Magnetic and Superconducting Properties of Underdoped Ba(Fe\(_{1-x}\)Co\(_x\))\(_2\)As\(_2\) Measured with Muon Spin Relaxation/Rotation

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(Dated: August 19, 2014)

We report muon spin relaxation/rotation (\(\mu\)SR) measurements of single crystal Ba(Fe\(_{1-x}\)Co\(_x\))\(_2\)As\(_2\) with \(x=0.038\) and 0.047. Zero field (ZF)-\(\mu\)SR and Transverse field (TF)-\(\mu\)SR measurements of these underdoped samples find the presence of magnetism and superconductivity. We find internal fields along the \(c\)-axis whose magnitude decreases with increasing doping. We find evidence for a low-temperature volume fraction that is only weakly magnetic, where that volume fraction increases with increasing Co doping of the sample. TF-\(\mu\)SR measurements show slight changes in the spectra that indicate magnetic inhomogeneities due to the loss of Fe moments in the system, the effect of which is larger in the higher Co doping. We discuss the existence of superconductivity in these samples in close proximity to strong magnetic order.

PACS numbers: 76.75.+i

I. INTRODUCTION

Of the various families of iron pnictide superconductors, the so-called 122 family has been extensively studied due to their high \(T_{C}\)'s and the ability to grow relatively large single crystals, which includes BaFe\(_2\)As\(_2\), SrFe\(_2\)As\(_2\) and CaFe\(_2\)As\(_2\). Unlike the case of the cuprates, superconductivity in these materials is apparently quite robust against in-plane disorder, brought about by electron-doping for Fe atoms either by Co, Ni or other transition metals. The transition temperatures remain fairly high for these substitutions, with \(T_{C} = 22\) K for Ba(Fe\(_{0.926}\)Co\(_{0.074}\))\(_2\)As\(_2\) \(^1\)\(^2\), 20.5 K for Ba(Fe\(_{0.952}\)Ni\(_{0.048}\))\(_2\)As\(_2\) \(^3\), 23 K for Ba(Fe\(_{0.9}\)P\(_{0.1}\))\(_2\)As\(_2\) \(^4\), 14 K for Ba(Fe\(_{0.961}\)Rh\(_{0.039}\))\(_2\)As\(_2\) \(^5\), 19.5 K for Sr(Fe\(_{0.8}\)Co\(_{0.2}\))\(_2\)As\(_2\) \(^6\), 9.5 K for Sr(Fe\(_{0.925}\)Ni\(_{0.075}\))\(_2\)As\(_2\) \(^7\) and 12 K for Ca(Fe\(_{0.972}\)Co\(_{0.028}\))\(_2\)As\(_2\) \(^8\).

Like the cuprate superconductors, these materials exist in close proximity to magnetism. However, the nature of the competition or coexistence of magnetism and superconductivity in the pnictides has been debated, particularly since there may be different behaviours in different families \(^9\). This continues with the nature of the gap symmetry which also may vary across different families \(^10\) and where the temperature-dependence of the superfluid density raises the possibility of multi-band superconductivity \(^11\) \(^12\).

II. EXPERIMENTAL

Muon spin rotation (\(\mu\)SR) is a powerful local microscopic tool for characterizing the magnetic properties of materials in superconducting or other states. A thorough description of the application of \(\mu\)SR to studies of superconductivity can be found elsewhere \(^13\). Each implanted muon spin precesses around the local magnetic field until the muon decays into a positron, which is preferentially ejected along the direction of the muon spin at the time of decay (as well as two neutrinos which are not detected). Due to the large gyromagnetic ratio of the muon, local fields as small as 0.1G can be detected.

Single crystals of Ba(Fe\(_{1-x}\)Co\(_x\))\(_2\)As\(_2\) with \(x=0.038\) and 0.047 were grown at Ames from self flux as described in detail elsewhere \(^2\). The crystals, each of roughly 1 cm\(^2\) area, were mounted in a helium gas flow cryostat on the M15 and M20 surface muon beamlines at TRIUMF, using a low background arrangement such that only positrons originating from muons landing in the specimens were collected in the experimental spectra.

III. MAGNETISM AND COEXISTENCE

Using Zero Field (ZF)-\(\mu\)SR, magnetic fields at the muon site can be measured through precession of the muon spins in the local field. The presence of magnetic order in the samples is manifested as an oscillation in the ZF-\(\mu\)SR spectra, as was the case in the samples measured here. Characteristic spectra for the samples with \(x=0.038\), 0.047 and the parent material, BaFe\(_2\)As\(_2\), at \(T=1.65\) K are shown in Fig. 1, with the spectra vertically offset for clarity. Here and elsewhere in this work, the data for the parent compound was originally reported in Ref. 14. Motivated by previous studies of the underdoped and parent compounds in which two muon sites have been observed \(^14\) \(^15\), as well as DFT calculations...
supporting two electrostatic minima in the crystal structure [16], the ZF-µSR data was fit to a fitting function:

$$A(t) = A_1 \cos(\omega_1 t) e^{-\lambda_1 t} + A_2 \cos(\omega_2 t) e^{-\lambda_2 t} + A_3 e^{-\lambda_3 t}$$

(1)

This is a model containing two precessing muon signals and a relaxing, non-precessing signal. Furthermore, a single damped exponential did not fit the data well. In Fig. 1 we can see the clear precession indicative of well-ordered magnetism that is present in the parent material is not long-lived in the doped samples. The highly damped precession indicates a broad distribution of local fields at the muon sites, indicating a substantial degree of disorder in the magnetic ordering.

![Graph showing asymmetry as a function of time for different Co dopings.](image)

**FIG. 1**: (Color online) The zero-field (ZF)-µSR spectra taken at T=1.65K for the parent (x=0, blue), x=0.038 (red) and x=0.047 (black) samples. The x=0.038 data is vertically offset by +0.15 and the x=0 data is vertically offset by +0.3 for clarity. The solid lines are the fits to the data, as described in the text. Here, we see clear precession in the parent material, but the precession in the doped compounds is strongly damped, indicating significantly disordered magnetism.

The precession frequencies, $\omega_1$ and $\omega_2$, extracted from this model are shown as a function of temperature in Fig. 2. The onset of the magnetic ordering was estimated to be 71(3)K for Ba(Fe$_{0.962}$Co$_{0.038}$)As$_2$ and 45(2)K for Ba(Fe$_{0.953}$Co$_{0.047}$)As$_2$, in excellent agreement with transport and scattering measurements [2] [17].

In these measurements, we find that the magnitudes of the two frequencies scale with one another, which is evidence for two inequivalent muon sites with different local fields. This scaling would not be expected in the case of muons landing in different regions of the sample with distinct environments, such as multiphase samples. The scaling dependence was found during the initial analysis and so the frequencies and asymmetries of the two signals were constrained to be a constant multiple of one another. These ratios were fit to a temperature-independent value using the whole temperature range below $T_N$. The frequency ratio was found to decrease with doping, being 0.24(1) for the parent material, 0.20(4) at 3.8% Co doping and 2.3(4) $\times$ 10^{-3} at 4.7%. The decrease in the ratio may reflect the growing Co concentration, which distorts the magnetically-ordered lattice and the electrostatic potential at the muon site [18]. At the 4.7% doping, the extremely small value of the ratio of high- and low-field sites may represent the nearly complete loss of long-range order, so that all sites appear to have different local fields, consistent with the data from Fig. 1 where the oscillations disappear within the first 0.1-0.2$\mu$s. The frequencies observed in our measurements are lowest in the 4.7% sample, while both are lower than for the parent compound [14]. This indicates that the average magnetic field at both muon sites decreases with increasing doping. This could be due to the replacement of Fe moments with Co atoms, which reduces the size of the internal field. The replacement of Fe moments with Co atoms may also cause disorder in the local field, leading to a high muon relaxation rate in the ZF-µSR measurements. As seen in Fig. 1, the relaxation rates of the oscillating components are highest in the 4.7% sample and slightly lower in the 3.8% sample, while the oscillations in the parent material persist to much longer times. This is in agreement with other work on the underdoped Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ compounds, where the precession signals observed are fairly short-lived, indicating that the magnetic ordering in these samples is most likely strongly disordered or incommensurate [15]. Incommensurate magnetic order has been observed by neutron scattering in dopings above $x=0.056$ [22].

![Graph showing frequency as a function of temperature.](image)

**FIG. 2**: (Color online) (a) ZF-µSR spectra for Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ with x=0.038 ($T_N=71$K and $T_C=8$K) and x=0.047 ($T_N=45$K and $T_C=15$K). There is a clear onset at $T_N$, where the precession frequency becomes non-zero. There is no significant change below $T_C$.

When ZF-µSR spectra are measured with the initial muon polarization in the direction of the local field, $\vec{P}_\mu \parallel \vec{H}_{loc}$, the spectra do not relax to zero asymmetry in the ordered region. This behaviour was observed...
when the direction of the initial muon spin was parallel to the crystallographic \( \hat{c} \) direction. This was verified by measuring ZF-\( \mu \)SR spectra in an orientation in which the muon spin is perpendicular to the \( \hat{c} \)-axis, \( \vec{P}_c \perp \vec{H}_{loc} \), where the spectra do relax to zero at long times. The fits of the ZF-\( \mu \)SR spectra give internal fields at the high-field muon site to be approximately 0.13T in \( x=0.038 \) and 0.11T in \( x=0.047 \). This is consistent with other studies of \( \text{Ba(Fe}_{1-x}\text{Co}_x)\text{As}_2 \) that have found strong internal fields (\( >0.15T \)) along the \( \hat{c} \)-axis in various dopings, from the parent compound up to the loss of magnetic order [14].

We also measured these samples in a transverse field (TF) of 4mT to probe the paramagnetic volume fraction. In a weakly magnetic or paramagnetic region the muon ensembles see a local field equal to the applied field, \( H_{app} \), so it can be fit to a simple exponentially-damped cosine:

\[
A(t) = A \cos(\omega t) e^{-\lambda t} \tag{2}
\]

where \( \omega = \gamma_\mu H_{app} \).

Below a magnetic ordering transition, the local field becomes a vector sum of the ordered and external fields, so the muons no longer precess at the frequency given by the external field. The amplitude of the measured signal, \( A \), of the precessing signal decreases, though any para- or weakly-magnetic region will still precess at this frequency. This allows TF-\( \mu \)SR to be used for measurements of the magnetic volume fractions in magnetically-ordered materials. In these samples, we find that the volume fraction of the magnetically-ordered region increases sharply below \( T_N \) (as shown in Fig. 3(c)), saturating within 15K of the ordering temperature. We see that in the 4.7% sample, there is a residual signal that still precesses down to the lowest temperatures measured, but has a relaxation that increases below \( T_N \) indicating that it is weakly magnetic. This low-field region is less than 10% in the \( x=0.038 \) sample, while it is approximately 40% for \( x=0.047 \). This is an indication that the fraction of the sample that sees weak magnetism increases with increasing Co concentration.

Fig. 3(a) and (b) show the precession frequencies, \( \omega \), and relaxation rates, \( \lambda \), present in the TF-\( \mu \)SR spectra. The relaxation rate measures the rate of the depolarization of the muon spins, which may be caused by inhomogeneities in the local magnetic field among other factors. We see in Fig. 3(a) that the frequency shows a decrease below the magnetic ordering transition. This can be attributed to the antiferromagnetic order that emerges and causes inhomogeneities in the local field; this is significant evidence for disordered magnetism at these dopings. This inhomogeneity in the local field is also seen in the TF-\( \mu \)SR relaxation rates (Fig. 3(b)) that show a sharp increase at the magnetic ordering transition. The relaxation rates saturate, and remain relatively constant down to \( T = 0 \). When this is taken together with the ZF and with the TF-\( \mu \)SR spectra in different orientations, we find a magnetic structure that displays a large internal field oriented along the \( \hat{c} \)-axis. As was seen with \( \mu \)SR measurements of the parent material [14], these samples see magnetism everywhere, but in the doped samples the magnetism is highly disordered. This is in agreement with NMR [15, 19] and Mössbauer [20] measurements that find disordered, possible incommensurate magnetic order in the underdoped \( \text{Ba(Fe}_{1-x}\text{Co}_x)\text{As}_2 \) compounds.

This is somewhat in contrast to neutron scattering measurements that see well-defined magnetic scattering, which continues to be sharp below \( T_C \) [21]. Neutron scattering also sees incommensurate magnetic order at higher dopings, for \( x=0.056 \) to \( x=0.060 \) [22]. This incommensuration depends on composition, though the magnetic Bragg peaks remain well-defined. The fact that neutron scattering sees a well-ordered magnetic structure may be due to fluctuations in the local magnetism on timescales slower than that detectable by neutron scattering, but observable by \( \mu \)SR. This is in agreement with other techniques that operate on similar timescales, such as Mössbauer (\( \sim 10^{-7}s \)) and NMR (\( \sim 10^{-5}s \)). Incom- mensuration has been observed at dopings near the loss of antiferromagnetic order with a very small splitting [22]. In lower doped samples, such as those in this study, it is possible that the incommensuration is too small to be observed by neutron scattering but results in disorder in the local field, consistent with what has been observed in the Ni-doped system [23]. Another possible explanation is that the magnetic moments are a superposition of an ordered and a disordered component. While neutron scattering is sensitive only to the ordered component, muons would see both components, resulting in a largely disordered local field. This is consistent with neutron measurements of \( \text{Ca(Fe,Co)}_2\text{As}_2 \), which find a decrease in the size of the Fe ordered moment with increasing Co-doping [24, 25]. In the latter two cases, the disorder would increase with increasing Co-doping, as is seen in both ZF- and TF-\( \mu \)SR relaxation rates.

Below the superconducting transition, we see only small changes in the TF-\( \mu \)SR parameters. We see a slight decrease in the relaxation rate in the 4.7% sample below \( T_C = 15K \), while we do not have enough data points around and below \( T_C = 8K \) in the 3.8% sample to see any changes in behaviour. By using a higher transverse field of 0.02T, we find more direct evidence of superconductivity. This was measured at \( T = 1.65K \) for both field-cooled (FC) and zero field-cooled (ZFC) orientations. Compared to the FC data, the ZFC spectrum displayed a strongly increased increase in the relaxation, indicative of flux pinning, clearly indicating that we have superconductivity in these samples. Superconductivity may exist in the low-field regions since this region exhibits a growing volume fraction with increasing doping and by \( x=0.061 \) there is no static magnetism present [26]. The entire sample displays magnetism but some regions have small local fields, so there may be phase separation on the nanoscale with superconductivity existing in or near the magnetic regions of the sample. This is in agreement with scanning tunnelling spectroscopy (STS) results, that see no phase separation on length scales larger than tens
of nanometers\textsuperscript{27}.

IV. CONCLUSIONS

We have measured single crystals of Ba(Fe\textsubscript{1-x}Co\textsubscript{x})\textsubscript{2}As\textsubscript{2} that show both magnetism and superconductivity using Zero-Field and Transverse-Field muon spin relaxation/rotation. These measurements demonstrate the clear onset of a local magnetic field below $T_N$, largely along the $\hat{c}$-axis, the magnitude of which decreases with increasing Co doping.

TF-\textmu SR measurements suggest some inhomogeneity in the local field, where the disorder is greater in the 4.7\% Co doping from less than 10\% in BaFe(0.963Co0.038)\textsubscript{2}As\textsubscript{2} to 40\% in BaFe(0.953Co0.047)\textsubscript{2}As\textsubscript{2}. A possible source of this disorder may be the loss of Fe moments, which results in lower internal fields in the higher doped sample. These measurements are consistent with other techniques that suggest an incommensurability of the magnetic order that increases with Co-doping. This is reflected in the ZF-\textmu SR measurements, that show reduced precession frequencies and increased relaxation rates at higher dopings, suggesting lower internal fields and increased magnetic disorder.

Measurements in a larger transverse field of 0.02T display flux pinning and superconductivity, which may display nanoscale phase separation existing in the low-field regions of the sample. However, there is no detectable change in the local magnetic field or magnetic volume fractions below $T_C$. This suggests that the entire sample sees some magnetic order, indicating that the superconductivity exists in or near regions of large local magnetic order.

The authors acknowledge helpful earlier work on this project done by N. Ni. We also appreciate the hospitality of the TRIUMF Centre for Molecular and Materials Science where the majority of these experiments were performed. Research at McMaster University is supported by NSERC and CIFAR. Work at Columbia was supported by NSF-DMR-0502706 and NSF-DMR-0806846. Work by P.C.C. and S.L.B. at Ames Laboratory was supported by the Department of Energy, Basic Energy Sciences under Contract No. DE-AC02-07CH11358.

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FIG. 3: (Color online) TF-µSR measurements of x=0.038 and 0.047 in TF=4mT. (a) The frequency shows a drop below the magnetic ordering temperature, but slowly increases again towards T=0. This may be indicative of field-induced ordering. (b) The relaxation rate increases sharply below T_N, saturating well before T=0. This indicates that the local field is fairly inhomogeneous in these samples, which causes dephasing of the muon spin polarization in the ordered part of the phase diagram. (c) We see a 100% paramagnetic signal above T_N, which drops sharply at the transition. Below the ordering temperature, there is a residual paramagnetic signal, about 40% for x=0.047 and less than 10% for x=0.038. Due to the small residually-precessing volume fraction present in the 3.8% sample, the points below 50K are omitted from (a) and (b).