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Structural and Magnetic Phase Transitions near Optimal Superconductivity in BaFe$_2$(As$_{1-x}$P$_x$)$_2$

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We use nuclear magnetic resonance (NMR), high-resolution x-ray, and neutron scattering studies to study structural and magnetic phase transitions in phosphorus-doped BaFe$_2$(As$_{1-x}$P$_x$)$_2$. Previous transport, NMR, specific heat, and magnetic penetration depth measurements have provided compelling evidence for the presence of a quantum critical point (QCP) near optimal superconductivity at $x = 0.3$. However, we show that the tetragonal-to-orthorhombic structural ($T_s$) and paramagnetic to antiferromagnetic (AF, $T_N$) transitions in BaFe$_2$(As$_{1-x}$P$_x$)$_2$ are always coupled and approach $T_N \approx T_s \approx T_c$ ($\approx 29$ K) for $x = 0.29$ before vanishing abruptly for $x \geq 0.3$. These results suggest that AF order in BaFe$_2$(As$_{1-x}$P$_x$)$_2$ disappears in a weakly first-order fashion near optimal superconductivity, much like the electron-doped iron pnictides with an avoided QCP.

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A determination of the structural and magnetic phase diagrams in different classes of iron pnictide superconductors will form the basis from which a microscopic theory of superconductivity can be established [1–5]. The parent compound of iron pnictide superconductors such as BaFe$_2$As$_2$ exhibits a tetragonal-to-orthorhombic structural transition at temperature $T_s$ and then orders antiferromagnetically below $T_N$ with a collinear antiferromagnetic (AF) structure [Fig. 1(a)] [3,4]. Upon hole doping via partially replacing Ba by K or Na [6,7], the structural and magnetic phase transition temperatures in Ba$_{1-x}$Fe$_2$As$_2$ (A = K, Na) decrease simultaneously with increasing $x$ and form a small pocket of a magnetic tetragonal phase with the $c$-axis-aligned moment before disappearing abruptly near optimal superconductivity [8–11]. For electron-doped Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ ($T = \text{Co, Ni}$), transport [12,13], muon spin relaxation [14], nuclear magnetic resonance (NMR) [15–17], x-ray, and neutron scattering experiments [18–23] have revealed that the structural and magnetic phase transition temperatures decrease and separate with increasing $x$ [18–23]. However, instead of a gradual suppression to zero temperature near optimal superconductivity as expected for a magnetic quantum critical point (QCP) [15,16], the AF order for Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ near optimal superconductivity actually occurs around 30 K ($> T_c$) and forms a short-range incommensurate magnetic phase that competes with superconductivity and disappears in the weakly first-order fashion, thus avoiding the expected magnetic QCP [20–23].

Although a QCP may be avoided in electron-doped Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ due to disorder and impurity scattering in the FeAs plane induced by Co and Ni substitution, phosphorus-doped BaFe$_2$(As$_{1-x}$P$_x$)$_2$ provides an alternative system to achieve a QCP since substitution of As by the isovalent P suppresses the static AF order and induces superconductivity without appreciable impurity scattering [24–27]. Indeed, experimental evidence for the presence of a QCP at $x = 0.3$ in BaFe$_2$(As$_{1-x}$P$_x$)$_2$ has been mounting, including the linear temperature dependence of the resistivity [28], an increase in the effective electron mass seen from the de Haas-van Alphen effect [26], magnetic penetration depth [29,30], heat capacity [31], and normal state transport measurements in samples where superconductivity has been suppressed by a magnetic field [32]. Although these results, as well as NMR measurements [33], indicate a QCP originating from the suppression of the static AF
BaFe
P-doped samples, RRR quantum criticality cannot exist at compositions higher than experiments directly measuring FIG. 1 (color online). (a) The AF-ordered phase of 
114, with the earlier results obtained from powder samples near single crystals focused on determining the P-doping T shows the expanded view of the P-concentration dependence of 
indicated the mesoscopic coexisting AF and SC phases. The dashed region represents the temperature and doping dependence of the nor-
malized magnetic Bragg peak intensity. The color bar indicates the temperature and doping dependence of the normalized magnetic Bragg peak intensity. The dashed region indicates the mesoscopic coexisting AF and SC phases.

order near x = 0.3, recent neutron powder diffraction experiments directly measuring T_s and T_N in BaFe_2(As_1-xP_x)2 as a function of x suggest that structural quantum criticality cannot exist at compositions higher than x = 0.28 [34]. Furthermore, the structural and magnetic phase transitions at all studied P-doping levels are first order and occur simultaneously within the sensitivity of the measurements (~0.5 K), thus casting doubt on the presence of a QCP [34]. While these results are interesting, they were carried out on powder samples and, thus, are not sensitive enough to the weak structural or magnetic order to allow a conclusive determination on the nature of the structural and AF phase transitions near optimal superconductivity.

In this Letter, we report systematic transport, NMR, x-ray, and neutron scattering studies of BaFe_2(As_1-xP_x)2 single crystals focused on determining the P-doping evolution of the structural and magnetic phase transitions near x = 0.3. While our data for x \leq 0.25 are consistent with the earlier results obtained from powder samples [34], we find that nearly simultaneous structural and magnetic transitions in single crystals of BaFe_2(As_1-xP_x)2 occur at T_s \approx T_N \geq T_c = 29 K for x = 0.28 and 0.29 (near optimal doping) and disappear suddenly at x \geq 0.3. While superconductivity dramatically suppresses the static AF order and lattice orthorhombicity below T_c for x = 0.28 and 0.29, the collinear static AF order persists in the superconducting state. Our neutron spin echo and NMR measurements on the x = 0.29 sample reveal that only part of the sample is magnetically ordered, suggesting its mesoscopic coexistence with superconductivity. Therefore, despite reduced impurity scattering, P-doped BaFe_2As_2 has remarkable similarities in the phase diagram to that of electron-doped Ba(Fe_1-xT_x)2As_2 iron pnictides with an avoided QCP.

We have carried out systematic neutron scattering experiments on BaFe_2(As_1-xP_x)2 with x = 0.19, 0.25, 0.28, 0.29, 0.30, and 0.31 [35] using the C5, RITA-II, and MIRA triple-axis spectrometers at the Canadian Neutron Beam center, Paul Scherrer Institute, and Heinz Maier-Leibnitz Zentrum (MLZ), respectively. We have also carried out neutron resonance spin echo (NRSE) measurements on the x = 0.29 sample using TRISP triple-axis spectrometer at MLZ [36]. Finally, we have performed high-resolution x-ray diffraction experiments on identical samples at Ames Laboratory and Advanced Photon Source (APS), Argonne National Laboratory (ANL) (see the Supplemental Material [37]). Our single crystals were grown using a Ba_3As_2/Ba_3P_3 self-flux method, and the chemical compositions are determined by inductively coupled plasma analysis with 1% accuracy [35]. We define the wave vector Q at (q_x, q_y, q_z) as \{H, K, L\} = \{q_x a/2\pi, q_y b/2\pi, q_z c/2\pi\} reciprocal lattice units using the orthorhombic unit cell suitable for the AF-ordered phase of iron pnictides, where a \approx b \approx 5.6 Å and c = 12.9 Å. Figure 1(b) shows temperature dependence of the resistivity for x = 0.31 sample, confirming the high quality of our single crystals [28].

Figure 1(c) summarizes the phase diagram of BaFe_2(As_1-xP_x)2 as determined from our experiments. Similar to previous findings on powder samples with x \leq 0.25 [34], we find that the structural and AF phase transitions for single crystals of x = 0.19, 0.28, and 0.29 occur simultaneously within the sensitivity of our measurements (~1 K). On approaching optimal superconductivity as x \to 0.3, the structural and magnetic phase transition temperatures are suppressed to T_s \approx T_N \approx 30 K for x = 0.28, 0.29 and then vanish suddenly for x = 0.3, 0.31 as shown in the inset of Fig. 1(c). Although superconductivity dramatically suppresses the lattice orthorhombicity and static AF order in x = 0.28, 0.29, there is still remnant static AF order at temperatures well below T_c. However, we find no evidence of static AF order and lattice orthorhombicity for x = 0.3 and 0.31 at all temperatures. Since our NMR measurements on the x = 0.29 sample suggest that the magnetic order takes place in about ~50% of the volume fraction, the coupled T_s and T_N AF phase in BaFe_2(As_1-xP_x)2 becomes a homogeneous superconducting phase in the weakly first-order fashion, separated by a phase with coexisting AF clusters and superconductivity [dashed region in Fig. 1(c)].
reveals a rather sudden change at $T_N$ and a dramatic suppression of the magnetic intensity below $T_s$. Figures 2(e) and 2(f) indicate that the magnetic order in the $x = 0.29$ sample behaves similar to that of the $x = 0.28$ crystal without much reduction in $T_N$. On increasing the doping levels to $x = 0.3$ (Supplemental Material [37]) and 0.31 [Fig. 2(f)], we find no evidence of magnetic order above 2 K. Given that the magnetic order parameters near $T_N$ for the $x = 0.28, 0.29$ samples look remarkably like those of the spin cluster phase in electron-doped Ba(Fe$_{1-x}$T$_x$)$_2$As$_2$ near optimal superconductivity [22,23], we have carried out additional neutron scattering measurements on the $x = 0.29$ sample using TRISP, which can operate as a normal thermal triple-axis spectrometer with instrumental energy resolution of $\Delta E \approx 1$ meV and a NRSE triple-axis spectrometer with $\Delta E \approx 1 \mu$eV [36]. Figure 2(h) shows the triple-axis mode data which reproduces the results in Fig. 2(f). However, identical measurements using NRSE mode reveal that the magnetic scattering above 30.7 K is quasielastic and the spins of the system freeze below 30.7 K on a time scale of $\tau \sim \hbar/\Delta E \approx 6.5 \times 10^{-10}$ s [23]. This spin freezing temperature is almost identical to those of nearly optimally electron-doped Ba(Fe$_{1-x}$T$_x$)$_2$As$_2$ [21–23].

Figure 3 summarizes the key results of our x-ray scattering measurements carried out on samples identical to those used for neutron scattering experiments. To facilitate quantitative comparison with the results on Ba(Fe$_{1-x}$T$_x$)$_2$As$_2$, we define the lattice orthorhombicity $\delta = (a-b)/(a+b)$ [19,22]. Figure 3(a) shows the temperature dependence of $\delta$ for BaFe$_2$(As$_{1-x}$P$_x$)$_2$ with temperature dependence of the scattering at (1,0,3), revealing a continuously increasing magnetic order parameter near $T_N$ and a dramatic suppression of the magnetic intensity below $T_s$. Figures 2(e) and 2(f) indicate that the magnetic order in the $x = 0.29$ sample behaves similar to that of the $x = 0.28$ crystal without much reduction in $T_N$. On increasing the doping levels to $x = 0.3$ (Supplemental Material [37]) and 0.31 [Fig. 2(f)], we find no evidence of magnetic order above 2 K. Given that the magnetic order parameters near $T_N$ for the $x = 0.28, 0.29$ samples look remarkably like those of the spin cluster phase in electron-doped Ba(Fe$_{1-x}$T$_x$)$_2$As$_2$ near optimal superconductivity [22,23], we have carried out additional neutron scattering measurements on the $x = 0.29$ sample using TRISP, which can operate as a normal thermal triple-axis spectrometer with instrumental energy resolution of $\Delta E \approx 1$ meV and a NRSE triple-axis spectrometer with $\Delta E \approx 1 \mu$eV [36]. Figure 2(h) shows the triple-axis mode data which reproduces the results in Fig. 2(f). However, identical measurements using NRSE mode reveal that the magnetic scattering above 30.7 K is quasielastic and the spins of the system freeze below 30.7 K on a time scale of $\tau \sim \hbar/\Delta E \approx 6.5 \times 10^{-10}$ s [23]. This spin freezing temperature is almost identical to those of nearly optimally electron-doped Ba(Fe$_{1-x}$T$_x$)$_2$As$_2$ [21–23].

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other frequencies, suggesting that the magnetic-ordered data from Ref. [34] lost NMR spectral weight above because of radio frequency screening. We find that the factor, for single crystals with orthorhombicity in the temperature region of \(26 \leq T \leq 32.5\) K [filled circles in Fig. 3(b)] (Supplemental Material [37]). The open symbols represent \(\delta\) estimated from the enlarged half-width of single peak fits (Supplemental Material [37]). Although the data show a reentrant tetragonal phase and vanishing lattice orthorhombicity at low temperature, the presence of weak collinear AF order seen by neutron scattering [Figs. 2(c) and 2(d)] indicates that the AF-ordered parts of the sample should still have orthorhombic lattice distortion [19,22]. Figures 3(c) and 3(d) show temperature dependence of the longitudinal scans along the \([H,0,0]\) direction for the \(x = 0.29\) and 0.31 samples, respectively. While the lattice distortion in the \(x = 0.29\) sample behaves similarly to that of the \(x = 0.28\) crystal, there are no observable lattice distortions in the probed temperature range for the \(x = 0.31\) sample.

To further test the nature of the magnetic-ordered state in BaFe\(_2\)(As\(_{1-x}\)P\(_x\))\(_2\), we have carried out \(^{31}\)P NMR measurements under an 8-T \(c\)-axis-aligned magnetic field (Supplemental Material [37]). Figure 4(a) shows the temperature dependence of the integrated spectral weight of the paramagnetic signal, normalized by the Boltzmann factor, for single crystals with \(x = 0.25\) and 0.29. For \(x = 0.25\), the paramagnetic spectral weight starts to drop below 60 K and reaches zero at 40 K, suggesting a fully ordered magnetic state below 40 K. For \(x = 0.29\), the paramagnetic to AF transition becomes much broader, and the magnetic-ordered phase is estimated to be about 50% at \(T_c = 28.5\) K. Upon further cooling, the paramagnetic spectral weight drops dramatically below \(T_c\) because of radio frequency screening. We find that the lost NMR spectral weight above \(T_c\) is not recovered at other frequencies, suggesting that the magnetic-ordered phase does not take full volume of the sample, similar to the spin-glass state of BaFe\(_2\)(P\(_{1-x}\))\(_2\)As\(_2\) [21–23].

Figure 4(b) shows the P-doping dependence of the ordered moment squared \(M^2\) in BaFe\(_2\)(As\(_{1-x}\)P\(_x\))\(_2\) including data from Ref. [34]. While \(M^2\) gradually decreases with increasing \(x\) for \(x \leq 0.25\), it saturates to \(M^2 \approx 0.0025\mu_B^2\) at temperatures just above \(T_c\) for \(x = 0.28\) and 0.29 before vanishing abruptly for \(x \geq 0.30\). The inset in Fig. 4(b) shows the P-doping dependence of the \(M^2\) above and below \(T_c\) near optimal superconductivity. While superconductivity dramatically suppresses \(M^2\), it does not eliminate the ordered moment. Figure 4(c) shows the P-doping dependence of \(\delta\) in BaFe\(_2\)(As\(_{1-x}\)P\(_x\))\(_2\) below and above \(T_c\). Consistent with the P-doping dependence of \(M^2\) [Fig. 4(b)] and \(T_N\) [Fig. 1(c)], we find that \(\delta\) above \(T_c\) approaches \(\sim 3 \times 10^{-4}\) near optimal superconductivity before vanishing at \(x \geq 0.3\).

\(x = 0.19\), obtained by fitting the two Gaussian peaks in longitudinal scans along the (8,0,0) nuclear Bragg peak (Supplemental Material [37]). We find that the lattice orthorhombicity \(\delta\) exhibits a first-order-like jump below \(T_s = 72.5\) K consistent with previous neutron scattering results [34,37]. We also note that the lattice distortion value of \(\delta \approx 17 \times 10^{-4}\) is similar to those of Ba(Fe\(_{1-x}\)Ta\(_x\))\(_2\)As\(_2\) with \(T_s \approx 70\) K [19,22].
Summarizing the results in Figs. 2–4, we present the refined phase diagram of BaFe$_2$(As$_{1-x}$P$_x$)$_2$ in Fig. 1(c). While the present phase diagram is mostly consistent with the earlier transport and neutron scattering work on the system at low P-doping levels [30,34], we have discovered that the magnetic and structural transitions still occur simultaneously above $T_c$ for $x$ approaching optimal superconductivity, and both order parameters vanish at optimal superconductivity with $x = 0.3$. Since our NMR and TRISP measurements for samples near optimal superconductivity suggest spin-glass-like behavior, we conclude that the static AF order in BaFe$_2$(As$_{1-x}$P$_x$)$_2$ disappears in the weakly first-order fashion near optimal superconductivity. Therefore, AF order in phosphorus-doped iron pnictides coexists and competes with superconductivity near optimal superconductivity, much like the electron-doped iron pnictides with an avoided QCP. From the phase diagrams of hole-doped Ba$_{1-x}$Fe$_2$As$_2$ [8–11], it appears that a QCP may be avoided there as well.

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Note added.—We became aware of a theory preprint predicting the first order AF phase transition in BaFe$_2$(As$_{1-x}$P$_x$)$_2$ after the submission of this Letter [38].

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*Author’s note added*.

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