Muon-spin-relaxation studies of magnetic order and superfluid density in antiferromagnetic NdFeAsO, $BaFe_2As_2$, and superconducting $Ba_{1-x}K_xFe_2As_2$

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Zero-field (ZF) muon-spin-relaxation (μSR) measurements have revealed static commensurate magnetic order of Fe moments in NdFeAsO below $T_N \sim 135$ K, with the ordered moment size nearly equal to that in LaFeAsO, and confirmed similar behavior in $BaFe_2As_2$. In single crystals of superconducting $Ba_{0.55}K_{0.45}Fe_2As_2$, μSR spectra indicate static magnetism with incommensurate or short-ranged spin structure in $\sim$70% of volume below $T_N \sim 80$ K, coexisting with the remaining volume which exhibits a superfluid response below $T_c \sim 30$ K.

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I. INTRODUCTION

The discovery of the iron oxypnictide superconductor LaFeAs(O,F) ($T_c \sim 26$ K) (Ref. 1) has triggered an unprecedented burst of quality research. Notable progress includes synthesis of the "1111" materials with higher T_c's (Refs. 2–4) containing different rare-earth (RE) elements, hole doping,[5] and/or reduced oxygen content.[4] More recently, the "122" systems AFe_2As_2 (A=Ba,Sr,Ca) have also been synthesized and in some cases have been found to become superconductors by (A,K) substitution[6,7] or by application of pressure.[8] In characterization, one finds discoveries of commensurate antiferromagnetism in the parent compounds LaFeAsO (Ref. 9) and $BaFe_2As_2$ (Ref. 10) by neutron scattering and nearly linear scaling of the superfluid density with T_c by muon-spin relaxation (μSR).[11–14] Studies of the electronic phase diagrams of iron oxypnictides by neutron scattering[15] and μSR (Ref. 16) have revealed remarkable similarities with the cases of cuprate systems. Very recent success in the fabrication of superconducting $(Ba,K)Fe_2As_2$ and $(Sr,K)Fe_2As_2$ single crystals[17,18] has enabled detailed studies by angle-resolved photoemission spectroscopy (ARPES) (Refs. 19 and 20) and scanning tunneling microscopy (STM).[21] Finally, quantum oscillations[22] have been observed in undoped crystals of $SrFe_2As_2$.

Several controversial issues, however, remain open to further studies. Neutron measurements found antiferromagnetic order of Fe moments below $T \sim 135$ K in LaFeAsO (Ref. 9) and CeFeAsO (Ref. 15) with an ordered Fe moment size of $0.36\mu_B$ and $0.8\mu_B$, respectively. Bos *et al.* and Qiu *et al.*,[23] however, reported the absence of corresponding antiferromagnetic order in NdFeAsO. Such a drastic dependence on RE elements is surprising and has to be re-examined by other magnetic probes. Reports on pairing symmetry are divided between those favoring a gap with[21,24] or without[20,25] nodes. Extensive characterization of single-crystal specimens is very important in the early stage of materials development to ensure improved crystal quality in the future. To address these issues, we have performed μSR measurements on ceramic specimens of NdFeAsO and $BaFe_2As_2$, as well as on superconducting single crystals of $Ba_{0.55}K_{0.45}Fe_2As_2$. As reported in this work, our results demonstrate that the existence of antiferromagnetism in the former two systems is nearly identical to that of LaFeAsO. In the superconducting single crystals, we found coexisting signals due to incommensurate or short-ranged static magnetism from a partial volume fraction and to a superfluid response from the remaining volume.

II. EXPERIMENTAL PROCEDURES

Ceramic specimens of NdFeAsO and $BaFe_2As_2$, with dimensions of 8–10 mm in diameter and 1–2 mm thickness, were synthesized at the Institute of Physics (IOP) in Beijing following the methods published elsewhere.[7,26] Single crystals of $Ba_{0.55}K_{0.45}Fe_2As_2$ were prepared at Ames Laboratory, Iowa following the method in Ref. 17. Small crystals, with a typical size of $2\times2\times0.1$ mm^3, were aligned with their large faces (*ab* plane) parallel to each other to form a mosaic specimen of 65 mg in weight covering an area of $\sim$7 mm in diameter. μSR measurements were performed at TRIUMF, Vancouver, with the beam direction perpendicular to the large faces of the crystals, following a standard method described in Refs. 27–29.

III. EXPERIMENTAL RESULTS

In zero-field μSR measurements of NdFeAsO, long-lived and single-component muon-spin precession was observed

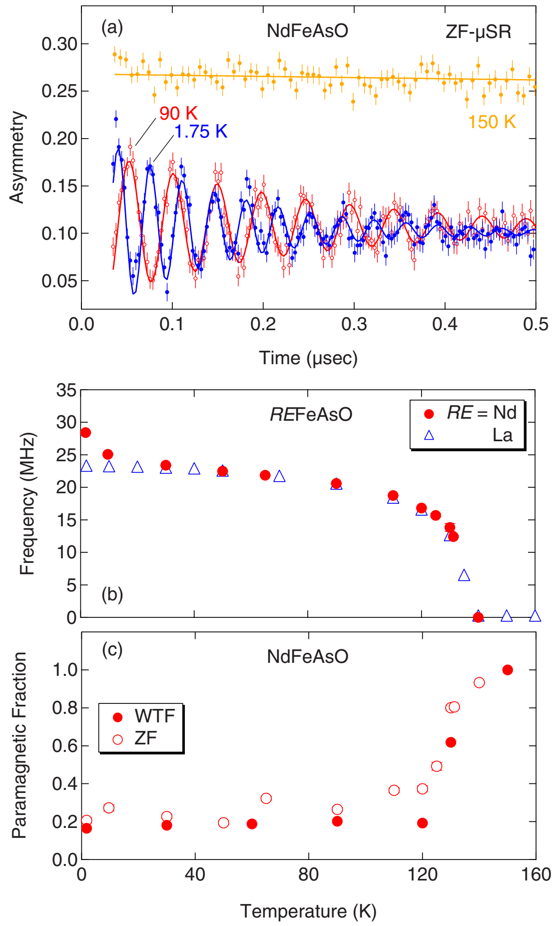

FIG. 1. (Color online) (a) Time spectra of ZF-μSR in NdFeAsO. (b) Temperature dependence of the precession frequency of ZF-μSR in NdFeAsO (present study) and LaFeAsO (Ref. 13). (c) Fraction of muons in a paramagnetic environment estimated from the precession amplitude in a WTF of 100 G, as well as from the amplitude of the nonoscillating signals in the ZF-μSR spectra.

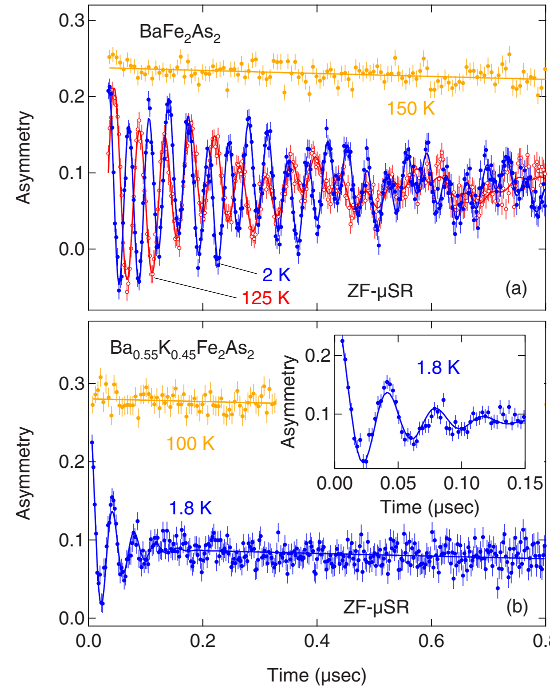

FIG. 2. (Color online) Time spectra of ZF-μSR in (a) a ceramic specimen of $BaFe_2As_2$ and in (b) a single-crystal mosaic of $Ba_{0.55}K_{0.45}Fe_2As_2$. The inset of (b) compares the observed spectra with a Bessel function multiplied by a small exponential damping (solid line). The long-lived oscillation signal in (a) indicates commensurate magnetic order, while the Bessel function shape in (b) suggests the possibility of an incommensurate or stripe spin structure.

below $T_N \sim 135$ K, as shown in Fig. 1(a). The precession amplitude corresponds to the value expected for $\sim(70-80)\%$ of the muons. The temperature dependence of the frequency, as derived by fitting to a damped cosine function, is shown in Fig. 1(b). The anomaly in the frequency below $T=5$ K is presumably due to magnetic ordering of Nd moments, which was detected by neutron scattering.[23] The frequency in NdFeAsO is nearly equal to that observed in LaFeAsO (Ref. 13) at $T>5$ K, indicating that the ordered Fe moments for these two systems are roughly of the same size for temperature regions unaffected by the Nd ordering. Figure 1(c) shows the fraction of muons in a paramagnetic environment derived from the precessing asymmetry observed in a weak transverse field (WTF) of 100 G and also from the ZF-μSR spectra. This figure indicates that a small fraction $[\sim(20-30)\%]$ of muons remains in the paramagnetic environment even below T_N, which could either be due to a minor paramagnetic volume fraction or to a possible cancellation of the static local field at the corresponding muon sites for symmetry reasons. Despite this uncertainty, the overall results in Fig. 1 clearly demonstrate the existence of commensurate antiferromagnetic order in most of the volume fraction of NdFeAsO below T_N.

Figure 2(a) shows ZF-μSR spectra in $BaFe_2As_2$. Below $T_N=140$ K, we find long-lived oscillations with two frequencies, so we fit these data to the sum of two damped cosine functions. As $T\to 0$, the main frequency from 80% of the muons approaches 28.8 MHz and the other frequency from 20% of the muons approaches 7 MHz, as depicted in Fig. 3(a). The two frequencies scale quite well with one another and so are likely due to the presence of two magnetically inequivalent muon sites in the unit cell of this system. The total volume fraction of the magnetically ordered region is essentially 100% below T_N [Fig. 3(b)]. Static magnetic order of this system was identified by Rotter *et al.*[30] who observed a Mössbauer hyperfine field H_{Moss} of 5.47 T as $T\to 0$ which corresponds to an ordered Fe moment of $0.4\mu_B$. In the 1111 parent system LaFeAsO, the Mössbauer field H_{Moss} was 4.86 T,[31] while the main frequency in μSR was 23.0 MHz as $T\to 0$.[13,31] The ratio of the main μSR frequencies $28.9/23.0=1.25$ of the 122 and 1111 systems is nearly equal to the ratio $5.47/4.86=1.13$ of H_{Moss}, indicating that the effective hyperfine coupling constants for muons in these two systems are within 15% of one another. Combined μSR, Mössbauer, and neutron results demonstrate that antiferromagnetism of $BaFe_2As_2$ is nearly identical to that of LaFeAsO and NdFeAsO in terms of T_N, spin structure,[9,10] ordered moment size, and (nearly full) ordered volume fraction.

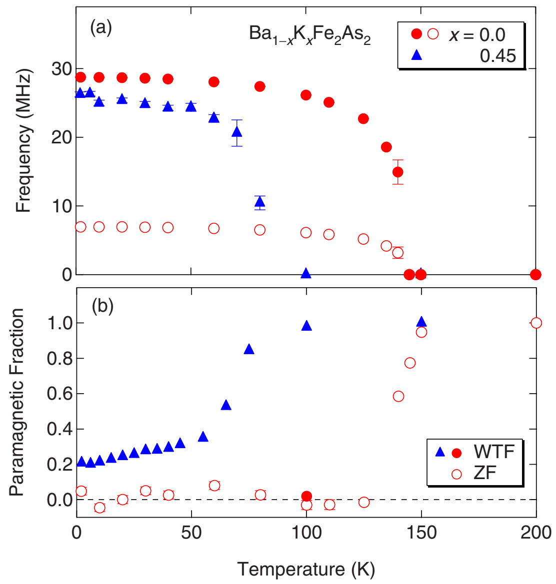

FIG. 3. (Color online) (a) Muon-spin precession frequency in zero field as determined by fitting to the sum of two damped cosine signals for polycrystalline $BaFe_2As_2$ and a single Bessel function for single-crystal $Ba_{0.55}K_{0.45}Fe_2As_2$. Note that our transition temperature for $BaFe_2As_2$ indeed corresponds to that expected for polycrystalline specimens rather than single crystals. (b) The fraction of the signal from muons in a paramagnetic/nonmagnetic environment measured in both a ceramic $BaFe_2As_2$ specimen and single crystals of $Ba_{0.55}K_{0.45}Fe_2As_2$. The paramagnetic fraction was derived as described in the caption of Fig. 1(c).

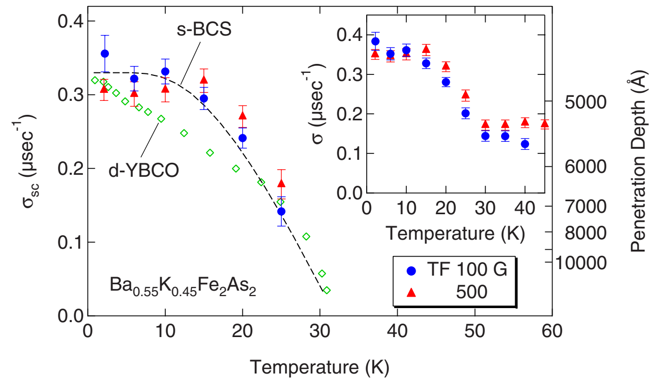

FIG. 4. (Color online) The relaxation rate σ_{sc} due to superconductivity in single crystals of $Ba_{0.55}K_{0.45}Fe_2As_2$, in transverse fields of 100 and 500 G parallel to the c axis, obtained after quadratically subtracting the background relaxation due to nuclear dipolar fields from the observed Gaussian relaxation rate σ shown in the inset. The temperature dependence is compared to BCS theory for isotropic s-wave pairing (broken line) and scaled results from the YBCO cuprate superconductor (Ref. 27) (small open diamond symbols).

In the K-doped single crystals $Ba_{0.55}K_{0.45}Fe_2As_2$, we observed ZF-$\mu$SR spectra with a damped precession signal below $T=70-80$ K, as shown in Fig. 2(b), only when the initial muon polarization was perpendicular to the c-axis direction. This implies the onset of a highly inhomogeneous static field, parallel to the c axis, at the muon site(s). Although the oscillating spectra can be fit either to a damped cosine or damped Bessel function, the Bessel fit [solid line in the inset of Fig. 2(b)] gives an initial phase $\phi \sim 7°$ at time $t=0$ consistent with the experimental condition, unlike the cosine fit with $\phi \sim -35°$ which is significantly off phase. This suggests the possibility of an incommensurate and/or stripe spin structure in the ordered region.[29,32,33] Note that a similar damped Bessel signal was observed also in 3% F-doped $LaFeAsO_{0.97}F_{0.03}$.[13]

Figure 3(a) shows the precession frequency in the K-doped crystals, which is close to that in undoped $BaFe_2As_2$. This indicates a gradual evolution of the spin configuration with K doping. The fraction of muons in a paramagnetic environment in Fig. 3(b), derived from the corresponding asymmetry of the μSR spectra in WTF and ZF, demonstrates that the magnetic order in $Ba_{0.55}K_{0.45}Fe_2As_2$ is detected by $V_\mu=(60-80)\%$ of the muons below $T\sim 60$ K. This implies that there is a volume fraction $V_M \sim (50-70)\%$ with static magnetism.[29]

The remaining paramagnetic signal allowed measurements of the superfluid response below $T_c \sim 30$ K in $Ba_{0.55}K_{0.45}Fe_2As_2$ single crystals. The inset of Fig. 4 shows the muon-spin-relaxation rate σ observed in transverse external fields of 100 and 500 G parallel to the c axis, obtained in a fit to a Gaussian damping $\exp(-\sigma^2 t^2/2)$. This relaxation is related to the magnetic-field penetration depth λ and n_s/m^* (superconducting carrier density/effective mass) as $\sigma \propto \lambda^{-2} \propto n_s/m^*$.[27,28,34,35] Limited counting statistics of the data due to a small amount of specimens, a small paramagnetic fraction, and slow relaxation prevented us from fitting the damping to a more complicated line shape calculated for an Abrikosov vortex lattice. The relaxation rate σ_{sc} related to superconductivity has been obtained by quadratically subtracting the background nuclear dipolar relaxation, determined at $T=30-40$ K, from the observed data. The temperature dependence of σ_{sc} in Fig. 4 is compared to the nodeless weak-coupling s-wave Bardeen-Cooper-Schrieffer (BCS) curve (broken line) as well as to scaled results from $YBa_2Cu_3O_7$ (YBCO) cuprates[27] (open diamonds) representing a d-wave gap with line nodes. The present results clearly exhibit better agreement with the case of a nodeless isotropic gap.

IV. DISCUSSIONS AND CONCLUSION

The observed $\sigma_{sc}(T\rightarrow 0) \sim 0.33$ μs^{-1} for the single-crystal specimens imply that the superfluid density in the present $Ba_{0.55}K_{0.45}Fe_2As_2$ specimens is about 30% of that in LaFeAs(O,F) with comparable T_c. Furthermore, using the conversion procedure outlined in Ref. 36, the penetration depth as $T\rightarrow 0$ is estimated to be 4700 Å for $Ba_{0.55}K_{0.45}Fe_2As_2$. The rather small superfluid density and a large volume with static magnetism suggest that this system consists of a mixture of regions with static magnetic order and superconductivity. This picture is consistent with the results of the specific-heat jump reported in Ref. 17, as well as with STM studies which found two distinct responses.[21]

Both structural and magnetic phase transitions at $T=85$ K were found in single crystals of undoped $BaFe_2As_2$ prepared with the Sn-flux method.[17] This is in contrast to the structural or magnetic phase transition at $T_N \sim 140$ K reported for polycrystalline $BaFe_2As_2$.[30] As outlined in Ref. 17, this discrepancy is likely due to the great sensitivity of this transition to the Sn flux used to grow single crystals. In any event, the magnetic order we are observing in the K-doped crystals below $T \sim 80$ K then suggests the possibility of a wide spatial spread of K concentrations in these crystals, even beyond the $\pm 7\%$ layer-by-layer spread around the 45% nominal K concentration found in Ref. 17. At this moment, however, it is not clear whether the superconducting and magnetically ordered regions are phase separated on a microscopic scale, similarly to oxygen-overdoped $La_{2-x}Sr_xCuO_4$ (LSCO) cuprates,[29] or on a more macroscopic length scale.

The present studies are made on the first set of superconducting single crystals ever produced among the bilayer iron-based systems. Therefore, the results should be received with caution that improved quality, homogeneity, and size of the specimen and data with improved statistics could possibly alter the essential message, as has been experienced for the cases of cuprate systems. Within such limitations, however, the present results indicate (a) decent superfluid response of the crystals, (b) the possibility of intrinsic microscopic coexistence of regions with and without static magnetic order, (c) a nodeless energy gap, which might be intrinsic or be related to the scattering of the carriers due to the magnetic fraction, and (d) the possibility of the involvement of incommensurate or stripe spin correlations near the border of the antiferromagnetic and superconducting states.

Our μSR results on NdFeAsO are clearly inconsistent with the neutron studies of NdFeAsO (Ref. 23) which found an absence of corresponding Fe ordering. Neutron measurements on $BaFe_2As_2$ (Ref. 10) and LaFeAsO (Ref. 9) estimate the size of the ordered Fe moment to be $0.87\mu_B$ and $0.36\mu_B$, respectively. In contrast, μSR and Mössbauer studies found a comparable moment size in these two systems. The origin of these disagreements is unclear at this moment. For the estimate of ordered moment size, however, local probes such as μSR and Mössbauer generally provide more accurate information than the volume-integrated Bragg-peak intensity of neutron scattering.

In summary, our μSR measurements of Fe-based high-T_c systems have revealed static magnetic order of NdFeAsO, demonstrated similar magnetic behavior in parent compounds of monolayer and bilayer systems NdFeAsO, LaFeAsO, and $BaFe_2As_2$, and elucidated coexisting static magnetism and superconducting responses in single crystals of $Ba_{0.55}K_{0.45}Fe_2As_2$.

Note added. In parallel to when this work was submitted for publication, a paper appeared on the cond-mat server which is now published.[37] This work describes neutron measurements of NdFeAsO which provide evidence for the ordering of the Fe moments with the moment size of $0.26(7)\mu_B$ per Fe in this material. The present work is qualitatively consistent with this result.

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