# Neutron-irradiation effects in polycrystalline $LaFeAsO_{0.9}F_{0.1}$ superconductors

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(Received 20 July 2009; revised manuscript received 8 October 2009; published 11 November 2009)

The effect of atomic disordering induced by fast neutron irradiation on normal and superconducting state properties of polycrystalline $LaFeAsO_{0.9}F_{0.1}$ samples was investigated. Fast neutron ($E>0.1$ MeV) irradiation to a "moderate" fluence $\Phi=1.6\times10^{19}$ cm$^{-2}$ at $T_{irr}=50\pm10$ °C leads to a complete suppression of superconductivity which recovers almost completely after annealing at $T_{ann}\leq750$ °C. It is shown that decrease of $T_c$ under disordering is determined mainly by reduction in electronic relaxation time $\tau$. Such behavior is qualitatively described by the universal Abrikosov-Gor'kov equation, which testifies to an anomalous type of electron pairing in Fe-based superconductors.

DOI: 10.1103/PhysRevB.80.174512 PACS number(s): 74.62.Dh, 72.15.Gd

The discovery of high-temperature superconductivity in layered iron-based compounds[1] stimulated active experimental and theoretical studies of these systems. Systematic study of disordering effects in new superconductors is especially important in the case of possible anomalous types of Cooper pairing.[2] According to the Anderson theorem,[3] nonmagnetic impurities do not lead to suppression of superconductivity in the case of a conventional $s$-type isotropic pairing. If singlet pairing is mediated by spin excitations exchange, its requirement is symmetry with a sign-reversal order parameter.[4] Evidently, such requirement is fulfilled in high-$T_c$ cuprates, where pairing with $d$-wave symmetry is realized, while the pairing process proper is destroyed by intraband scattering induced by nonmagnetic impurities.[4–6] In Fe-based superconductors, the ordering parameter has an $s$-type symmetry, therefore generally accepted is the $s^{\pm}$-model, which supposes a superconducting state with opposite signs of the ordering parameter for electrons and holes.[4,7] In this case nonmagnetic impurities must lead to suppression of superconductivity due to interband scattering between the electron- and hole-type Fermi surfaces.[4,5,8] So the study of response of a superconducting system to atomic disordering allows the type of symmetry of the ordering parameter to be revealed.

Fast neutron irradiation is the most effective method of atomic disordering. This method was successfully applied earlier in investigating a number of superconducting compounds. We may refer to an example of qualitatively different behavior of superconducting properties of high-temperature superconductors $MgB_2$ and $YBa_2Cu_3O_7$ under neutron irradiation given in.[9] The $MgB_2$ demonstrated a relatively weak change of $T_c$ under irradiation, which is typical of the systems with strong electron-phonon interaction and isotropic $s$-type pairing. The $YBa_2Cu_3O_7$ showed rapid decrease of critical temperature $T_c$ with neutron fluence growth. Fast and complete degradation of superconductivity observed in $YBa_2Cu_3O_{7-\delta}$ testifies to a different, more exotic (nonfononic), pairing mechanism. This work presents the results of investigation of the effect of atomic disordering induced by fast neutron ($E>0.1$ MeV) irradiation to fluence $\Phi=1.6\times10^{19}$ cm$^{-2}$ at $T_{irr}=50\pm10$ °C and isochronal (30 min) annealing in the range of $T_{ann}=100-750$ °C on the normal and superconducting states of a sample of $LaFeAsO_{0.9}F_{0.1}$ nominal composition. The first results of the effect of irradiation with fast neutrons and heavy ions on the properties of oxypnictides are also published in our paper[10] and Refs. 11–13.

Polycrystalline samples of $LaO_{0.9}F_{0.1}FeAs$ consisting of $1-100$ $\mu$m grains were prepared with the use of a two-stage solid-state reaction followed by annealing under vacuum.[14] The data of $T_c$ and resistivity $\rho(T)$ are in a good agreement with the data of Ref. 15 for the samples with a similar composition. Resistivity $\rho$ and Hall coefficient $R_H$ were measured on a sample $2.0\times1.5\times0.4$ mm in size using the standard four-point method, with the reverse directions of dc current and magnetic field and switching over between the current and potential leads.[16] The electric contacts were made by ultrasonic soldering with indium. Measurements were performed in the temperature range $T=1.5-380$ K in up to 13.6 T magnetic fields.

Irradiation to "moderate" fast neutron fluence $\Phi=1.6\times10^{19}$ cm$^{-2}$ suppresses superconductivity and results in significant changes in the temperature dependences of resistivity $\rho(T)$ and Hall coefficient $R_H(T)$. Consecutive annealing in the range of $T_{ann}=100-750$ °C leads to a practically complete restoration of the sample properties in both the normal and the superconducting states.

The dependence $\rho$(T) measured at $H=13.6$ T under irradiation and subsequent annealing at different temperatures is shown in Fig. 1(a). Disordering results in a smaller temperature slope in the high temperatures range and an appearance of sections with a negative slope in the low temperatures range, with a logarithmic-type dependence typical of Kondo-type magnetic scattering.

The Hall-coefficient temperature dependences $R_H(T)$ in $LaO_{0.9}F_{0.1}FeAs$ under magnetic field $H=13.6$ T are shown in Fig. 1(b). $R_H(T)$ is negative in the normal state, which means that conduction is dominated by the contribution of electronlike charge carriers. $R_H(T)$ of the initial sample decreases as the temperature goes down to the superconducting transition temperature $T_c$, reaching a value of about $10^{-2}$ cm$^3$/C, which is in agreement with similar measurements in $LaO_{0.9}F_{0.1}FeAs$.[17] Irradiation causes an approximately two-times decrease in the value of $R_{H40}$, as measured at $T=40$ K. Annealing in the range of $T_{ann}=500-550$ °C restores the temperature dependence $R_H(T)$. However, the

 

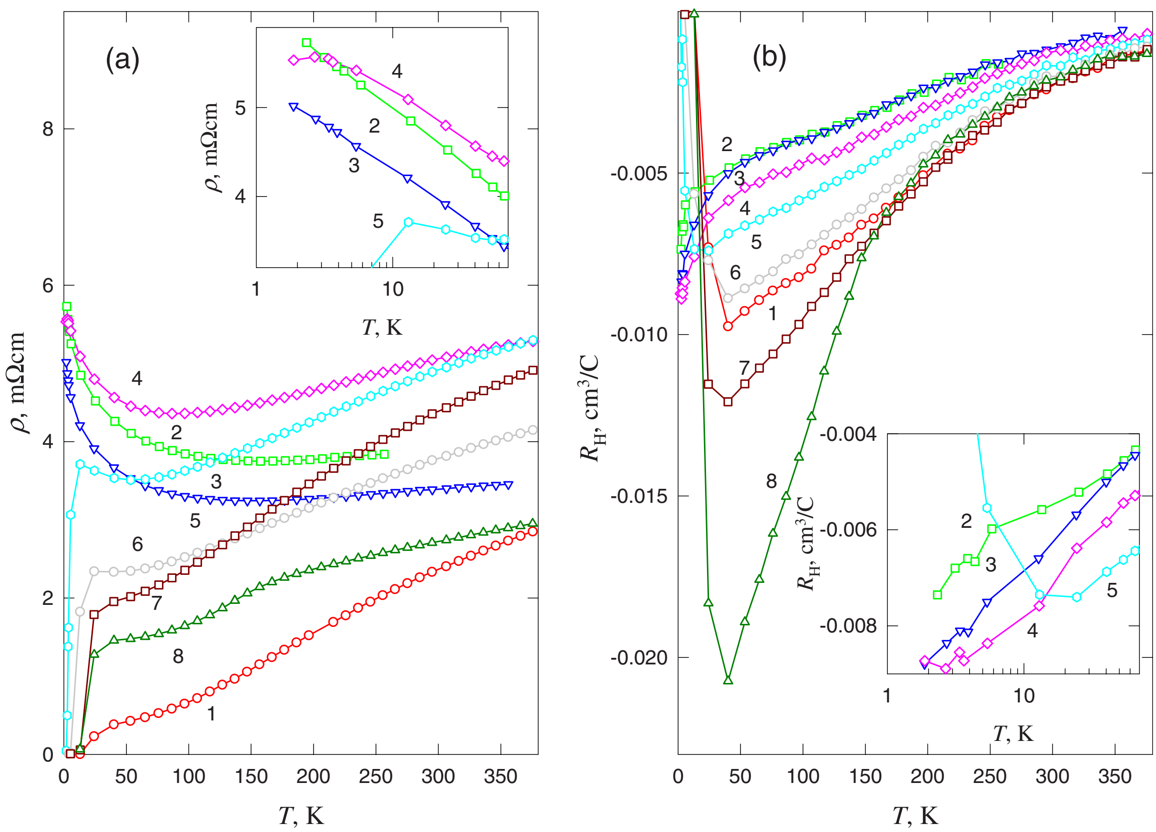


FIG. 1. (Color online) Temperature dependences of (a) resistivity $\rho$ and (b) Hall coefficient $R_H$ in magnetic field $H=13.6$ T of $LaO_{0.9}F_{0.1}FeAs$ sample: initial (1), irradiated to neutron fluence $\Phi=1.6\times10^{19}$ $cm^{-2}$ (2), and annealed at 200 (3), 300 (4), 400 (5), 500 (6), 600 (7), and 750 °C (8). Insets show $\rho(T)$ and $R_H(T)$ on a semilogarithmic scale.

value of $T_c$ still remains much below the initial value of $T_{c0}$. Subsequent annealing at $T_{ann}=600-750$ °C leads to a further two-times increase in $R_{H40}$ and a practically complete restoration of the initial $T_c$ value. It means that $R_H$ is not that sole parameter that governs the $T_c$ value. In the irradiated state of the sample (with superconductivity suppressed), at $T<30$ K, an additional contribution to $R_H(T)$ is present, which shows an approximately logarithmic dependence [Fig. 1(b)]. This behavior correlates with the upturn of $\rho$ in this temperature range.

Figure 2 shows the curves of resistivity transitions of the initial, irradiated and annealed states in magnetic fields 0, 1, 2, 4, 6, 8, 10, 12, and 13.6 T. Transition broadening with the magnetic field in a sample in the initial state is in agreement with similar measurements of samples of the same $LaO_{0.9}F_{0.1}FeAs$ composition made in Ref. 15. The irradiated and annealed (at $T_{ann}=400$ °C) sample features a narrower (in absolute value) superconducting transition, which is in agreement with the usual assumption of a spatially uniform (here, on a coherence length scale) distribution of radiation defects. Further annealing leads to gradual displacement of the curves toward the initial state; however, after annealing at $T_{ann}=750$ °C, the value of $T_c$ is still ~2 K below $T_{c0}$.

In order to define the upper critical-field slope $-dH_{c2}/dT$ from the resistivity curves of transition to a superconducting state, a criterion 0.9 or (rarely) 0.5 of the value of resistivity $\rho_n$ in the normal state is usually applied; at that, magnetoresistance noticeably affecting the accuracy of respective values definition should also be taken into account.

Shown in Fig. 3 are dependences $H_{c2}(T)$ defined by these two criteria. It is worth notice that both of the above dependences are quite far from being straight lines in practically all regions of magnetic fields, particularly for the sample in the states with high values of $T_c$. The most natural explanation of such behavior is that the initial (nonirradiated) sample is strongly inhomogeneous in composition, probably in fluorine and oxygen content, and the fact that the shape of the resistive transition curve in polycrystalline layered materials is determined primarily by crystallites with the *ab* planes oriented nearly perpendicular to the direction of the magnetic field.

In a first approximation, the principal changes of $-dH_{c2}/dT$ in samples with different degrees of imperfection are nevertheless not too great. The upper critical-field slope $-dH_{c2}/dT$, as defined by criterion 0.9 with respect to the value of resistivity $\rho_n$ in the normal state, is equal to approximately 3 T/K for the initial sample and increases approximately two times under irradiation.

At low temperatures, a small ($\leq 2\%$) negative contribution to magnetoresistance $\Delta\rho/\rho$ is observed, being present even in the superconducting samples at $T\geq T_c$ (Fig. 4). Such behavior of magnetoresistance clearly points to significant magnetic scattering of the Kondo type, which usually makes a contribution to both resistivity and Hall coefficient (skew Hall effect), which contributes to Hall resistivity $\rho_{xy}$ as a sum[18] $\rho_{xy}=c_H\rho M+R_H H$. In expectation of $M=$const for met-

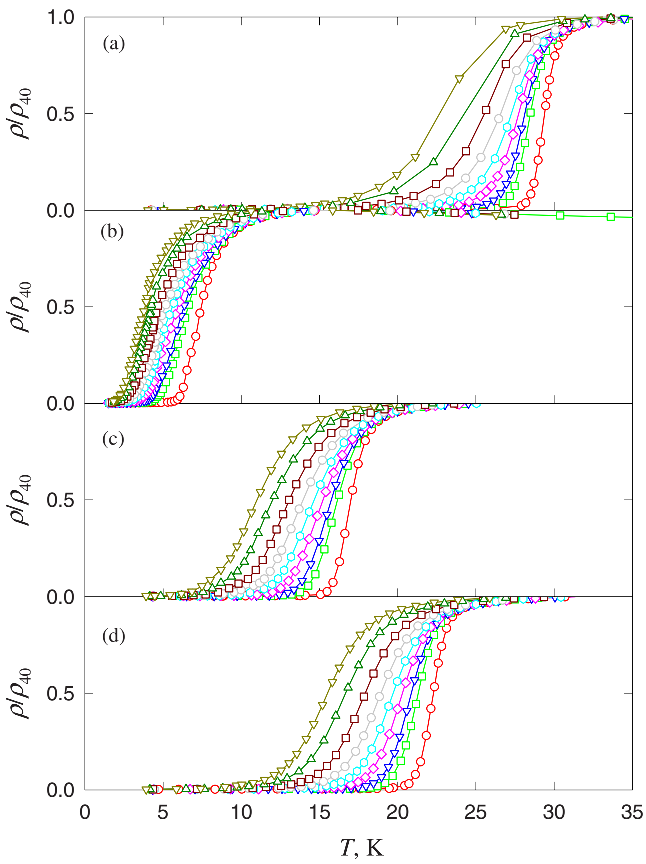


FIG. 2. (Color online). Temperature dependences of reduced resistivity $\rho/\rho_{40}$ in magnetic field $H$=0, 1, 2, 4, 6, 8, 10, 12, and 13.6 T of $LaO_{0.9}F_{0.1}FeAs$ sample: initial (a), irradiated to neutron fluence $\Phi = 1.6 \times 10^{19}$ cm$^{-2}$ and annealed at 400 (b), 500 (c), and 600 °C (d).

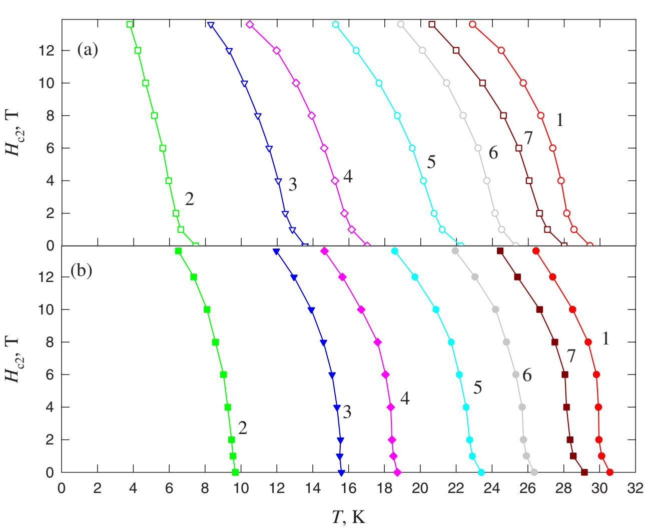


FIG. 3. (Color online) Temperature dependences of upper critical field $H_{c2}$, defined at 0.5 (a) and 0.9 (b) of $\rho_n$ in normal state (magnetoresistance was taken into account) for initial (1), irradiated and annealed at 400 (2), 450 (3), 500 (4), 550 (5), 600 (6), and 750 °C (7) $LaO_{0.9}F_{0.1}FeAs$ sample.

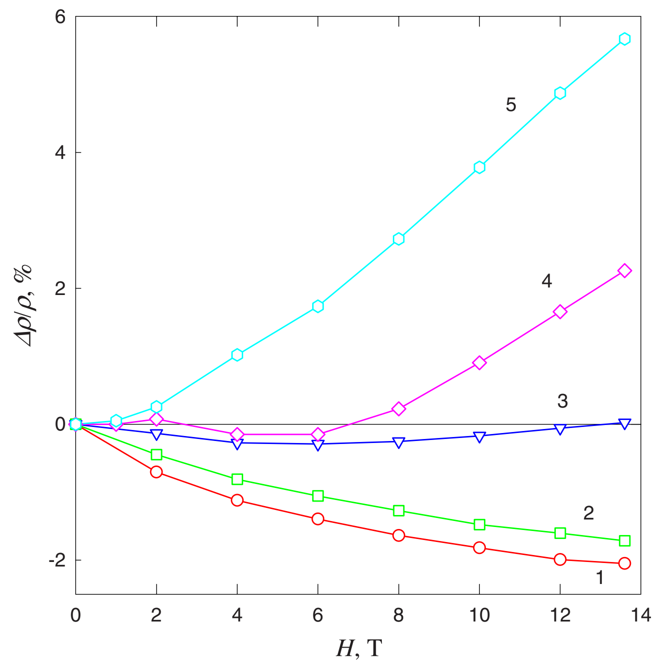


FIG. 4. (Color online) Reduced magnetoresistance $\Delta\rho/\rho$ vs $H$ at $T > T_c$ (superconducting sample) or $T$=4.2 K (nonsuperconducting sample) of $LaO_{0.9}F_{0.1}FeAs$ sample: irradiated to neutron fluence $\Phi = 1.6 \times 10^{19}$ cm$^{-2}$ (1) and annealed at 200 (2), 300 (3), 600 (4), and 750 °C (5).

als, we obtain an approximately equal (logarithmic) contribution at low temperatures, which is present both in $R_H(T)$ and $\rho(T)$.

In order to understand the strong temperature dependences $R_H(T)$ and a quite complex behavior of $\rho(T)$ as a function of temperature and disorder level (see Fig. 1), let us calculate Hall concentration $n_H = 1/(R_H e)$. According to band calculations and different experimental data,[2,19] the Fermi surface in LaOFeAs consists of several hole and electron bands on a characteristic Fermi energy scale of about 0.1–0.2 eV, therefore $R_H(T)$ must have a weak dependence on temperature. But Hall concentration $n_H = 1/(R_H e)$ in the temperature range of T ≥ 40 K is described by an exponential dependence of the kind $n_H(T) = n_0 + n_1 \exp(-E_g/T)$ with close values of $n_1$ and $E_g \approx 800$ K, while the main changes in $R_H(T)$ are connected with the changes in $n_0$ only. This is shown in the inset in Fig. 5(a), where background $n_0$ has been subtracted. Such behavior may be interpreted as excitation of charge carriers to a partially populated electron band from another band (or bands) separated by a gap of the order of $E_g$. Then, assuming that $n_H(T)$ is a real electron concentration, some useful parameters may be calculated. The mean-free path for a cylindrical Fermi surface[2]

$$l^* = (2\pi d)^{1/2}\hbar \, \{R_H\}^{1/2}/(\rho e^{3/2}), \quad (1)$$

where $d$ is interplane spacing, $d$=8.7 Å. Besides, it should be borne in mind that we are dealing with a ceramic sample of noticeable porosity (normally 15–20 %), and besides, due to an expected significant (of the order of 10 and over) anisotropy of resistivity, only an average of 1/3 of the sample makes the principal contribution to conductivity. According

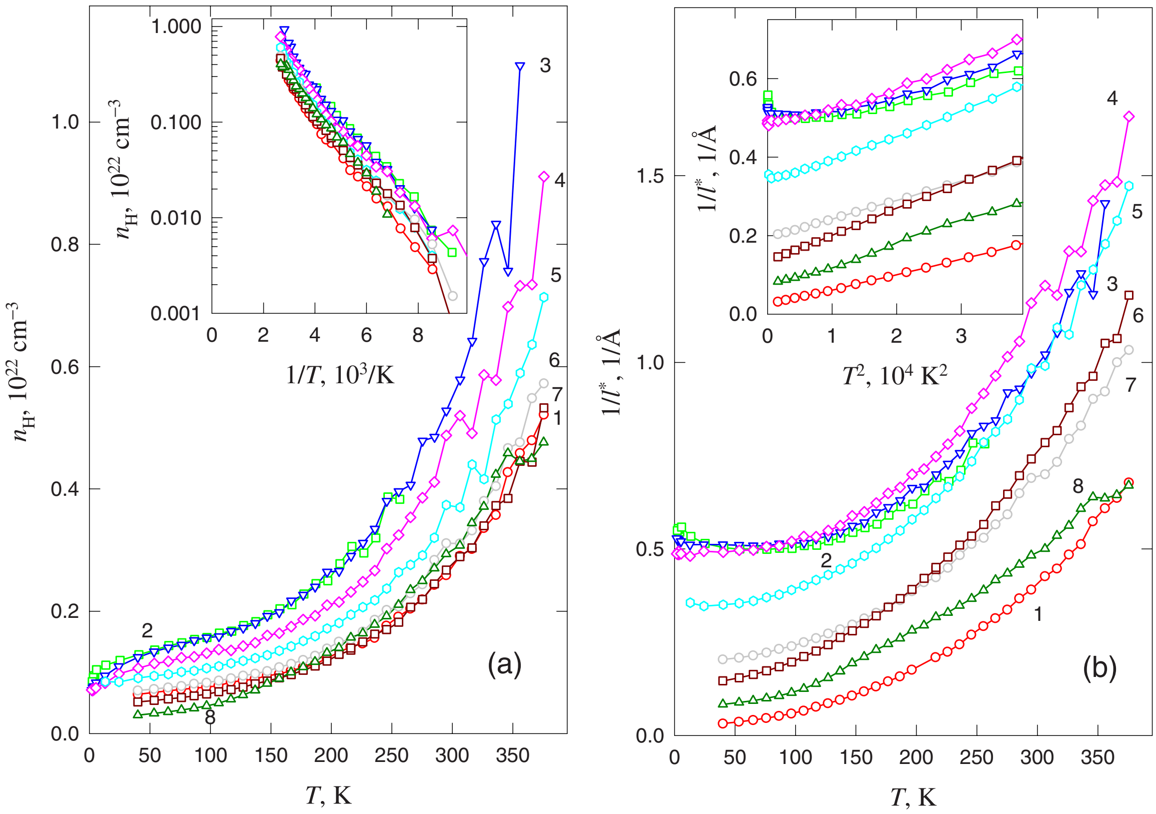


FIG. 5. (Color online) Temperature dependence of (a) Hall concentration $n_H=1/(R_H e)$ and (b) inverse mean-free path $1/l^*$ defined in text [Eq. (1)]. Insets show $n_H$ vs $1/T$ in semilogarithmic scale (background is subtracted, see text) and $1/l^*$ vs $T^2$. Curve notations are the same as in Fig. 1.

to the percolation theory,[20] for ~50% of the well conducting phase, resistivity increases by a factor of $k^*\approx 5$, thus true path $l$ is related to the value of $l^*$ found from Eq. (1) as

$$l \cong l^* k^*. \tag{2}$$

The temperature dependences of inverse path $1/l$ found from Eq. (1) vary monotonously at irradiation and annealing. At $T\leq 200$ K, these dependences are well described by a quadratic dependence characteristic of the prevailing electron-electron scattering [Fig. 5(b)] $1/l^*=a_0+a_2T^2$ with approximately equal values of $a_2$. Disordering decreases $l=l^*k^*$ from ~300 to ~12 Å at low temperatures, and from ~15 to ~5 Å at high temperatures, which looks quite reasonable. In the context of our interpretation, the complex behavior of $\rho(T)$ at annealing, see Fig. 1(a), is due to the diverse effects of two parameters: the decrease in the value of $1/l^*(T)$, see Fig. 5(b), leading to drop of $\rho(T)$, and the decrease in $n_H(T)$, see Fig. 5(a), leading to growth of $\rho(T)$.

Besides, knowing coefficient $a_2$, we may calculate effective electron mass $m^*$,[9,21]

$$(m^*)^2 = a_2\hbar^4/\{2\pi V_{cell}(k_B)^2\},$$

where unit-cell volume $V_{cell}\approx 140$ Å$^3$. Estimates also yield quite "reasonable" values of $(m^*/m_e)\approx 3$ for samples with different degree of disordering. Note that a similar "one-band" intepretation of the Hall coefficient was used in Ref. 22 in describing an experiment in the $Ba(Fe_{1-x}Co_x)_2As_2$ system, $x=0.04-020$.

And finally, the behavior of principal experimental parameters as a function of annealing temperature $T_{ann}$ is summarized in Fig. 6. At $300<T_{ann}<600$ K, principal recovery of the values of $T_c$ and $1/l^*$ takes place, and at $T_{ann}>600$ K, these parameters vary relatively little. Such nonmonotone behavior usually reflects the complex nature of defects emerging under irradiation and getting successively recombined in the course of annealing. On the other hand, $R_H$ displays quite monotone increase with the increase of $T_{ann}$ and does not display clear correlation with the value of $T_c$ (particularly in the region of $T_{ann}>600$ K), which is probably due to more essential processes of loss of a volatile component (fluorine, oxygen).

For comparison with the theoretical models, we made use of the universal Abrikosov-Gor'kov (AG) equation describing superconductivity suppression by magnetic impurities (defects) for the case of $s$ pairing and nonmagnetic impurities for the case of $d$ pairing,[23]

$$\ln(T_{c0}/T_c) = \psi(\alpha + 1/2) - \psi(1/2), \tag{3}$$

where $\alpha=\mathrm{h}/(4\pi^2 k_B T_c \tau)$, $\psi$ is digamma function, $T_{c0}$ and $T_c$ are superconducting temperatures of initial and disordered systems, respectively, $\tau$ is electronic relaxation time, which may be constructed from the experimental values,

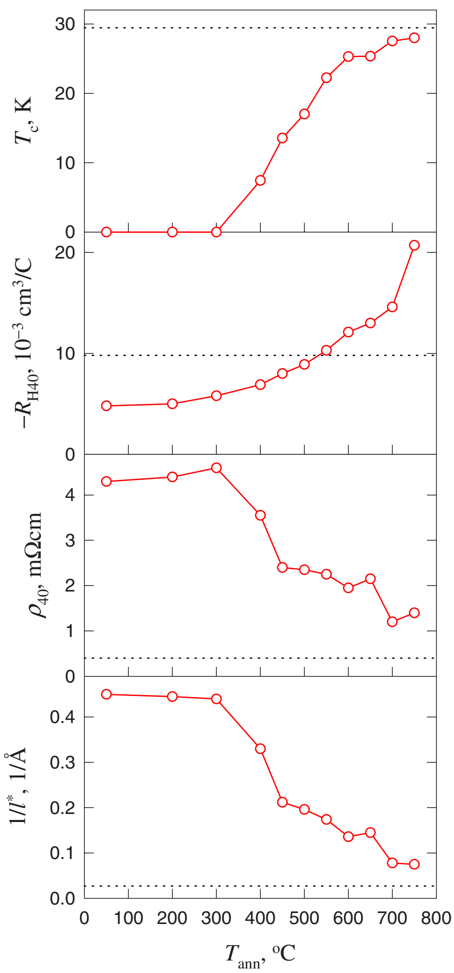


FIG. 6. (Color online) Superconducting temperature $T_c$, as defined by criterion 0.5 with respect to the value of resistivity $\rho_n$ in the normal state in magnetic field $H=0$ T, measured at $T=40$ K Hall coefficient $R_{H40}$ and resistivity $\rho_{40}$, extrapolated to $T=0$ inverse electronic mean-free path $1/l^*$ as a function of annealing temperature $T_{ann}$ for $LaO_{0.9}F_{0.1}FeAs$ sample irradiated to neutron fluence $\Phi=1.6\times10^{19}$ cm$^{-2}$. Shown with dotted lines are value levels for nonirradiated state.

$$\tau=(m^*R_H)/(e\rho). \quad (4)$$

Like in Eq. (2), percolation coefficient $k^*$ should be also taken into account. Relation (3) describes the decrease of $T_c$ as a function of inverse relaxation time $\tau^{-1}$, superconductivity is suppressed at $\alpha>\alpha_c=0.88$.

Note that the one-band model used here for determining relaxation time $\tau$, in spite of its obvious contradiction with band calculations and some experimental data (pointing to the presence of holes and electrons in comparable concentrations), gives in some cases[22,24] a good agreement of low-temperature Hall concentration of electrons $n_H$ with the doping level. As noted in Ref. 24, such agreement is most likely connected with the holes mobility being much below the electrons mobility, so it is mainly the electrons that contribute to the Hall effect. In such a case we may expect that the relaxation time, as determined by formula (4) with the use of the low-temperature values of $R_H$ and $\rho$, will be close to its real value.

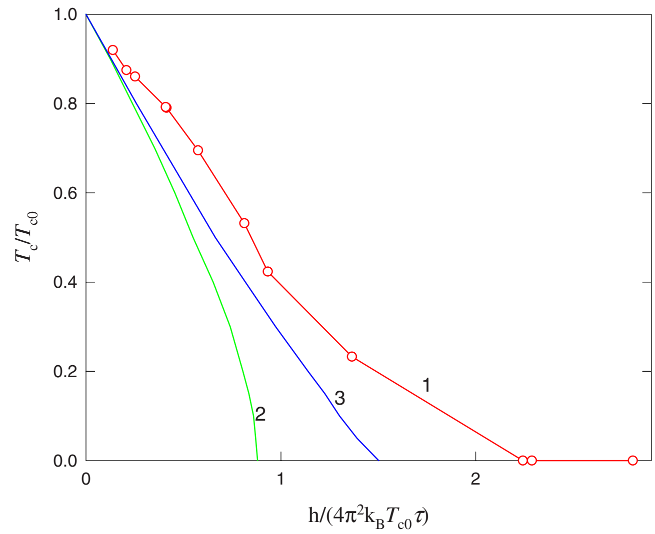


FIG. 7. (Color online) $T_c/T_{c0}$ as a function of $h/(4\pi^2k_BT_{c0}\tau)$, $T_{c0}=32$ K of initial, irradiated and annealed $LaO_{0.9}F_{0.1}FeAs$ sample. 1: experiment; 2: theoretical prediction [Eq. (3)]. Line 3 shows $T_c$ suppression of $YBa_2Cu_3O_7$ (Ref. 25).

Experimental dependences are compared in Fig. 7 with an AG model in nondimensional coordinates of $T_c/T_{c0}$ as a function of $\alpha=h/(4\pi^2k_BT_{c0}\tau)$. There is a good qualitative agreement, particularly if we remember that $\alpha$ does not contain any fitting parameters. We may hardly expect detailed agreement bearing in mind a number of assumptions made and other uncertainties, especially with regard to the value of percolation coefficient $k^*$. Note that a very similar behavior of superconductivity was observed for HTSC systems, such as $YBa_2Cu_3O_7$ (Refs. 9 and 25) (see Fig. 7). Thus the experimental data for both Cu- and Fe-based superconductors are in good agreement with the AG theory, which implies the presence of an exotic mechanism of electron pairing in these systems, differing from the isotropic $s$-wave symmetry.

It is usually considered that radiation defects are equivalent to nonmagnetic impurities in their influence on superconductivity. Nevertheless, as noted in Ref. 4, defects of any kind in iron oxypnictides may, in principle, induce a static magnetic moment in neighbor Fe sites, leading to magnetic scattering. The observed low-temperature logarithmic contributions to $R_H(T)$ and $\rho(T)$ and the negative magnetoresistance $\Delta\rho/\rho$ clearly point to such scattering. The authors of Ref. 26 studied theoretically, in the $s^{\pm}$ pairing symmetry approximation, the influence of magnetic impurities on superconductivity which is suppressed due to intraband scattering. In such approximation, they were able to quantitatively describe the effect of neutron irradiation on $T_c$ in $LaO_{0.9}F_{0.1}FeAs$.[10] Since, within the scope of the $s^{\pm}$ model, nonmagnetic impurities in the case of interband scattering are identical to magnetic impurities in the case of intraband scattering,[8,27] and in the absence of any magnetic data on the irradiated $LaO_{0.9}F_{0.1}FeAs$ samples, it would be too early to jump to a conclusion with regard to which kind of disordering (magnetic or nonmagnetic) leads to suppression of superconductivity in this case.

In conclusion, we investigated the effect of irradiation with fast neutrons and subsequent isochronal annealing on the properties of normal [resistivity $\rho(T)$, Hall coefficient $R_H(T)$] and superconducting (transition temperature $T_c$, upper critical field $H_{c2}$) states of the $LaO_{0.9}F_{0.1}FeAs$ polycrystalline sample. The observed suppression of superconductivity is accompanied by effective system doping (electrons concentration growth). Assuming that radiation defects lead predominantly to nonmagnetic interband scattering (defects are equivalent to nonmagnetic impurities), fast suppression of superconductivity under irradiation most probably points to an anomalous type of Cooper pairing in this system.

The authors would like to thank C. Hess, R. Klingeler, B. Buechner, A. N. Vasiliev, and O. S. Volkova for fruitful discussions and providing us with high quality samples. This work was carried out with partial support of the Program of Basic Research of the Presidium of RAS "Condensed Matter Quantum Physics" (Project No. 4 UB RAS) and RFBR Project No. 07-02-00020-a.

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