Two-gap superconducting properties of alkaline-earth intercalated $A_x(NH_3)Fe_2Se_2$ ($A = Ba$ or $Sr$)

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Abstract

Superconducting properties were studied on high quality superconductors $Ba_x(NH_3)Fe_2Se_2$ ($T_c = 39$ K) and $Sr_x(NH_3)Fe_2Se_2$ ($T_c = 44$ K) prepared by intercalating Ba/Sr atoms into tetragonal $\beta$-FeSe by liquid ammonia. The elongated $c$-axis and almost unchanged $a$-axis of $Ba_x(NH_3)Fe_2Se_2$, compared with $\beta$-FeSe, suggests an unchanged intra-$Fe_2Se_2$-layer structure and the $T_c$ enhancement is due to a 3D to 2D-like Fermi surface transformation. The superconducting coherent lengths $\xi(0)$, the Ginzburg–Landau parameters $\kappa$ and penetration depths $\lambda(0)$ obtained from the extrapolated lower and upper critical fields $B_{c1}(0)$ and $B_{c2}(0)$ indicate that both compounds are typical type-II superconductors. The temperature dependence of $1/\lambda^2(T)$ of $Ba_x(NH_3)Fe_2Se_2$ deduced from the low-field magnetic susceptibility shows a two-gap $s$-wave behavior with superconducting gaps of $\Delta_1 = 6.47$ meV and $\Delta_2 = 1.06$ meV.

Keywords: iron chalcogenide, two-gap $s$-wave superconductor, alkaline-earth intercalated FeSe, penetration depth, Ginzburg–Landau parameter

(Some figures may appear in colour only in the online journal)

1. Introduction

Since the discovery of iron-based superconductors in 2008 [1], the $T_c \approx 8$ K superconducting $\beta$-FeSe [2] has attracted much interest due to its simple lattice structure and the fact that it shares a common electronic origin for its superconducting mechanism with the much more complicated iron-arsenide systems [3, 4]. The $T_c$ of iron-selenide can be raised up to 14.5 K by tellurium partial substitution on the selenium site [5]. A breakthrough of the iron-selenide system was achieved by the discovery of the superconductors $A_{1-x}Fe_{2-x}Se_2$ ($A = K$, Rb, Cs or Th, FeSe-122) [6, 7], which have a high of $T_c \approx 32$ K and are a direct comparison system in terms of electronic structure with iron-arsenide 122 systems (FeAs-122) and relative pnictide systems [8–10]. However, the unavoidable nano-scaled coexistence of antiferromagnetic insulated 245-phases ($T_N \approx 470$–560 K) inside the superconducting FeSe-122 phase interferes with the discussion of fundamental superconducting and electronic properties [8–13]. Recently, an exciting development in pure superconductors with $T_c$ up to 46 K was reported for metal intercalated iron-selenides by ammonothermal reaction [14, 15]. Neutron diffraction studies indicated that ammonia molecules were inserted together with lithium atoms between the Fe$_2$Se$_2$-layers in the $T_c = 43$ K Li$_x$(NH$_3$)Fe$_2$Se$_2$ superconductor [15]. It was found that the $T_c$ of iron-selenide systems increases with increasing adjacent Fe$_2$Se$_2$-layer distance [16]. On the other hand, structural variations of intra-Fe$_2$Se$_2$-layer and electron doping as the origin of $T_c$ enhancement after Li and NH$_3$ intercalation has also been reported [15, 17].

In this report, the structural and superconducting properties of $A_x(NH_3)Fe_2Se_2$ ($A = Ba$ or $Sr$) were studied. The structural analysis suggested that the intra-Fe$_2$Se$_2$-layer structure of $Ba_x(NH_3)Fe_2Se_2$ remains the same as the parent
superconducting FeSe compound. The enhancement of $T_c$ could be attributed to the more 2D-like electronic structure from a shortened Brillouin zone $z$-axis due to inter-Fe$_2$Se$_2$-layer intercalation. The superconducting parameters, coherence length $\xi(0)$, penetration depth $\lambda(0)$ and the Ginzburg–Landau parameter $\kappa$ extracted from magnetic measurements, indicate that both systems are typical type-II superconductors. Furthermore, the superfluid density $n_s \approx \lambda^2(T)$ of Ba$_x$(NH)$_3$Fe$_2$Se$_2$ is found to be well described by a two-gap s-wave model similar to that of the parent compound [18].

2. Experimental details

High quality powder samples of A$_x$(NH)$_3$Fe$_2$Se$_2$ ($A =$ Ba, Sr) were synthesized by intercalating Ba (or Sr) atoms into tetragonal $\beta$-FeSe using liquid ammonia (LA) [14, 15]. First, high-purity superconducting $\beta$-FeSe was prepared by high temperature reactions [2]. Iron granules (99.98%) and selenium shots (99.999%) with a 1.008:1 molar ratio were placed in an alumina crucible and sealed in a quartz tube. The tube was slowly heated to 750 °C and held 1 day for complete reaction, then melted by heating to 1080 °C followed by quenching to 420 °C and held for 2 days for the pure $\beta$ phase. The intercalating reaction was carried out by placing $\beta$-FeSe powder with Ba (99.7%) or Sr (99%) metal in a 4:1 molar ratio in an evacuated autoclave cooled in a liquid nitrogen bath [14]. The gaseous ammonia was slowly condensed into a liquid until a Ba/Sr in LA concentration of 0.2–0.3 at % was reached. The vessel was kept at room temperature and magnetically stirred for 3 days. The obtained fine sample powders were then pressed into pellets and encapsulated by epoxy to prevent sample degradation due to ammonia escape. The lattice structure analysis was carried out by x-ray diffraction (XRD) with a PHILIPS X’PERT diffractometer for a 2θ range of 5°–60°. The magnetic measurements were carried out by a Quantum Design MPMS2 SQUID magnetometer with temperatures down to 5 K and an applied magnetic field up to 1 T.

3. Results and discussion

The powder XRD patterns of A$_x$(NH)$_3$Fe$_2$Se$_2$ ($A =$ Ba or Sr), as shown in figure 1, can be well indexed by a body-centered-tetragonal (bct) Li$_2$O$_4$(NH)$_3$Fe$_2$Se$_2$-type structure (space group: I4/mmm) [15]. Minor phases of impurities were barely observed for hexagonal FeSe (marked by ‘H’) and iron oxide by ‘Fe$_2$O$_3$’, and unknown phases (by asterisks). No trace of the superconducting precursor $\beta$-FeSe was observed. The derived lattice parameters for A = Ba are $a = 0.3787$ nm and $c = 1.6885$ nm, and for A = Sr are $a = 0.3834$ nm and $c = 1.7318$ nm. The greatly elongated $c$-axes observed are consistent with the literature and can be attributed to NH$_3$ molecule co-intercalation [14, 15, 19, 20]. The larger lattice parameters and unit cell volume observed for the Sr-compound ($V = 0.2542$ nm$^3$) than Ba-compound ($V = 0.2422$ nm$^3$) suggest a higher content of intercalated metal atoms in the former, which is consistent with the preliminary refinement results that the stoichiometric parameter $x$ equals ~0.25 and ~0.4 for A = Ba and Sr, respectively. However, due to the high signal-to-noise ratio, the XRD data are not conclusive enough, we left the Ba/Sr content as undetermined in the following discussion.

The observed superconducting transition temperature $T_c$ for Ba$_x$(NH)$_3$Fe$_2$Se$_2$ is 39 K and for Sr$_x$(NH)$_3$Fe$_2$Se$_2$ is 44 K, as shown in figure 2. A relatively large paramagnetic background was observed in the normal state, which could be attributed to the minor magnetic phases observed in the XRD patterns. By using the Ba and Sr contents and densities of the compounds from the XRD analysis to estimate the volume susceptibility, and further assuming a spherical powder
particle shape, which has a demagnetization factor of 1/3, simple estimations of the superconducting volume fraction at $T = 5$ K were derived as 15.9% for $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$ and 31.3% for Sr$_y$(NH$_3$)$_2$Fe$_2$Se$_2$. These volume fractions confirmed that the observed superconductivity was of bulk superconductivity for both powder compounds. The relatively small volume fraction of $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$ was due to the fine powder grain sizes of about 1–5 µm which were only slightly longer than the magnetic field penetration length $\lambda$. As a result, the 10 G applied magnetic field penetrated into the powder particles with a depth of $\sim \lambda$ and rendered the observed diamagnetic superconducting volume greatly reduced. On the other hand, the fine powder sizes similar to penetration depth also make the superconducting diamagnetic signal sensitive to the temperature variation of $\lambda(T)$. An enhancement of diamagnetism below 14 K can be clearly observed in $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$. This phenomenon is attributed to the opening of a second superconducting gap in the weak-coupling two-gap scenario. Similar behavior was hardly seen for the Sr-compound due to the much larger particle sizes of the powder, which is reflected in its larger volume fraction.

Among all intercalated iron-selenide superconductors, the enhancement of $T_c$ to around 40 K is believed to be due to the $c$-axis expansion and carrier density tuning by atom/molecule intercalation [2, 15, 19]. However, the almost unchanged $a$-axis length and very low content of inserted Ba in $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$ suggests that one can consider $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$ as directly separating Fe$_2$Se$_2$ layers in $\beta$-FeSe with inserted atoms/molecules without changing its intra-layer structure. This insertion elongates the $c$-axis length which weakens the inter-Fe$_2$Se$_2$-layer coupling. Consequently, it shortens the corresponding Brillouin zone $c'$-axis length in the reciprocal space, which makes the Fermi surfaces more cylindrical and more 2D-like, assuming the band structure is mainly determined by the almost unchanged intra-Fe$_2$Se$_2$-layer structure [17]. In addition, the intercalated Ba could donate extra electrons onto the conducting Fe$_2$Se$_2$-layer [15, 16], which increases the density of states at the Fermi level and a further enhancement of $T_c$ thus results [21].

The inset of figure 2 shows the higher-field $\chi_d$ for $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$ which were measured right after the 10 G measurement to avoid any sample degradation problems. The observed $T_c$ determined by the deviation point from the normal state decreases slightly to 37 K for $B_a = 1$ T. The magnitude of the diamagnetic signal decreases rapidly with increasing applied field. The ZFC $\chi_d(1$ T) curve did not even change sign down to 5 K due to the fine powder grain size of the sample powder and the strong paramagnetic background.

The initial mass magnetization curves, $M_s(T)$ of $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$ are shown in figure 3. The lower critical field $B_{c1}(T)$ is determined by the deviation point from the low-field linear Meissner response. The obtained $B_{c1}(T)$ decreases monotonically with increasing temperature from 28 G for $T = 5$ K, to 22 G for 20 K, and then to 6 G for 35 K. Measurements on Sr$_y$(NH$_3$)$_2$Fe$_2$Se$_2$ also revealed a similar behavior of $B_{c1}(T)$, decreasing from $B_{c1}(10$ K) = 23 G, to $B_{c1}(20$ K) = 18 G, then to $B_{c1}(30$ K) = 12 G. The observed low $B_{c1}$ values are comparable to those observed for the $B_{c2}$ axis in $\beta$-FeSe [22], despite the much higher $T_c$ in these intercalated superconductors.

The superconducting critical fields, $B_{c1}(T)$ and $B_{c2}(T)$, of $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$ obtained from magnetic measurements are summarized in a $B_{c2}$–$T$ phase diagram as shown in figure 4. The irreversible line, $B_{irr}$, determined from the deviation points of the ZFC and FC curves, is also shown as a boundary between the vortex glass and the liquid states. The zero temperature lower critical field $B_{c1}(0)$ ~30 G was easily obtained by extrapolation. An empirical value of $B_{c2}(0)$ = 13.4 T was obtained by using the Werthamer–Helfand–Hohenberg formula with a linear slope $dB_{c2}(T)/dT = -0.497$ T K$^{-1}$. The Ginzburg–Landau parameter $\kappa_{\text{Ba}} = 102$ was derived from these extrapolated $B_{c2}(0)$ and $B_{c2}(0)$ values by solving $B_{c2}/B_{c1} = 2\kappa^2/\ln\kappa$, which indicates that $\text{Ba}_x(\text{NH}_3)_2\text{Fe}_2\text{Se}_2$ is a typical type-II superconductor as expected. Since the superconducting coherent
length $\xi_0(0) = 4.96$ nm can be easily derived by using the formula $B_{c2} = \Phi_0/2\pi \xi^2$, it is straightforward to calculate the field penetration depth $\lambda_0(0) = \kappa \xi(0) = 508$ nm. Similar analysis was performed on Sr$_x$(NH$_3$)$_2$Fe$_2$Se$_2$ and the obtained critical fields were $B_{c2}(0) = 61.4$ T and $B_{c1}(0) = 24$ G, from which the superconducting parameters $\kappa_{Sr} = 266$, $\xi_{Sr}(0) = 2.33$ nm, and $\lambda_{Sr}(0) = 620$ nm were derived.

To further investigate the superconductivity of the system, the temperature dependence of the superfluid density, proportional and represented by $1/\lambda^2$, of Ba$_x$(NH$_3$)$_2$Fe$_2$Se$_2$ was estimated and is plotted in figure 5. The London penetration depth $\chi(T)$ was derived by solving the equation

$$\chi(T) = \chi_0 \sqrt{1 - \frac{3\lambda^2}{r^3}}$$

where $\chi_0$ is the susceptibility of perfect diamagnetic spheres and $g(r)$ is the grain size distribution function, which was obtained by counting sample powder grains under an optical microscope as $g(r) = 95 \exp\left(-((\log r + 0.5)/0.382)^2\right)$. A constant paramagnetic background was subtracted from the susceptibility to completely count the diamagnetic signal.

Since the 10 G applied field became larger than $B_{c2} \approx B_{c1}$ for temperatures higher than $32$ K, the penetration depth $\lambda$ values obtained at those temperatures were contaminated by vortex formation, thus the $1/\lambda^2$ data for $T > 32$ K were excluded from further discussion. At the low temperature side, $1/\lambda^2$ for temperatures below $15$ K apparently deviated from the saturation behavior of the conventional BCS single-gap model. Referring to similar systems, [18, 23] a two-gap model was used for analysis and consistent results were obtained. The temperature dependence of the penetration depth of Ba$_x$(NH$_3$)$_2$Fe$_2$Se$_2$ was fitted by a weakly coupled two-gap s-wave model [22-24]

$$\frac{\chi^2(T)}{\chi^2(0)} = \omega \frac{\chi^2(T, \Delta_i)}{\chi^2(0, \Delta_i)} + (1 - \omega) \frac{\chi^2(T, \Delta_2)}{\chi^2(0, \Delta_2)}$$

where $\lambda(0)$ is the zero temperature penetration depth, $\Delta_i$ is the $i$th superconducting gap at $T = 0$ K and $\omega$ is the weighting factor of the first gap [23]. Each component can be expressed within the local London approximation as

$$\frac{\chi^2(T)}{\chi^2(0)} = 1 + \int_{\Delta_1}^{\infty} \left( \frac{\partial f}{\partial E} \right) \frac{EdE}{\sqrt{E^2 - \Delta_i(T)^2}}$$

where $f = 1/(1 + \exp(E/k_B T))$ is the Fermi function, and the temperature dependence of the gap is approximated as $\Delta_i(T) = \Delta_i \tanh(1.82(10^{18}(T_c/T - 1)^{2}))$. Reference [18] The two-gap s-wave model, the brown curve in figure 5, describes the temperature dependence of the penetration depth very well. The zero temperature gap values obtained for Ba$_x$(NH$_3$)$_2$Fe$_2$Se$_2$ are $\Delta_1 = 6.47$ meV and $\Delta_2 = 1.06$ meV with $\omega = 0.69$. These gap values are similar to those observed in related intercalated iron-selenide systems by muon spin rotation, such as $\Delta_1 = 9.76$ meV and $\Delta_2 = 1.28$ meV of Li$_{0.6}$(NH$_3$)$_2$Fe$_2$Se$_2$ ($T_c = 43$ K) [15], and $\Delta_1 = 6.82$ meV and $\Delta_2 = 0.98$ meV of Li$_{2}$Cu$_{2}$Fe$_2$Se$_2$ ($T_c = 40$ K) [23]. The observed similar gap sizes, $T_c$, and lattice structures in these systems indicate that all three compounds have similar electronic structures and a common superconducting mechanism. On the other hand, the superconducting gaps observed are considerably smaller than the gaps of (Li$_{1-x}$Fe$_x$)OH/FeSe measured by scanning tunneling microscopy, $\Delta_1 = 14.3$ meV and $\Delta_2 = 8.6$ meV [25]. The smaller gaps in Ba$_x$(NH$_3$)$_2$Fe$_2$Se$_2$ than (Li$_{1-x}$Fe$_x$)OH/FeSe could be due to different electronic structures (or inter-band coupling) in two systems, or due to the procedure employed for penetration depth $\lambda(T)$ estimation, which is particle-shape-sensitive, and the spherical shape assumption may cause the superconducting gaps to be underestimated.

The slightly bigger value for $2\Delta_1/k_B T_c$ compared to the BCS value of $2\Delta_0/k_B T_c = 3.35$, suggests that the weak-coupling assumption in the two-gap s-wave model used is a good approximation. Since our samples were made-up of randomly orientated powder, the influence of the temperature variation of $\chi(T)$ should be observed in the diamagnetic susceptibility. However, by taking the typical anisotropy of iron-selenide systems and the obtained $\lambda(0)$, the estimated value of $\lambda_c(0)$ is about 2 $\mu$m which is larger than most (~80%) of the grain sizes of the powder. When the $c$-axes of the single crystal grains have large angles to the applied magnetic field, the magnetic field penetrates into the grains completely even at low temperatures which means that the effects of temperature dependence are not observed. Thus the estimated $\lambda(0)$ value of ~506 nm could be regarded as the upper bound of $\lambda_{Sr}(0)$ and the temperature dependence of $1/\lambda^2$ represents the supercurrent behavior in the Fe$_2$Se$_2$-layer.

In conclusion, superconducting properties of A$_x$(NH$_3$)$_2$Fe$_2$Se$_2$ ($A = Ba$ or Sr) were studied by magnetic measurements. The high $T_c$ after alkali-metal and ammonia molecule intercalation is due to a 3D-like to 2D-like Fermi
surface change as a result of c-axis elongation as well as electron doping by Ba intercalation. The temperature dependence of the London penetration depth was derived from the diamagnetic susceptibility for Ba$_x$(NH$_3$)$_3$Fe$_2$Se$_2$ which is well described by a two-gap s-wave model with gap values $\Delta_1 = 6.47 \text{ meV}$ and $\Delta_2 = 1.06 \text{ meV}$ and $\lambda(0) \sim 506 \text{ nm}$.

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