Phase diagram of CeFeAs$_{1-x}$P$_x$O obtained from electric resistivity, magnetization, and specific heat measurements

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We performed a systematic study on the properties of CeFeAs$_{1-x}$P$_x$O (0 $\leq x \leq 1$) by electrical resistivity, magnetization and specific heat measurements. The c-axis lattice constant decreases significantly with increasing P content, suggesting a remarkable chemical pressure. The Fe-3$d$ electrons show the enhanced metallic behavior upon P-doping and undergo a magnetic quantum phase transition around $x \approx 0.4$. Meanwhile, the Ce-4$f$ electrons develop a ferromagnetic order near the same doping level. The ferromagnetic order is vanishingly small around $x = 0.9$. The data suggest a heavy-fermion-like behavior as $x \geq 0.95$. No superconductivity is observed down to 2 K. Our results show the ferromagnetic ordered state as an intermediate phase intruding between the antiferromagnetic bad metal and the nonmagnetic heavy fermion metal and support the cerium-containing iron pnictides as a unique layered Kondo lattice system.

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I. INTRODUCTION

The homologous quaternary iron pnictides, $R$Fe$XO$, ($R$: rare earths, $X$: As or P) show a diversity of physical properties although they have the same ZrCuSiAs-type crystal structure with a unique Fe$X$-layer sandwiched by the RO-layer. Typically, LaFeAsO is an antiferromagnetic (AFM) metal below 140 K, and is a superconductor (SC) with maximal $T_c$ = 26 K upon F-doping. $T_c$ can be surprisingly increased up to 41 K when La is replaced by Ce$^3+$ or even higher if replaced by other rare earths. Meanwhile, the parent compounds (denoted as Ln-1111) of these SC's, such as CeFeAsO, are still Fe-3$d$ itinerant AFM's similar to LaFeAsO. However, a noticeable $f$-electron AFM ordering of Ce$^{3+}$ is also observed at a much lower temperature ($\sim$4 K). On the other hand, the stoichiometric LaFePO is a low $T_c$ SC without any trace of the AFM ordering. By contrast, CeFePO is a heavy fermion (HF) metal with Kondo temperature $T_K \sim 10$ K.

These discoveries put the rare-earth iron pnictides on the boundary between the high-$T_c$ superconductors and the heavy fermion metals, and open an avenue for searching the complicated interplay of various $d$- and $f$-electron correlations. Within the iron-pnictogen layer, the $d$-electrons are expected to be more itinerant in the phosphides than in the arsenides, with the former being recently identified as a moderately correlated metal. In contrast to the cuprates, the inter-layer distance between the transition metals and the rare-earths in the iron pnictides is critically significant so that the coupling between them may play an important role even in the parent compounds of the high-$T_c$ Fe-based SC's.

Recent neutron scattering and muon spin relaxation experiments indeed provide evidence for a sizable inter-layer coupling in CeFeAsO. So far it is still unclear whether this coupling is due to some kind of polarization effect raised by the ordered moment of Fe$^{2+}$-ions or more microscopically the effective hybridization between the 3$d$- and 4$f$-orbitals bridged by the pnictogens. A first-principle local-density approximation (LDA) plus dynamical mean-field theory (DMFT) study suggested that applying physical pressure will enhance the $d$-$f$ hybridization, leading to the Kondo screening of the Ce-moments. However a sufficiently high pressure is required in order to observe this effect. It turns out that CeFeAs$_{1-x}$P$_x$O, i.e., P doping at As sites in CeFeAsO, may provide, among others, a unique layered Kondo lattice system to probe the intriguing 3$d$-4$f$ electron interplay under ambient pressure. Chemical-pressure-induced superconductivity has been observed in P-doped LaFeAsO.

We report a systematic study on the doping evolution of the physical properties of CeFeAs$_{1-x}$P$_x$O using electrical resistivity $\rho(T)$, magnetic susceptibility $\chi(T)$, isothermal magnetization $M(H)$ and specific heat $C(T)$ measurements. A series of 21 different P-doped polycrystalline samples were synthesized. Our results reveal a rich phase diagram consisting of an AFM quantum critical point (QCP) of the Fe-3$d$ electrons and a possible ferromagnetic (FM) QCP of the Ce-4$f$ electrons. An intermediate FM phase emerges in between the AFM bad metal and the nonmagnetic HF metal. Because P doping does not introduce extra electrons but shortens the c-axis significantly, these findings provide a rare example of chemical-pressure-induced HF metals with strong FM fluctuations.
II. EXPERIMENTAL

We synthesized a series of CeFeAs$_{1-x}$P$_x$O ($0 \leq x \leq 1$) polycrystalline samples by solid state reaction, during which, Ce, Fe, As, P and CeO$_2$ of high purity (≥ 99.95%) were used as starting materials. First, CeAs (or CeP) was presynthesized by reacting Ce discs and As (or P) powders at 1323 K for 72 h. FeAs (or FeP) was prepared by reacting Fe and As (or P) powders at 1173 K (or 1023 K) for 20 h. Second, powders of CeAs, CeP, CeO$_2$, FeAs and FeP were weighed according to the stoichiometric ratio, thoroughly ground, and pressed into a pellet under a pressure of 600 MPa in an Argon filled glove box. The pellet was sealed into an evacuated quartz tube, which was then slowly heated to 1448 K and kept at that temperature for 50 h.

Powder X-ray diffraction (XRD) was performed at room temperature using a D/Max-rA diffractometer with Cu-K$_{\alpha}$ radiation and a graphite monochromator. Lattice parameters were refined by a least-squares fit using at least 30 XRD peaks, and the structural refinements were performed using the programme RIETAN 2000. The dc magnetization measurement was carried out in a Quantum Design Magnetic Property Measurement System (MPMS-5). Physical Property Measurement System (PPMS-9) was used to take the resistivity measurement, as well as specific heat.

III. RESULTS AND DISCUSSION

The room-temperature powder x-ray diffraction (XRD) patterns of CeFeAs$_{1-x}$P$_x$O are shown in Fig.1. All the XRD peaks can be well indexed based on the tetragonal ZrCuSiAs-type structure with the space group P4/mnm (No.129), and no obvious impurity phases can be detected, suggesting high quality of the samples. As shown in Fig.1(a-b), the XRD peaks shift toward right-hand, especially for the high angle reflections. The (003) peaks shift much faster than the (110) peaks so that they merge into one peak in the high doping case, indicating that the c-axis shrinks more severely than the a-axis.

This observation is consistent with the lattice parameter calculation results shown in Fig.1(c). Fig.1(d-e) present the Rietveld refinement profiles for the two end members CeFeAsO and CeFePO, which give details of the crystal structure. The remarkable differences in structure lie in the positions of Ce and As/P. For CeFePO, the thickness of FeP layers is much smaller, meanwhile, Ce atomic layers are much closer to the FeP layers. This structural feature supports the strong coupling between Fe-3d and Ce-4$f$ electrons.

The temperature dependent resistivity is shown in Fig.2, where four prominent features can be identified. (i) The overall suppression of the room temperature resistivity with increasing doping. The resistivity at $T = 300$ K decreases gradually from 300 $\mu$Ohm for $x=0$ to 12 $\mu$Ohm for $x=1$, indicating that P-doping enhances the metallic behavior significantly. (ii) The resistivity anomaly, mostly pronounced for $x = 0$. This anomaly was ascribed to the structure distortion and the accompanied Fe-AFM transition$^{20,21}$. Upon doping, the anomaly is suppressed monotonically and soon becomes an unremarkable kink at lower temperatures. No clear kink can be identified for $x > 0.3$. The suppression of this anomaly with P-doping is consistent with the suppression of AFM order of Fe ions, which is confirmed by a recent neutron study$^{22}$. We mark arrows in Fig.2(a) to show the Fe-AFM transition ($T_{N1}$, data from Ref.22). (iii) The resistivity upturn at low temperatures for $x \leq 0.4$. The upturn behavior is suppressed by doping or magnetic field, see the inset in Fig.2(b). (iv) The metallic behavior in the entire measured temperature range for $x > 0.4$. In particular, the resistivity drops rapidly at low temperatures for larger $x$.

Since a drop in resistivity has already been observed in the HF metal CeFePO in the previous report$^{23}$. However, no SC is observed down to $T=2$ K for the entire P doping range.

It should be noted that the value of resistivity itself may not reflect the poly-crystalline sample’s quality. Actually the most likely impurity phases in the Ln-1111 pnictides could be FeAs or Fe$_2$As, and Ln$_2$O$_3$. Both FeAs and Fe$_2$As are highly metallic, more metallic than CeFeAsO itself at low temperatures. The metallic impurity phase like FeAs could distribute most likely in the grain boundaries and thus result in a lower resistivity if the main phase is a bad metal. The intrinsic resistivity behavior of Ln-1111 parent pnictides below the Fe-AFM order is still ambiguous. For example, the resistivity of single crystal of LaFeAsO shows an upturn at low temperatures$^{23}$, meanwhile the resistivity of both poly-crystalline and single-crystalline CeFeAsO samples shows clear metallic behavior$^{24}$. However, our more than 20 CeFeAs$_{1-x}$P$_x$O samples with different P content were prepared under same conditions and the XRD patterns show that they all are of same high purity, the systematic decrease in resistivity indeed suggests the increasing metallicity with increasing P content.

The temperature dependence of dc magnetic susceptibility is shown in Fig.3. In the high temperature range, $\chi(T)$ increases with decreasing temperature down to 150 K following a Curie-Weiss law for all $0 \leq x \leq 1$. The derived effective moment $\mu_{eff}$ is $2.54 \mu_B$ for $x=0$, and $2.56 \mu_B$ for $x=1$ [insets of Fig.3], very close to that of a free Ce$^{3+}$ ion, $2.54 \mu_B$. By lowering temperatures, a clear peak was observed at 4.16 K for $x=0$, related to the formation of the Ce$^{3+}$ AFM order. With increasing $x$, $\chi(T)$ increases and the peak becomes round (or shoulder-like); meanwhile the corresponding AFM ordering temperature does not change too much. For $x \geq 0.4$, an obvious divergence between zero-field-cooling (ZFC) and field-cooling (FC) is seen at lower temperatures [see Fig.3(b)], manifesting the FM ordered ground state of the Ce-4$f$ electrons. The suppression of AFM and development of FM are consistent with the enhanced metallic transport behavior in the course of P-doping. The FM
ordered state persists until $x \sim 0.9$ where the divergency of $\chi(T)$ vanishes.

The evolution of Ce$^{3+}$ magnetism can be further demonstrated by isothermal field-dependent magnetization measurements, which are shown in Fig.4. The temperatures are fixed at 2 K and 10 K, respectively. For $x = 0$ and $T=2$ K, a kink in the magnetization curve is clearly seen, indicating a possible spin flop in the AFM state of Ce$^{3+}$. When $x$ increases the kink becomes weaker. As $x \geq 0.4$, $M(H)$ starts to increase rapidly for small fields and tends to saturate at higher fields, i.e., it exhibits a nonlinear field dependence, see Fig.4(d) for $x = 0.4$. For $0.5 \lesssim x < 0.9$, a clear hysteresis loop can be observed and the saturated $M$ value reaches the maximum around $x = 0.6$ [see Fig.4(e)]. Therefore, the long-range FM order exists in a wide doping range in between $x \sim 0.4$ and $x \sim 0.9$. The largest saturated magnetic moment is about 0.95 $\mu_B$, close to 1 $\mu_B$ expected for a Ce$^{3+}$ doublet ground state. For $0.9 \lesssim x < 0.95$ the hysteresis loop is hardly observable down to the lowest measured temperature while the saturation tendency persists. This may be due to strong FM fluctuations at the quantum critical point associated with the 4f-electrons. On the other hand, both the susceptibility and magnetization show a non-magnetic HF-like phase for $x \geq 0.95$ [see Fig.3(b) and Fig.4(g-h)], consistent with the resistivity measurement.

The metallic HF-like behavior can be further manifested by the specific heat measurement as shown in Fig.5. For $x = 0.95, 0.97,$ and 1, the specific heat coefficient $\gamma(T) = C(T)/T$ shows log$T$ behavior at low temperatures down to 2 K, see the inset of Fig.5. We define a characteristic temperature $T_\gamma$ (below which HF-like behavior appears) as the intersection of two extrapolate lines of the specific heat data. For the $x = 1$ sample, $T_\gamma \sim 8.6$ K. The saturated $\gamma$ should be about 700-800 mJ/molK$^2$ or larger, in agreement with the result of Ref.2 where $\gamma$ increases in proportion to log$T$ before it gets saturated below 1 K. As a comparison, $\gamma(T)$ shows a $\lambda$-peak maximized around 4.2 K and 6.8 K in the AFM and FM ordered phases, for $x = 0$ and 0.8, respectively, in agreement with the magnetization measurement. A sudden increase in the saturated $\gamma$ close to $x \sim 0.9$ can be inferred by the values of $\gamma(T = 2)$, accompanied by
a decrease in the saturated magnetization under $\mu_0H = 5$ T. This implies the enhancement of the Kondo screening near the FM instability. It should be noted that a very tiny kink was observed around 6 K on the $C/T − T$ curve of $x=0.95$. We ascribe this additional peak to the contribution from some impurities with lower P content. Roughly estimated from the intensity of the kink, the amount of the impurity phase should be less than 2%.

We summarize the experimental results by suggesting an electronic phase diagram shown in Fig.6. At low temperatures, both the Fe-3$d$ and Ce-4$f$ electrons show the long-range AFM ordering for $x \lesssim 0.37$. The d-electrons are Pauli paramagnetic(PM) for $x \gtrsim 0.4$, while the f-electrons are FM ordered until $x \sim 0.9$. For $x \gtrsim 0.95$, the f-moments are completely quenched and the whole system becomes a nonmagnetic HF-like metal. The HF-like region is roughly depicted by $T_c$. Therefore, in addition to the first magnetic QCP (denoted by $x_{c1}$) associated with the AFM-PM transitions of the d-electrons, there could be a another QCP associated with the FM-HF transition of f-electrons around $x$ of 0.92 (denoted by $x_{c2}$). $x_{c1}$ has been known to be 0.4 by the study of neutron scattering. A turning point (denoted by $x_{c2}$) associated with the AFM-FM transition of the f-electrons at $x_{c2} \sim 0.37$ is quite close to $x_{c1}$. Meanwhile the strong FM fluctuations show up in the vicinity of $x_{c2}$.

It should be noticed that the transition temperatures of the Fe-AFM ordering and the structure distortion can be approximately determined from the resistivity anomaly as shown in Fig.2 and neutron studies. They are undistinguishable in resistivity when $x > 0.2$. The neutron study revealed that both of them are suppressed at $x_{c2} \sim 0.4$, confirming an early theoretical prediction for the existence of a unique magnetic QCP driven by P-doping. Moreover, the d-electron QCP seems to be very close to the turning point ($x_{c2}$) of the f-electrons. This fact may indicate that the d-electron magnetic transition is of weak first order accompanied by a reconstruction of the electronic states across $x_{c2}$. It is not certain if the coincidence implies the AFM ordering in Ce subsystem being supported by the AFM order in the Fe plane, because the ordering patterns of the two subsystems are different and the f-electron AFM order can coexist with the d-electron SC order in the F-doping case. Besides this puzzling feature, other implications of our experimental results should be addressed as follows.

First, as the suppression of the a-axis is only a frac-

FIG. 2: (Color online) Temperature dependence of resistivity in CeFeAs$_{1−x}$P$_x$O. (a), For $0 \leq x \leq 0.4$. Arrows denotes the temperature of Fe-AFM order, $T_N$, which were taken from Ref. (b), For $0.4 \leq x \leq 1$. The inset shows the resistivity of $x=0.3$, 0.4 and 0.5, under zero field (solid lines) and $\mu_0H$ of 5 T (dashed lines).

FIG. 3: (Color online) Temperature dependence of dc magnetic susceptibility under ZFC (solid symbols) and FC (open symbols) protocols for CeFeAs$_{1−x}$P$_x$O samples under a magnetic field of 1000 Oe. Arrows are guides to eyes to indicate the FM transition temperature $T_C$ which is defined from the extrapolation of $1/\chi − T$ plot. The insets show the Curie-Weiss behavior of susceptibility above 150 K for CeFeAsO and CeFePO respectively.
shown. The insets of (e) and (f) display the magnetic hysteresis loop at 2 K.

FIG. 4: (Color online) Isothermal magnetization of CeFeAs$_{1-x}$P$_x$O. (a), $x = 0$. (b), $x = 0.2$. (c), $x = 0.3$. (d), $x = 0.4$. (e), $x = 0.6$. (f), $x = 0.8$. (g), $x = 0.9$. (h), $x = 1.0$. For clarity, only the data of 2 K (solid lines) and 10 K (dashed lines) are shown. The insets of (e) and (f) display the magnetic hysteresis loop at 2 K.

FIG. 5: (Color online) Specific heat measurements of CeFeAs$_{1-x}$P$_x$O. A $\lambda$-peak can be observed around $T = 4.2$ K and 6.8 K for $x = 0$ and 0.8, respectively, corresponding to the AFM and FM transitions. Inset: For $x = 0.95$, 0.97 and 1, the specific heat coefficient $\gamma(T) = C(T)/T$ shows log $T$ behavior at low temperatures down to 2 K, and tends to saturate to $\gamma(0)$ when $T \to 0$. The $\gamma(0)$ value was extrapolated to be about 700-800 mJ/K$^2$ mol or larger for $x = 1.0$.

FM transition could be explained by assuming the existence of a bare $d$-$f$ Kondo coupling ($J_K$) in the ordered phases, as it can mediate the indirect Ruderman-Kittel-Kasuya-Yosida (RKKY) interaction ($J_{RKKY}$) between the Ce-$4f$ local moments. Intuitively, the exchange interaction between the local $f$-spins, $J_f$, is given by $J_f = J_f^{(0)} + J_{RKKY}$, with $J_f^{(0)}$ being the exchange interaction in the absence of $J_K$, and $|J_{RKKY}| \sim J_K^2 N_F$, with $N_F$ being the density of states at Fermi level (of mainly $d$-electron characteristic). $J_{RKKY}$ is negative if $N_F$ is small, as it should be since the parent compound CeFeAsO is a bad metal. Upon P doping, both $J_K$ and $N_F$ should increase, leading to a sign change in $J_f$. Third, the negative $J_f$ is also the driving force for the strong FM fluctuations in the vicinity of the critical point $x_{c2}$, where the Kondo scale $T_K$ (which increases exponentially with $J_K N_F$) starts to dominate over the magnetic ordering energy scale, $T_I \approx |J_f|$.

It is well-established that the RKKY interaction between the local moments is mediated by the Kondo-coupling between the itinerant charge carries and local moments. The Kondo coupling can lead to both the Kondo effect and the RKKY interaction, with the latter being responsible to the magnetic ordering of the local moments. The crucial point is that the dependences on the Kondo coupling (together with the DOS of the charge carries) of the Kondo energy scale (describing the Kondo effect) and of the RKKY interaction (characterizing the ordering temperature of the local moments) are different, as described by the intuitive formulas in the inset of Fig. 6. Based on the similar intuitive formulas, Doniach first suggested that a quantum phase transition from magnetic ordering phase to the paramagnetic HF...
FIG. 6: (Color online) Electronic phase diagram of CeFeAs$_{1-x}$P$_x$O ($0 \leq x \leq 1$). The shaded region (in red) in the left side shows the antiferromagnetic ordering of Fe-$3d$ moments (denoted by Fe-AFM below $T_N$). In the lower temperature portion of this region, simultaneously, Ce-$4f$ moments order antiferromagnetically (marked with Ce-AFM) below $T_N$. The small pink area at the lower right corner displays the nonmagnetic state with heavy fermion (HF) behaviour. In the lower middle regime (in green), the $4f$-moments are ferromagnetically ordered (labeled by Ce-FM) below $T_C$. Inset: The schematic competition between $J_{f}$ and $T_K$ accounting for the the AFM-FM-HF evolution of the $f$-electrons.

phase could exist in a Kondo lattice. Here, we suggest that the AFM-FM-HF evolution of the $f$-electrons in CeFeAs$_{1-x}$P$_x$O is due to the competition between $T_K$ and $J_f$ (instead of $J_{RKKY}$ in the conventional Kondo lattice), see the inset in Fig.6. Notice that $J_f^{(0)}$ is positive and relatively small in the absence of $J_{RKKY}$. Thus in the AFM phase $T_K$ is mainly suppressed by the AFM ordering gap of the $d$-electrons. This picture provides a natural explanation for the strong FM fluctuations in a class of homologous HF compounds including CeFePO$_2$ and CeRuPO$_2$. Therefore the observed $f$-electron phase diagram of CeFeAs$_{1-x}$P$_x$O system is consistent with the Doniach picture. In addition, it also predicts a possible weak Kondo phase in a narrow doping region enclosing the turning point $x_{c3}$ (at very low temperatures). As $x_{c3}$ is very close to $x_{c1}$, it may account for the observed suppression of the SC near the AFM QCP. The existence of this peculiar phase and its possible connection with the transport and magnetic properties still need to be clarified.

IV. CONCLUSION

In summary, our experimental study on CeFeAs$_{1-x}$P$_x$O ($0 \leq x \leq 1$) reveals a rich phase diagram which consists of an AFM quantum phase transition of the Fe-$3d$ electrons and an AFM-FM-HF evolution of the Ce-$4f$ electrons. The AFM QCP of the $d$-electrons is very close to the turning point of the AFM-FM transition of the $f$-electrons, while the $d$-$f$ hybridized HF state is separated by an $f$-electron FM instability. In contrast to the F-doping case where the high-$T_C$ SC is induced, no SC is observed down to 2 K in the present case, signalling the unique role of P-doping in suppressing the $d$-electron correlation.$^{13,25}$ These results highlight the importance of the inter-layer Kondo physics$^{15,31}$ and the interplay of the $d$- and $f$-electron correlations in the rare-earth iron pnictides.

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