Physica C 476 (2012) 68–72

Contents lists available at SciVerse ScienceDirect

Physica C

journal homepage: www.elsevier.com/locate/physc

Irreversibility line of $Ba_{1-x}K_xFe_2As_2$ (T_c = 36.9 K) superconductor studied with ac-susceptibility measurements

M. Pissas [a,*], M. Zeibekis [a], D. Stamopoulos [a], Chunlei Wang [b], Yanwei Ma [b]

[a] Institute of Materials Science, NCSR, Demokritos, 15310 Aghia Paraskevi, Athens, Greece
[b] Key Laboratory of Applied Superconductivity, Institute of Electrical Engineering, Chinese Academy of Sciences, PO Box 2703, Beijing 100190, China

ARTICLE INFO

Article history:
Received 26 October 2011
Received in revised form 8 February 2012
Accepted 16 February 2012
Available online 24 February 2012

Keywords:
Irreversibility line
Vortex matter properties
Ac-susceptibility measurements
Pnictides

ABSTRACT

We have studied the phase diagram of a $Ba_{1-x}K_xFe_2As_2$ (T_c = 36.9 K) single crystal superconductor by employing ac-susceptibility measurements, both as a function of temperature for constant external magnetic field and of magnetic dc-field for constant temperature for angles $\Theta = \angle(\mathbf{H}, c\text{-axis}) = 0°, 30°, 45°, 60°, 75°$ and 90°, between the c-axis and the magnetic field. The irreversibility lines ($H_{irr}(T, \Theta)$) are estimated from the onset of non-zero values of the amplitude of the third harmonic susceptibility. H_{irr}-lines for all the studied angles can be reproduced from the equation $H_{irr} = H_0(\Theta)(1 - T/T_c)^n$, with $n \approx 4/3$. From the angular dependence of $H_0(\Theta)$ parameter we estimated the anisotropy of the irreversibility lines. In the temperature interval [35, T_c] the anisotropy parameter was estimated $\gamma = H_{irr}^{ab}/H_{irr}^{c} = 2.2 \pm 0.1$. The measurements of the real part of the fundamental (first harmonic) ac-susceptibility $\chi'(T)$ for constant temperature as a function of dc-magnetic field revealed, for high values of the ac-field amplitude, a second peak in the critical current. The second peak line is located far from H_{irr}-line and exists up to T_c. Although the particular sample has high critical densities, contrary to the predictions of the Bean's model, the maximum of the imaginary part of the fundamental ac-susceptibility χ'' exhibits lower values. This behavior could be explained by assuming a reversible motion of the flux lines around the pinning centers for low values of the ac-magnetic field amplitude.

1. Introduction

The Abrikosov state in type II superconductors is a natural "scene" where one can observe interesting properties associated with the superconductivity and the condensed matter physics. The high critical temperature (T_c), the significant anisotropy and the small coherence length in cuprate superconductors have substantially changed the Abrikosov state in comparison to the low T_c superconductors [1,2]. In these materials the Abrikosov state is divided by a melting transition to solid and liquid states of flux lines (vortices). Depending upon the amount, strength and kind of pinning centers the solid Abrikosov state displays many interesting glass phases with novel physics.

The discovery of new superconductors adds complexity, challenging the existing knowledge. Worth noticing is the case of MgB_2 [3] where the contributions from two bands in the pairing mechanism induce novel flux lines properties, such as that of temperature dependance of the second critical field anisotropy. Twenty two years after the discovery of high-T_c in cuprates [4], the unexpected discovery of Kamihara et al. [5] that the quaternary rare-earth (RE) transition metal pnictides (oxypnictides) with the general formula $LaFeAsO_{1-x}F_x$ is a superconductor with significant critical temperature brought superconductivity again in the research front of condensed matter physics. This discovery marks a new period for superconductivity since this material is cooper free and the element which the electronic states contribute to the conductivity band is iron, a magnetic element with unpaired d-electrons. Soon after this seminal discovery, a new class of oxygen free iron pnictides, with chemical formula (Ba, Sr, Ca)$_{1-x}$(K, Na)$_x$-Fe_2As_2 were discovered [6–11] with a maximum $T_c \approx 38.5$ K for the compound $Ba_{1-x}K_xFe_2As_2$. The crystal structure of this compound is very similar to that of oxypnictides, consisting of a two dimensional square lattice of tetrahedrally coordinated iron atoms with the arsenic (with covalent bonding) separated by a mixed (Ba, Ca, Sr) or (K, Na) layer [12,13].

Since pnictides and oxypnictides are new families of superconductors, research on their flux line properties, is a very interesting topic of basic and applied physics. Also of interest is the comparison of their vortex matter properties with cuprates, MgB_2 and low T_c superconductors. As examples we can mention the estimation of basic superconducting parameters, the role of the thermal fluctuations, the order–disorder transition and the type of defects acting as pinning centers [1,2].

Several studies concerning the magnetic properties of oxypnictides and $Ba_{1-x}K_xFe_2As_2$ have shown that they are type II

* Corresponding author.
E-mail address: mpissas@ims.demokritos.gr (M. Pissas).

0921-4534/$ - see front matter © 2012 Elsevier B.V. All rights reserved.
doi:10.1016/j.physc.2012.02.018

superconductors with moderate anisotropy, and quite large second critical fields, both along *c*-axis and *ab*-plane, implying small coherence lengths, comparable with the size of atomic defects) [14–25]. This fact has as a consequence that chemical inhomogeneities (e.g. local fluctuations of K concentration) and structural defects could act as strong pinning centers, giving rise to high critical current densities.

The present article reports on ac-susceptibility measurements of $Ba_{1-x}K_xFe_2As_2$ superconductor, for $\mathbf{H}_{dc}\|\mathbf{H}_{ac}\|c$-axis ($\Theta = 0$), as function of temperature for constant dc-magnetic field and as a function of the dc-magnetic field for constant temperature. The measurements were also repeated for $\Theta = 30°, 45°, 60°, 75°$ and $90°$. In every case the locus of $H_{irr}(T, \theta)(T)$ line is estimated from the onset of the third harmonic in measurements of the real χ'_3 and imaginary χ''_3 parts of the ac-susceptibility.

2. Experimental details

$Ba_{1-x}K_xFe_2As_2$ single crystals were grown with a single step method by heating stoichiometric amount of Ba and Fe powders and excess of As and K pieces as is described elsewhere [26]. For ac susceptibility measurements the 9 T Physical Property Measurement System (Quantum Designed) sample environment platform, equipped with ac-susceptibility measuring option was used. Measurements in different angles between the *c*-axis of the crystal and the axis of both ac and dc magnetic fields were made by attaching the crystal *ab*-face in appropriately cut cylindrical plexiglass rod. The total magnetic field is given by $\mathbf{H}(t) = \mathbf{H}_{dc} + \mathbf{H}_0\sin(2\pi ft)$, where $\mathbf{H}_{dc}$ is the dc-field, $\mathbf{H}_0$ and f the amplitude and the frequency of the ac-field, respectively. In all measurements the dc and ac magnetic fields were parallel ($\mathbf{H}_{dc}\|\mathbf{H}_0$). The external magnetic field induced screen current that can be considered as a time dependant magnetization $\mathbf{M}(t) = (1/2c)\int \mathbf{r}\times\mathbf{J}dx^3$. The harmonic real and imaginary parts of ac-susceptibility are defined by the fourier series [27,28]

$$M(t) = \sum_{n=1}^{\infty}\left[\chi'_n H_0\sin(2\pi nft) + \chi''_n H_0\cos(2\pi nft)\right]$$

where $\chi'_n = (2f/H_0)\int_0^{1/f} M(t)\sin(2\pi nft)dt$ and $\chi'_n = (2f/H_0)\int_0^{1/f} M(t)\cos(2\pi nft)dt$ are the real and the imaginary parts of ac-susceptibility. Within Bean critical state model the magnetic field, inside a long cylinder with radius R, is characterized by the magnetic field for full penetration $H^* = 4\pi R\,J_c(T, H_{dc})/c$, where $J_c(T, H_{dc})$ is the critical current density. In this model only the odd components of ac-susceptibility are non-zero. The imaginary part takes the maximum value as a function of temperature or dc-field when $H^*(T, H_{dc}) = H_0$. In the ideal Bean state the ac-susceptibility is frequency independent and depends only on H_0.

For our measurements we selected a small $Ba_{1-x}K_xFe_2As_2$ single crystal with parallelepiped shape and dimensions $\approx 1500\times 600\times 50\ \mu m^3$. The critical temperature of the particular crystal is estimated $T_c = 36.9 \pm 0.1K$ ($\Delta T_c = 0.5$ K) from ac-susceptibility measurements, in zero dc magnetic field and $H_0 = 1$ Oe. Taking into account the variation of the critical temperature with the K content [7], the $T_c = 36.9$ K indicates a K concentration $0.35 \leqslant x \leqslant 0.45$.

3. Results and discussion

Fig. 1a shows the temperature dependance of real (χ') and imaginary (χ'') parts of the fundamental ac-susceptibility measured at various dc fields with ac-magnetic field amplitude $H_0 = 10$ Oe and frequency $f = 5111$ Hz. Both the dc and the ac magnetic fields have been applied parallel to the *c*-axis. The dominant characteristic of the $\chi'_1(T)$ curves is the shift of the diamagnetic onset towards lower temperatures (shown in Fig. 1b) with the increasing of the dc-magnetic field. This is the expected behavior

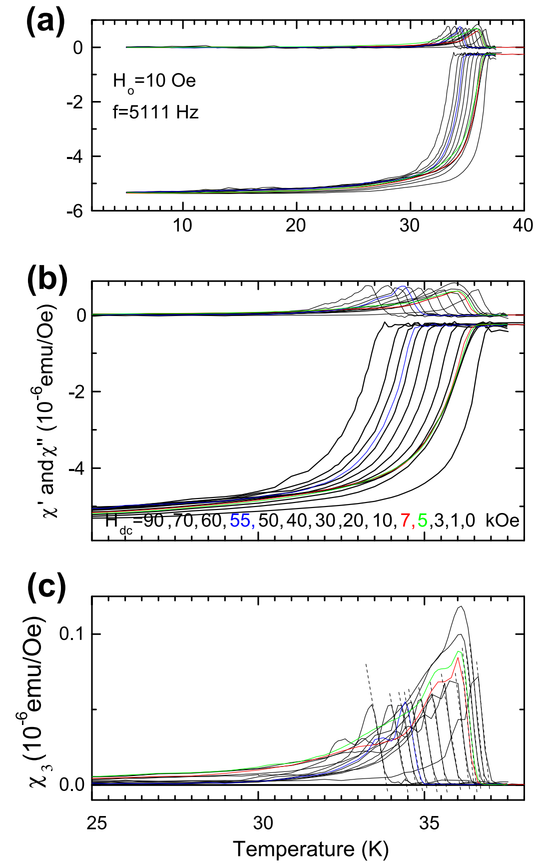

Fig. 1. (a) Temperature variation of χ' and χ'' for several dc magnetic, fields of $Ba_{1-x}K_xFe_2As_2$ superconductor measured for $H_0 = 10$ Oe and $f = 5111$ Hz. (b) A zoom of the measurements shown in panel (a) near the transition temperatures. (c) At the same temperature scale with panel (b) temperature variation of the amplitude of the third harmonic, measured for $H_0 = 10$ Oe and $f = 5111$ Hz. Both ac and dc-magnetic fields applied parallel to the *c*-axis ($\Theta = 0$).

for a type II superconductor when the irreversibility line is crossed. At low temperatures the diamagnetic current fully shields the ac-field and the ac response is linear. The $\chi''(T)$ curves display a positive maximum very close to the diamagnetic onset, representing the ac losses due to the irreversible entrance and exit of flux lines. The measurements for $H_{dc} = 7$ kOe seem to violate the parallel shifting near the diamagnetic onset, as the dc-field increases. This anomalous behavior is related with the occurrence of a second magnetization peak (see below).

The onset of non-linearity is defined from the onset of the third harmonic susceptibility. It is known that in the framework of Bean's critical state model ($E(J) = E_0(J/J_c)^{n\to\infty}$) the onset of $\chi_3 = \sqrt{(\chi'_3)^2 + (\chi''_3)^2}$ coincides with the condition $J_c \approx 0$, which could be considered as a condition defining the irreversibility line. If a narrow temperature interval separating irreversibility and second critical field lines (which represents a possible flux flow regime or a vortex liquid state) is ignored, then irreversibility line can be considered as a lower boundary of $H_{c2}(T)$ line. This assumption agrees with the specific heat and ac local Hall measurements of Kacmarcik et al. [17] in a (Ba, K) Fe_2As_2 single crystal. In some studies [14,22] the $H_{c2}(T)$ line is determined using the criterium $\rho(H, T) = 95\%\rho_n$, where $\rho(H, T)$ is the resistivity under a magnetic field and ρ_n is the normal state resistivity. The condition $\rho(H, T) = 0.05\%\rho_n$ is used to define the irreversibility line.

Fig. 1c shows the temperature dependance of the amplitude of the third harmonic susceptibility measured in several dc-magnetic