Vortex Structure and Anisotropic Superconducting Gaps in Ba[Fe(Ni)]$_2$As$_2$

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Abstract We studied nearly optimally Ni-substituted BaFe$_{2-x}$Ni$_x$As$_2$ (BFNA) single crystals with $T_C \approx 18.5$ K. In irreversible magnetization measurements, we determined the field dependence of the critical-current density and discuss the nature of observed strong bulk pinning. Using intrinsic multiple Andreev reflections effect (IMARE) spectroscopy, we directly determine two distinct superconducting gaps and resolve their moderate anisotropy in the momentum space. The BCS-ratio for the large gap $2\Delta_L/k_B T_C > 4.1$ evidences for a strong coupling in the $\Delta_L$-bands.

Keywords high-$T_C$ superconductivity · pnictides · vortex pinning · Andreev spectroscopy

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1 Introduction

The BaFe$_2$As$_2$ family (Ba-122) is currently the most intensively studied among the pnictide superconductors [1] due to rather high critical temperatures up to $T_C \approx 38$ K [2] and quite simple growth of large single crystals. Ba-122 possess a layered crystal structure with Fe–As blocks separated with Ba-based spacers along the crystallographic c-direction. Because of high critical fields and critical current density, the Ba-122 superconductors are very perspective for high-field applications. Significant efforts have been devoted to understanding their properties. Studying of vortex matter in Ba-122 compounds may provide an opportunity to understand the crossover between low temperature and high temperature superconductors. Among the 122 pnictide superconductors one of the most studied compounds is Co-doped BaFe$_2$As$_2$ [3] where high flux creep rates and a transition from collective to plastic creep have been reported. The pinning behaviour in Co-doped BaFe$_2$As$_2$ is rather complex, and different sources of pinning have been discussed: grain boundaries [4] and nanoscale variations of $T_C$ and/or the inhomogeneous distribution of dopant atoms [5,6]. In this paper, we study superconducting properties of the nearly optimum BaFe$_{2-x}$Ni$_x$As$_2$ single crystals. We found that strong bulk pinning in BFNA is similar to that in Co-doped Ba-122 and arises due to random point-like nanoparticles. In the high-field range, pinning is caused by combination of several mechanisms related to the point defects.

Several bands crossing the Fermi level produce quasi-two-dimensional Fermi surface sheets, the electron-like near the M point, and the hole-like near the $\Gamma$ point of the Brillouin zone [7]. Despite the intensive studies of the 122-family, the available experimental data are contradictive [8]. The very likely reason for this is a complex and unconventional nature of superconductivity, in particular, a theoretically supposed [9,10] anisotropy of superconducting properties in momentum space. In general, considering the importance of various intra- and interorbital interactions, theoretical studies offer two basic pairing models, the so called $s^{++}$ and $s^\pm$. In the $s^\pm$ model, superconductivity arises through
a strong interband coupling via spin fluctuations [9]. In contrast, recent $s^{++}$ calculations take into account two fundamental pairings, via spin and orbital fluctuations [10]. The competition of these mechanisms could result in anisotropic or even nodal order parameter [10]. Consequently, the gap symmetry and the pinning symmetry might be capable to reveal the underlying pairing mechanism.

Here we present detailed studies of superconducting properties of nearly optimal Ni-substituted $\text{BaFe}_{2-x}\text{Ni}_x\text{As}_2$ single crystals with $T_C \approx 18.5$ K. Using irreversible magnetization data, we discuss pinning effects in $\text{Ba}-122$. In Andreev spectroscopy studies, we directly determine the structure of superconducting order parameter.

2 Experimental details

BaFe$_{2-x}$Ni$_x$As$_2$ single crystals with $x=0.093$ and $x=0.1$ and critical temperature $T_C \approx 18.5$ K were grown using the self-flux method. High purity Ba, FeAs and NiAs were mixed in 1 : 5$(1-x) : 5x$ molar ratio, placed in alumina crucible and sealed in a quartz tube under 0.2 bar argon pressure. The ampoule was heated up to 1200$^\circ$C and then cooled down to 1000$^\circ$C at 2$^\circ$C/h rate. The grown crystals are up to $4 \times 2 \times 0.2$ mm$^3$ in size and possess a single superconducting phase. The irreversible magnetization $M(H,T)$ measurements were performed using the PPMS vibrating sample magnetometer in fields up to 9 T applied along the crystal $c$-axis. The typical field sweep rate was 100 Oe/s.

The superconducting order parameter was probed using multiple Andreev reflections effect (MARE) spectroscopy [11,13,14,15]. In ballistic superconductor-normal metal-superconductor (SnS) junction (the junction dimension $2a$ is less than the carrier mean free path $l$), MARE manifests itself as a pronounced excess conductance at low bias voltages (so called “foot”), and a subharmonic gap structure (SGS). In case of high-transparency SnS-junction, the SGS is a series of dynamic conductance dips at positions $V_n = 2\Delta/ev$, where $\Delta$ is the superconducting gap, $e$ — elementary charge, and $n = 1, 2, . . .$ — subharmonic order [16,17,18,19]. Therefore, the gap may be directly determined from the positions of Andreev subharmonics at $0 < T < T_C$ [19]. Obviously, two distinct gaps would cause two SGS in a dynamic conductance spectrum. An anisotropy of the superconducting gap strongly affects the shape of Andreev subharmonics. [20,21,22]. Isotropic ($s$-wave) gap produces high-intensive and symmetrical dips, while $d$-wave or fully anisotropic $s$-wave gap make subharmonics poorly visible and strongly asymmetric. Anisotropic gap with $\Delta(\theta) \sim \cos(4\theta)$ angle distribution in the $k$-space (which is very likely the case for $\text{Ba}-122$ [9]) for $c$-direction transport causes doublet-like features with two minima connected by an arch, which positions correspond to the higher and lower extremes of the gap angular distribution [22].

High-quality SnS-contacts were formed by a “break-junction” technique [22,23]. The single crystal plate was attached to a springy sample holder using pads of In-Ga paste, and then cooled down to $T = 4.2$ K. In the cryogenic environment, a gentle mechanical curving of the sample holder produced a cryogenic cleavage of the sample, thus creating two superconducting banks separated with a weak link (ScS-contact, $c$ — constriction). In $\text{Ba}-122$, the constriction usually acts as a thin normal metal, making it possible to observe MARE in a ballistic SnS-contact [10,13,15,24]. Since the superconducting banks are located at a tiny distance during the experiment, our technique preserve the crack from impurity penetration and provides clean cryogenic surfaces to probe the gap(s) magnitude almost unaffected by surface defects. When varying precisely the curvature of the sample holder, two cryogenic surfaces slide apart along the $ab$-plane, forming up to several tens of ScS-contacts with various dimension and resistance. This helps to collect a large amount of data with one and the same sample in order to check data reproducibility to be aware of dimensional effects.

Another unique feature of the “break-junction” is the formation of ScSc$\ldots$-S arrays [11,15,12] when probing a layered sample. The layered single crystal usually exfoliates along the $ab$-planes with the formation of steps and terraces along the $c$-direction, where an intrinsic multiple Andreev reflections effect (IMARE) occurs. Strictly speaking, IMARE is similar to intrinsic Josephson effect [25], firstly observed in cuprates [26]. When considering an Andreev array as a sequence of $m$ identical SnS-junctions, the position of SGS scales with $m$: $V_n = \frac{2\Delta}{en} \times m, \quad n = 1, 2, \ldots$ In order to determine $m$ and the gap(s) value, one should find such natural numbers which scale the I(V) and $dI(V)/dV$ curves for various arrays onto each other, or to achieve the same position of gap features with those for a single-junction spectrum. In such array, the contribution of bulk effects well exceed that of the surface influence [15,22]. Strictly speaking, the IMARE spectroscopy is currently the only technique probing the bulk values of superconducting gap(s) locally (within the contact area of 10–50 nm) [22].

The $dI(V)/dV$ curves were directly measured using a current source and a standard modulation technique [27]. Obviously, in our case the current flows along the $c$-direction, and the corresponding velocity components of the charge carriers are lying almost in-plane,
with the single vortex regime; (II) a power-law dependence \( J_c \sim H^p \), associated with strong pinning centers; (III) a regime related with the fish-tail; and (IV) a fast drop in \( J_c(H) \) where the vortex dynamics changes to plastic. As one can clearly see several marked area in Fig. 2 are related with different pinning regimes. Regime I is observed at low fields up to 150 Oe. The power law behaviour \( J_c \sim H^p \) extends up to 2 T. The obtained values of \( p \) were from 0.45 to 0.58 for temperature interval between 18 to 4 K respectively. The value of the exponent is in good agreement with the theoretical prediction of \( p \approx 5/8 \). Such value of the exponent \( p \) indicates strong vortex pinning \( [35] \). The III regime behaviour is related with crossover or/and correlation between strong pinning by defects and weak intrinsic pinning by large amount of centers — the so-called “caging” effect (CE). This leads to the conclusion that pinning in fields < 2 T originates from different mechanisms. The IV region is associated with disruption of magnetic vortices from pinning centers, which leads to the dissipation of energy and a rapid drop of the critical current. The onset of the IV regime is the limit of practical applicability of the superconductor. It should be noted that almost the same behavior of \( J_c \) is observed in optimally doped BFNA.

4 Andreev spectroscopy

Figure 3 shows current-voltage characteristic (CVC) and its derivative for a ScS-array formed in BFNA single crystal. The bias voltage axis was scaled with the natural \( m \) to a single-junction spectrum, therefore, all the data corresponds to a single junction. The pronounced foot in CVC is typical for a clean SnS-mode \([16,17,18,19]\). To check whether the contact is ballis-
tic, we take the normal-state bulk resistivity $\rho(22\text{K}) \approx 1.9 \times 10^4 \Omega \text{cm}$ for the sample under study, and the contact resistance $R \approx 200 \Omega$ (see Fig. 3) and plug them into the Sharvin formula $a = \sqrt{\frac{4}{\pi} \frac{\Delta^2}{l}}$. Given the product of the bulk resistivity and the carrier mean free path for Ba-122 determined elsewhere $[37] \rho^a l \approx 1.7 \times 10^{-9} \Omega \text{cm}$, we immediately get $2a \approx 36 \text{nm} < l \approx 90 \text{nm}$, thus proving the contact is ballistic and favourable for MARE observation. Note the both parts of CVC (the positive and negative current) in Fig. 3 are symmetrical and non-hysteretic. In the $dI(V)/dV$ spectrum (red line), two Andreev subharmonics for the large gap (observable in bias voltages $V_1 = 2\Delta_L/e \approx 9.2 \text{mV}$, and $V_2 = \Delta_L/e \approx 4.6 \text{mV}$) are well pronounced. The position of these dips determine the large gap $\Delta_L \approx 4.6 \text{meV}$. Taking into account a strongly asymmetrical shape of both dips, one may suppose a gap anisotropy in the $k$-space $[20,22]$. If it is the case, the aforementioned dips at $V_1, V_2$ correspond to larger extremum of the gap distribution $\Delta_L^{\text{max}} \approx 4.6 \text{meV}$. The features located at 6.6 and 3.3 mV are very likely the subharmonics for the lower extremum $\Delta_L^{\text{min}} \approx 3.3 \text{meV}$. Given this assumption, the large gap anisotropy may be estimated as $A = |1 - \Delta_L^{\text{min}}/\Delta_L^{\text{max}}| \cdot 100% \approx 30%$. The complex fine structure of the observed Andreev dips is caused by the $ab$-plane gap distribution; it seems to differ from the simple $\Delta(\theta) \sim \cos(\theta)$ predicted e.g. in $[10]$. In the inset of Fig. 3, we show the position of $\Delta_L$ doublets $V_n$ versus their inverse number $1/n$. The linear dependence crossing the origin proves these features are related to one and the same SGS. The above figures demonstrate the observation of IMARE in BFNA single crystals.

Two Andreev spectra of the SnS-arrays formed sequentially (after a mechanical readjustment) in one and the same BFNA sample are shown in Fig. 4. Despite the different contact resistance, the spectra look similarly and possess features for both the large and the small gap. The main doublet-like dip located at $V_1 \approx 6.4-8.5 \text{mV}$ is nearly twice wider than the second feature for the large gap at $V_2 \approx 4.4-3.2 \text{mV}$. Together, these dips are the $n = 1, 2$ subharmonics for the anisotropic large gap $\Delta_L \approx 3.2-4.4 \text{meV}$, whereas their width corresponds to $\sim 30\%$ anisotropy of the large gap. The latter value is close to that determined using data of Fig. 3. The intensive doublets at 2.4–3.2 mV do not correspond to the large gap SGS, and therefore, may be interpreted as related to the small gap with moderate anisotropy $\Delta_S \approx 1.2-1.6 \text{meV} \sim 25\%$ anisotropy). Note that for the two spectra shown in Fig. 4, the position of both-gap features coincide. The gap values remain still unchanged during the contact readjustment confirming both a high quality of cryogenic interfaces and a high homogeneity of the single crystal under study. The uniform distribution of dopants within the typical contact area reduces a contribution of $\delta T_C$-pinning mechanism (associated with a spatial variation of the superconducting transition temperature $T_C$ throughout the sample). Therefore, pinning in the studied Ba-122 samples may be caused by randomly distributed point-like pinning centers ($\delta l$-pinning) at a sub-nanometer scale $[38]$.

From the IMARE spectra, we determined the BCS-ratio $2\Delta_L/k_BT_C \approx 4.1-5.8$. It is similar to that determined earlier in IMARE experiments with K-doped Ba-122 single crystals $[28,39]$ and exceeds the standard weak-coupling limit 3.52, thus proving a strong coupling. Taking into account the moderate gap anisotropy determined here, the BCS-ratio well covers all the range of $2\Delta_L/k_BT_C$ estimated in specific heat and $H_{C1}$ measurements with the BFNA samples from the same batch $[10]$, and in infrared spectroscopy and magnetization experiments with nearly optimal K-doped Ba-122 single crystals with $T_C = 35-37 \text{K} [28,39]$. It is also consistent with other published data for Ba-122 $[7,41,42,43]$. The BCS-ratio for the small gap is $2\Delta_S/k_BT_C \approx 1.5-2.0$ and lies well below the weak-coupling limit, thus proving the presence of the $k$-space proximity effect and substantial interband interaction.  

$$\text{Fig. 3 Non-hysteretic current-voltage characteristic (green and black lines for positive-bias part and reversed negative-bias parts, correspondingly) and dynamic conductance spectrum for Andreev array in BFNA single crystal. Andreev subharmonics of the large gap are shown by arrows. The inset shows the linear dependence of subharmonics position versus } 1/n \text{ for } \Delta_L, \text{ the length of the red dashes indicate a possible gap anisotropy } \sim 30\%.$$
Fig. 4 Normalized dynamic conductance spectra of Andreev arrays (with different number of junctions) formed in BFNA single crystal. Doublet-like subharmonics for the anisotropic large gap $\Delta_L = 3.2-4.4$ meV (∼30% anisotropy) are shown by thin black lines. Arrows indicate the main doublet features of the small gap $\Delta_S = 1.2-1.6$ meV (∼25% anisotropy).

5 Conclusions

We have studied the field dependence of the critical-current density and the structure of superconducting gaps with a moderate anisotropy in a $k$-space: $\Delta_L \approx 3.3-4.5$ meV (the range corresponds to ∼30% anisotropy), $\Delta_S \approx 1.2-1.6$ meV (∼25% anisotropy). The BCS-ratio for the large gap $2\Delta_L/k_BT_C \approx 4.1-5.8 > 3.52$ is close to that for K-doped Ba-122 and evidences for a strong coupling in the $\Delta_L$-bands.

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