and Hall coefficient (R_H) over a wide temperature range suggests that the electron type charge carriers dominate the conduction in this system. The strong temperature dependence of R_H is normally associated with a multiband effect (two band model with different charge carriers). Hall measurements also indicate that conduction electrons are added with increasing Co-doping.

Acknowledgments

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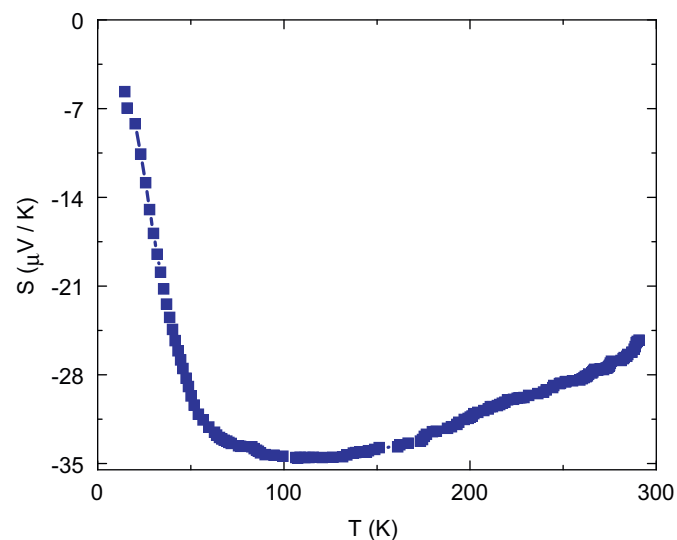


Fig. 6. Temperature dependence of thermopower (S) for $PrOFe_{0.9}Co_{0.1}As$ from room temperature to 13 K.

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