

Tuning of competing magnetic and superconducting phase volumes in $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$ by hydrostatic pressure

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(Received 5 July 2011; published 1 September 2011)

The interplay between magnetism and superconductivity in $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$ was studied as a function of hydrostatic pressure up to $p \simeq 2.4$ GPa by means of muon-spin rotation (μSR) and magnetization measurements. The application of pressure leads to a substantial decrease of the magnetic ordering temperature, reduction of the magnetic phase volume and, at the same time, to a strong increase of the superconducting transition temperature and the diamagnetic susceptibility. From the volume-sensitive μSR measurements it can be concluded that the superconducting and the magnetic areas, coexisting in the same sample, are inclined toward spatial separation and compete for phase volume as a function of pressure.

DOI: 10.1103/PhysRevB.84.100501

PACS number(s): 74.70.Xa, 74.25.Ha, 74.62.Fj, 76.75.+i

The interplay between superconductivity and magnetism in high-temperature superconductors (HTSs) remains an important open issue. In cuprate and Fe-based HTS the superconductivity can be induced in a magnetic parent compound by charge doping and/or by pressure (chemical or external). In most cuprate HTSs such a transformation follows an almost common scenario. On increasing the doping level the antiferromagnetically ordered phase develops into a purely superconducting state through a region where a spin-glass type of magnetism coexists with superconductivity.^{1,2} The situation with Fe-based HTSs is, somehow, different. For some families of Fe-based HTSs, such as, e.g., $\text{SmFeAsO}_{1-x}\text{F}_x$, $\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}$, and $\text{FeSe}_{1-x}\text{Te}_x$, the magnetism is continuously suppressed and superconductivity is enhanced by changing the F, Co, or Se content. In the intermediate region, bulk magnetism and bulk superconductivity are coexisting in space.^{3–5} In $\text{Ba}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2$ the magnetic and the superconducting areas are found to be separated microscopically as revealed, e.g., by atomic force microscopy and muon-spin rotation (μSR) experiments.^{6,7}

One of the most interesting cases is realized in the $\text{LaFeAsO}_{1-x}\text{F}_x$ family of Fe-based HTSs demonstrating an *abrupt* (first-order-like) transition between the magnetic and the superconducting phases. μSR and Mössbauer experiments show that above a certain x the samples become purely superconducting without visible traces of magnetism.⁸ Such a behavior seems to be rather different from the one observed for the other structurally related families of Fe-based HTSs in which the La atom is replaced by Sm, Pr, Ce, etc. All of them demonstrate a coexistence between superconductivity and magnetism for a certain doping level.^{3,9} Consequently, the question if a similar coexistence is present in $\text{LaFeAsO}_{1-x}\text{F}_x$ but within a much narrower, up to now not detected, doping region or if an abrupt change between the superconductivity and magnetism is a unique property of this particular family of Fe-based HTS needs to be resolved.

Hydrostatic pressure experiments on $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$, which is at the border to the superconducting state but still magnetic, were performed to distinguish between the two above-mentioned possibilities. This approach allows to follow the transformation of the material from the magnetic to the superconducting state in detail on one sample, i.e., without the necessity to synthesize a large number of samples with exactly defined stoichiometry near the phase boundary. Our measurements show that both the magnetic and superconducting states are most probably spatially separated in the crossover region of the phase diagram and compete for phase volume.

The sample with the nominal composition $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$ was prepared in a cubic anvil high-pressure cell from the stoichiometric mixture of LaAs, FeAs, Fe_2O_3 , Fe, and LaF_3 .^{10,11} A pressure of $\simeq 3$ GPa was applied at room temperature. By keeping the pressure constant, the temperature was first ramped up to a maximum value of 1320 °C, kept constant for 5.5 h, and then quenched to room temperature within a few minutes. X-ray measurements confirm the single-phase nature of the sample as well as the absence of a suitable amount of impurities.

The superconducting properties of $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$ were studied by magnetization experiments. The zero-field-cooled and field-cooled (FC and ZFC) dc magnetization measurements up to $p \simeq 1.1$ GPa were performed by using the commercial superconducting quantum interference device (SQUID) magnetometer (MPMS-XL7) and a piston-cylinder CuBe pressure cell (“EasyLab Mcell 10”). The ac experiments up to $p \simeq 2$ GPa were performed by using the cell made from MP35N alloy and a home-made ac magnetometer (ac frequency $\nu = 72$ Hz, ac field amplitude $\mu_0 H_{ac} \simeq 0.1$ mT). As the pressure transmitting medium “Daphne oil 7373” was used. This oil solidifies at $p \simeq 2.2$ GPa at room temperature.¹² Since the pressure was always applied at room temperature and most of the experiments presented in this Rapid Communication were performed below 2.2 GPa, we may assume that the pressure conditions were always nearly hydrostatic.

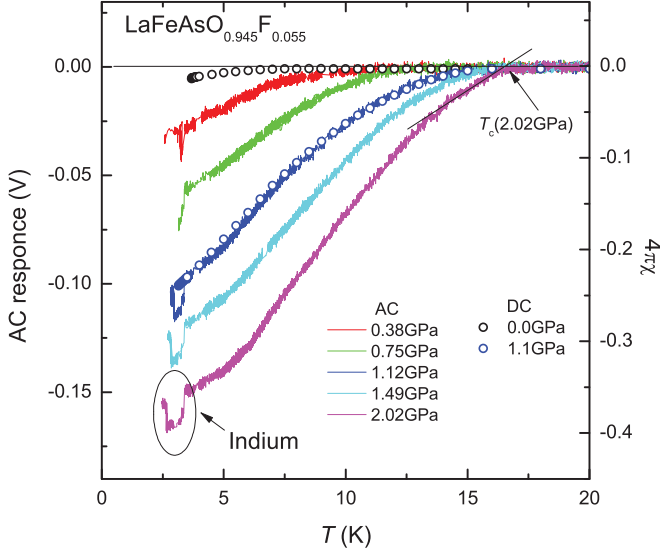


FIG. 1. (Color online) ac (solid curves) and ZFC $\mu_0 H = 5$ mT dc (open symbols) susceptibility curves as a function of temperature for different pressures of $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$. The transition temperature T_c is determined by the intersect of the linearly extrapolated magnetization curve with the zero line. The superconducting transitions of pure indium used as a pressure indicator in the ac magnetization experiment is highlighted by the oval for the highest pressure.

The results of the magnetization studies are presented in Fig. 1. At ambient pressure the diamagnetic susceptibility at $T \simeq 3.5$ K [$\chi(3.5 \text{ K})$] reaches $\sim 1\%$ of its ideal value ($\chi_{\text{id}} = -1/4\pi$). This suggests that the superconductivity at $p = 0.0$ is just filamentary and is present only within a small volume of the sample. With increasing pressure, both the onset temperature of the superconducting transition T_c [determined from the intersect of the linearly extrapolated $\chi(T)$ in the vicinity of T_c with the zero line—see Fig. 1] and the low-temperature value of the diamagnetic response ($-4\pi\chi$) increase quite substantially. According to Fig. 1, the increase of the external pressure from $p = 0.0$ to 2.02 GPa leads to the shift of T_c from $\simeq 7$ to $\simeq 16$ K and to an increase of $-4\pi\chi$ from $\sim 1\%$ to $\sim 35\%$. An additional set of dc magnetization measurements at $p \simeq 1.1$ GPa show that the diamagnetic response reduces by a factor of 2 for an applied field of 1 mT in ZFC and to a value of $4\pi\chi \simeq -0.05$ in $\mu_0 H = 5$ mT FC experiments, respectively (both are not shown). Such differences, which could be caused by the effect of pinning and the presence of weak links between the superconducting areas, do not permit a reliable evaluation of the genuine superconducting volume. One may only conclude that the superconducting volume fraction increases with increasing pressure but is always smaller than the whole sample volume.

The magnetic properties of $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$ were studied in zero-field (ZF) muon-spin rotation experiments. Pressures up to $\simeq 2.4$ GPa were generated in a double-wall piston-cylinder MP35N cell. Few representative muon-time spectra measured at $p = 0.0$ and 2.36 GPa are shown in the insets of Fig. 2. The data were analyzed by decomposing the signal on the contribution of the sample and the pressure cell as

$$A(t) = A_S(0) P_S(t) + A_{PC}(0) P_{PC}(t). \quad (1)$$

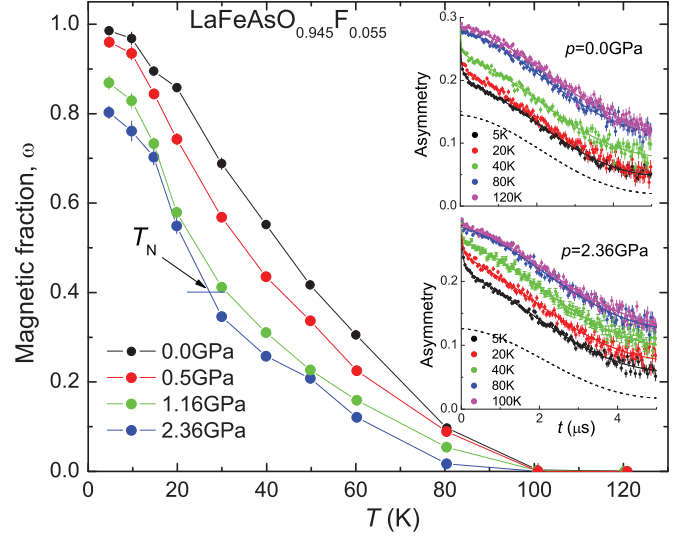


FIG. 2. (Color online) Temperature dependence of the magnetic volume fraction of $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$ at various pressures. The magnetic ordering temperature T_N is defined as the temperature where the magnetic fraction reaches 50% of its maximum low-temperature value. The insets show the ZF muon-time spectra at $p = 0.0$ and 2.36 GPa. The solid lines are fits by means of Eq. (1). The dotted lines represent the response of the pressure cell.

Here $A_S(0)$ and $A_{PC}(0)$ are the initial asymmetries and $P_S(t)$ and $P_{PC}(t)$ are the muon-spin polarizations belonging to the sample and the pressure cell, respectively. $P_{PC}(t)$ was measured in an independent experiment. The response of the sample was assumed to consist of a magnetic and a nonmagnetic contribution and is described as

$$P_S(t) = \omega \left[\frac{1}{3} e^{-\Lambda_{m,l}t} + \frac{2}{3} \{ \zeta e^{-\Lambda_{m,t1}t} + (1 - \zeta) e^{-\Lambda_{m,t2}t} \} \right] + (1 - \omega) e^{-\Lambda_{pm}t}.$$

Here ω is the relative weight (volume) of the magnetic fraction. $\Lambda_{m,l}$, $\Lambda_{m,t1}$, and $\Lambda_{m,t2}$ are the exponential depolarization rates representing the longitudinal (1/3) and the transversal (2/3) relaxing components within the parts of the sample being in the magnetic state. The two components within the curly brackets account for contributions of two different muon stopping sites with the relative weights ζ and $(1 - \zeta)$, respectively. As follows from Ref. 13, one site is located on the line connecting the La and As ions along the c direction, while the second one is located near the oxygen ions. Λ_{pm} is the relaxation within the parts of the sample remaining nonmagnetic. The exponential character of this relaxation, instead of the normally expected Kubo-Toyabe kind of behavior,¹⁴ is probably caused by the presence of the small amount of magnetic impurities, similar to the one observed in the so called “11” family of Fe-based HTS (see, e.g., Refs. 4 and 15). For each particular pressure the whole set of the data was fitted simultaneously with $A_S(0)$, $A_{PC}(0)$, ζ , and the ratio $\Lambda_{m,t1}/\Lambda_{m,t2}$ as common and ω , $\Lambda_{m,l}$, $\Lambda_{m,t1}$, and Λ_{pm} as individual parameters for each temperature point. The solid lines in the insets of Fig. 2 represent the result of the fit. The contribution from the cell at $T = 5$ K is shown as a dotted line.

The main panel of Fig. 2 shows the dependence of the magnetic fraction ω on temperature for $p = 0.0, 0.5, 1.16$, and

2.36 GPa. Two important points need to be considered. First, the magnetic volume fraction at each particular temperature is lowered by the application of pressure. Most noteworthy, with increasing pressure an increasingly large part of the sample remains in the paramagnetic state down to the lowest temperatures. Second, the magnetic ordering temperature T_N , defined as the temperature where the magnetic fraction reaches 50% of its maximum low-temperature value, initially decreases with increasing pressure but then demonstrates a tendency to saturate. $\omega(T)$ curves at $p = 1.16, 1.92$ (not shown), and 2.36 GPa being normalized to their values at $T \simeq 5$ K become almost identical.

In order to compare the influence of the pressure on the superconducting and the magnetic properties of $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$, the dependences of T_N , T_c , ω , and $4\pi\chi$ as a function of p are plotted in Fig. 3. The decrease of T_N and ω is associated with the corresponding increase of T_c and $4\pi\chi$. By applying a pressure of 2.36 GPa, T_N decreases from 44 to 27 K, while T_c more than doubles from $\simeq 7$ to $\simeq 16$ K upon the application of 2.02 GPa.

It should be noted here that the above presented data are pointing to a competition of superconductivity and magnetism, but alone do not allow to answer the question on how these two forms of order coexist within the $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$ sample. There are three possible scenarios. The first one is

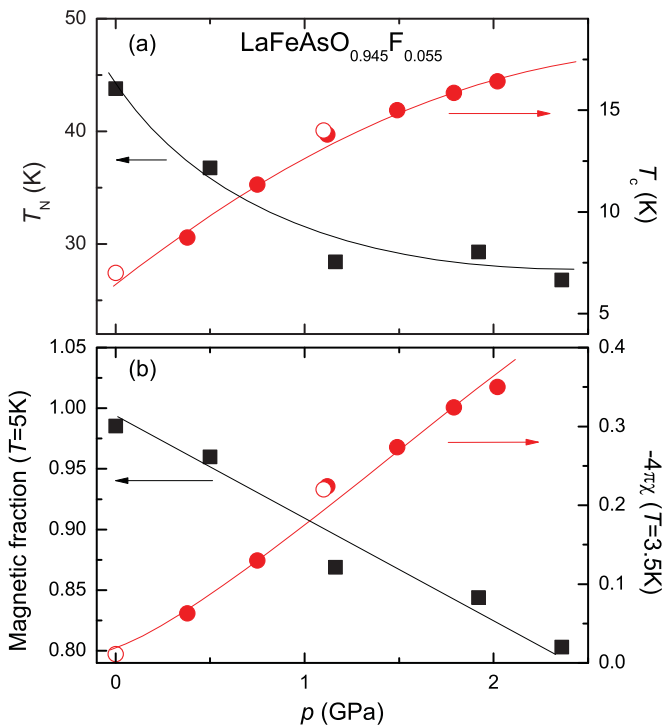


FIG. 3. (Color online) (a) Dependence of the magnetic ordering temperature T_N and the superconducting transition temperature T_c on pressure. The closed and the open circles correspond to T_c as obtained in ac and dc magnetization experiments, respectively. (b) The magnetic fraction at $T = 5$ K and ZFC diamagnetic susceptibility $-4\pi\chi$ at $T = 3.5$ K, $\mu_0 H = 5$ mT as a function of pressure. The closed and the open circles refer to the data obtained in ac and dc magnetization experiments, respectively. The lines are a guide for the eye.

the so-called *phase separation* scenario according to which the superconductivity develops just within the parts of the sample remaining nonmagnetic down to low temperatures. Such a phase-separated coexistence was observed, e.g., in $\text{Ba}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2$.^{6,7,16} The second possibility is an *atomic* coexistence of the superconducting and magnetic order parameters, which is consistent with models proposed in Ref. 17 and most probably realized within the so-called “11” family of Fe-based HTSs.^{4,18} The third possibility is a *nanoscale* segregation into magnetic domains, similar to that reported for cuprate HTSs.^{2,19} In underdoped cuprate HTSs, static, short-range, striplike magnetic correlations are thought to exist in the superconducting state and are assumed not to affect the superconducting carriers.² Muons are sensitive to dipolar fields at a distance of up to a few lattice spacings, so if nanoscale magnetic domains exist, then the fraction of muons experiencing static local magnetic fields could be significantly higher than the fraction of Fe sites carrying an ordered moment. Such a type of coexistence was found to be realized within the $\text{SmFeAsO}_{1-x}\text{F}_x$ and $\text{CeFeAsO}_{1-x}\text{F}_x$ families of Fe-based HTSs.^{3,9}

Since the muon is a local probe, the μSR signals from spatially different areas of the sample are not averaged but superimposed in the measured spectra. This feature allows to distinguish between the three above-mentioned scenarios. As discussed above, the ZF- μSR response of the magnetic areas of the sample is characterized by a fast relaxing signal visible at early times of the spectra, while a nonmagnetic volume shows slow relaxation, better visible at longer times, only. In Fig. 4 two ZF muon-time spectra taken at the same temperature ($T = 2.6$ K) and pressure ($p = 2.36$ GPa) with different magnetic histories are shown. The first muon-time spectrum was recorded after cooling the sample from $T \simeq 100$ to 2.6 K in zero magnetic field. By keeping the temperature constant, the second ZF spectrum was obtained after ramping the magnetic field up to $\mu_0 H \simeq 0.1$ T and then setting it back

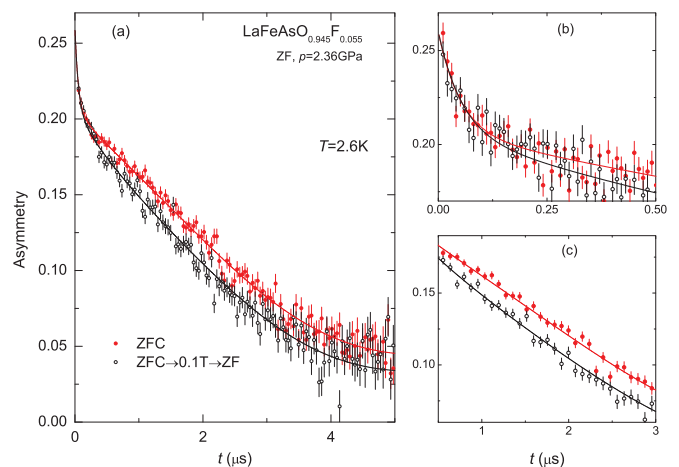


FIG. 4. (Color online) (a) The ZF- μSR time spectra obtained after cooling the sample in zero magnetic field from $T \simeq 100$ K down to 2.6 K (red/grays symbols) and after sweeping the magnetic field to 0.1 T and then setting it back to 0.0 without changing the temperature (black symbols). (b) and (c) show the extended parts of the muon-time spectra at the lower and the higher time, respectively. The solid lines are fits by means of Eq. (1).

TABLE I. Parameters as extracted from the fit of Eq. (1) to the muon-time spectra obtained after cooling the sample from $T \simeq 100$ to 2.6 K in zero magnetic field (ZFC) and after ramping the magnetic field up to $\mu_0 H \simeq 0.1$ T and setting it back to zero (ZFC \rightarrow 0.1 T \rightarrow ZF). A_S , A_{PC} , ζ , and ω were assumed to be the same for both spectra.

	$\Lambda_{m,t1}$ (μs^{-1})	$\Lambda_{m,t2}$ (μs^{-1})	Λ_{pm} (μs^{-1})
ZFC	19.7(2.7)	1.28(14)	0.165(26)
ZFC \rightarrow 0.1 T \rightarrow ZF	18.4(3.5)	1.20(28)	1.20(36)

to zero. Apparently, the ZF- μ SR response of the magnetic areas of the sample, as evidenced by the identically fast relaxations $\Lambda_{m,t1}$ and $\Lambda_{m,t2}$ at early times of the spectrum [see Fig. 4(b) and Table I], is not affected by the magnetic history. On the contrary, the ZF- μ SR signal representing the nonmagnetic volume of the sample exhibits a strongly larger relaxation Λ_{pm} after the application of an external field at low temperatures [see Fig. 4(c) and Table I]. This indicates that the superconductivity is most probably located within the nonmagnetic areas of the sample, since any changes of the magnetic field within a superconductor with nonzero pinning leads to trapping the magnetic flux and, as a consequence, to a very nonuniform field distribution inside the superconducting parts of the sample.²⁰

Our results point to a strong difference between $\text{LaFeAsO}_{1-x}\text{F}_x$ and the structurally related families of Fe-based HTSs with the La atom substituted by other rare-earth elements such as Sm, Ce, Pr, Nd, etc. In these families

bulk magnetism and bulk superconductivity are found to coexist on the nanoscale level.^{3,9} The magnetization and μ SR experiments reveal that in $\text{LaFeAsO}_{1-x}\text{F}_x$, the magnetism and superconductivity are not coexisting over the whole sample volume, i.e., this system is inclined toward phase separation. The reduction of the magnetic interaction and the simultaneous appearance of superconductivity indicate a much stronger competition between the two ordered parameters.

In conclusion, the interplay between magnetism and superconductivity was studied in $\text{LaFeAsO}_{0.945}\text{F}_{0.055}$ by performing muon-spin rotation and magnetization experiments as a function of pressure up to $p \simeq 2.4$ GPa. At ambient pressure the sample is purely magnetic, but at the border to the superconducting state of $\text{LaFeAsO}_{1-x}\text{F}_x$. The application of hydrostatic pressure leads to a substantial decrease of T_N and a reduction of the magnetic phase volume and, at the same time, to a strong increase of T_c and the diamagnetic susceptibility. Magnetic-history-dependent ZF- μ SR measurements show that superconductivity most probably develops in the areas of the sample that are nonmagnetic down to the lowest temperatures. This shows that in $\text{LaFeAsO}_{1-x}\text{F}_x$ magnetism and superconductivity are competing order parameters.

The work was performed at the S μ S, Paul Scherrer Institute (PSI, Switzerland) at the GPD instrument. The work of M.B. is supported by the Swiss National Foundation (SNF). G.P. and S.S. acknowledge the support of the NMI3 Access Programme. R.D.R. acknowledges MIUR PRIN 2008XWLWF9. The work of N.D.Z. and S.K. was partly supported by the NCCR program MaNEP.

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