Investigation of in-plane anisotropy of c-axis magnetoresistance for BiCh2-based layered superconductor NdO0.7F0.3BiS2

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We investigated the in-plane anisotropy of the c-axis magnetoresistance (MR) in both superconducting and normal states of the single crystals of a BiCh2-based (Ch: chalcogen) superconductor NdO0.7F0.3BiS2. In the superconducting states of NdO0.7F0.3BiS2, four-fold-symmetric in-plane anisotropy of the c-axis MR was dominant below the superconducting transition temperature. Since the crystal structure of NdO0.7F0.3BiS2 is tetragonal, the rotational symmetry in the superconducting state is preserved in the present compound. This result is clearly different from the cases in LaO1−xFxBiSSe single crystals, where the in-plane MR in the superconducting state showed clear two-fold symmetry such as nematic superconductivity. These differences between four-fold and two-fold symmetry in superconducting states could be attributed to constituent elements in the conducting layer (with or without Se). Therefore, the present results propose that switching from nematic to non-nematic superconductivity states could be achieved in the BiCh2-based system. The normal-state in-plane anisotropy was also investigated for NdO0.7F0.3BiS2. © 2021 The Japan Society of Applied Physics

1. Introduction

BiCh2-based (Ch: S, Se) superconductors1–3 are a new class of layered superconductor with a structure resembling the cuprate and Fe-based high-transition-temperature (high- Tc) superconductors.4,5 In a typical system, REOBiCh2 (RE: rare earth), fluorine substitutions for the oxygen site provide electron carriers in the BiCh2 layers, which results in the emergence of metallicity and superconductivity. The superconducting mechanisms in BiCh2-based superconductors have been extensively studied from both theoretical and experimental aspects. From theoretical studies, the possibilities of conventional s-wave, extended s-wave, d-wave, and g-wave states including weak topological superconductivity states have been investigated for BiCh2-based superconductors.6–12 Thermal conductivity measurement, magnetic penetration depth, specific heat, and muon spin resonance supported s-wave pairing mechanism, but recent angle-resolved photoemission spectroscopy (ARPES) measurement indicated the presence of anisotropic superconducting gap with nodes in NdO0.7F0.3BiS2.13 Also, the anomalous superconducting gap states with multi-gap nature and strong superconducting fluctuations have been proposed from scanning tunneling spectroscopy study on Nd(O,F)BiS2.14 Furthermore, unconventional mechanisms were proposed in BiCh2-based superconductors with a tetragonal structure.15,16 Therefore, the discussion about the superconducting mechanisms in BiCh2-based superconductors is still controversial.

Recently, electronic nematicity has been a hot topic in the field of superconductivity. Electronic nematicity above a Tc has been particularly studied in Fe-based superconductors.17,18 Notably, nematicity in superconducting states (nematic superconductivity) has been reported in doped Bi2Se3.19–22 In the nematic superconductivity states, the rotational symmetry of superconducting gap amplitude breaks the underlying symmetry of the crystal lattice. Recently, we have reported on two-fold symmetric in-plane anisotropy of the c-axis magnetoresistance (MR) in the superconducting states of BiCh2-based LaO1−xFxBiSSe single crystals (x = 0.1 and 0.5).23,24 Since its crystal structure is tetragonal (P4/mmm) with four-fold symmetry in the ab-plane, the two-fold-symmetric in-plane MR suggests the emergence of nematic superconductivity in BiCh2-based superconductors. For LaO1−xFxBiSSe with x > 0.05, low-temperature structure was confirmed to be tetragonal,25 which supports the electronic origin of the two-fold-symmetric feature. However, the universality of the symmetry-breaking MR in various types of BiCh2 superconductors is still an open question. Hence, in this study, we have investigated in-plane anisotropy of the c-axis MR for NdO0.7F0.3BiS2. We found that four-fold symmetry of the c-axis MR is dominant in the superconducting states. This result is clearly different from the nematic superconducting behavior observed in LaO1−xFxBiSSe.23,24 Another difference between NdO0.7F0.3BiS2 and LaO1−xFxBiSSe was also observed in the normal states. Two-fold symmetry of the MR with a small amplitude was unexpectedly observed for NdO0.7F0.3BiS2 in the normal states. This normal-state trend is different from that observed in LaO1−xFxBiSSe. We compare the emerging in-plane anisotropy in NdO0.7F0.3BiS2 and LaO1−xFxBiSSe in both superconducting and normal states.

2. Experimental details

NdO0.7F0.3BiS2 single crystals were grown by a high-temperature flux method in an evacuated quartz tube as reported in Ref. 26. Polycrystalline powder of NdO0.7F0.3BiS2 (0.5 g), which was synthesized by the reaction of powders of Nd2O3 (99.9%), Bi2O3 (99.999%), and BiF3 (99.9%) and grains of Bi (99.99%) and S (99.99%) at 700 °C, was mixed with CsCl/KCl flux (3.0 g), and the mixture was sealed into an evacuated quartz tube. The tube was heated at 800 °C for 15 h and slowly cooled to 600 °C with a rate of −1 °C/h, followed by furnace cooling to room temperature. The product was filtered and washed by pure water. Single crystals were analyzed by scanning electron microscopy on TM3030 (Hitachi high-tech). As shown in Fig. 1(a), plate-like crystals were obtained. The plate surface with a square shape is corresponding to the...
Fig. 1. (Color online) (a) SEM image of a NdO$_{0.7}$F$_{0.3}$BiS$_2$ single crystal. (b) Schematic image of the terminal configuration for the c-axis resistivity measurement performed on NdO$_{0.7}$F$_{0.3}$BiS$_2$ single crystals. (c) Temperature dependence of the c-axis resistivity for NdO$_{0.7}$F$_{0.3}$BiS$_2$ for Sample A. The inset shows the enlarged resistivity data near a superconducting transition.

ab-plane of tetragonal NdO$_{0.7}$F$_{0.3}$BiS$_2$. The chemical composition was investigated by energy-dispersive X-ray spectroscopy on TM3030. The analyzed ratio was Nd: Bi: S = 1:1.0:2.1, which was normalized by the Nd value. The analyzed atomic ratio was almost consistent with the nominal composition.

The MR measurements were performed using a superconducting magnet at the high field laboratory of Institute for Materials Research (IMR), Tohoku University. To precisely control the magnetic field direction, a two-axes rotational probe was used. Figure 1(b) shows a schematic image of the terminal configuration for the c-axis resistivity measurements. The terminals were made using Au wires and Ag pastes.

3. Results and discussion

Figure 1(c) shows the temperature (T) dependence of the c-axis resistivity ($\rho_c$) of NdO$_{0.7}$F$_{0.3}$BiS$_2$. The inset of Fig. 1(c) shows the enlarged temperature dependence of the $\rho_c$ ($\rho_c$–T) near the superconducting transition. The estimated $T_{c\text{net}}$ and $T_{c\text{zero}}$ are 5.8 and 5.2 K. A small upturn behavior on the $\rho_c$–T is observed at low temperatures. Similar upturn behavior has been observed for some layered superconductors having two-dimensional transport characteristics, such as cuprate, Fe-based, and BiCh$_2$-based superconductors. The localization nature along the c-axis could be caused by high anisotropy between ab-plane and c-axis in a layered structure.

The definition of $\theta$ and $\phi$ angles are summarized in Fig. 2(a). $\theta$ is measured from the c-axis, and $\phi$ is azimuth angle measured from the a-axis to the b-axis, while the a-axis and the b-axis are equivalent in a tetragonal structure. Note that the charge current is always perpendicular to the magnetic field when the magnetic field is in the conducting plane since the c-axis resistivity was measured. Hence, the Lorentz force on the vortices is basically identical with respect to the $\phi$ angle dependence. Figure 2(b) shows the $\theta$ angle dependence of the $\rho_c$ at $\phi = 180^\circ$, $B = 14$ T, and $T = 2.7$ K for Sample A. As shown in Fig. 2(b), $\rho_c^{B|ab}$, which was defined as the MR where the magnetic field is exactly parallel to the ab-plane, was estimated and used to investigate the in-plane anisotropy.

Figure 2(c) shows the $\phi$ angle dependences of the $\rho_c^{B|ab}$ at $B = 14$ T measured at temperatures from 10.0 to 2.5 K for Sample A. The plotted data at $T = 2.5$ and 2.7 K were fitted by a function of $\cos \{2(\phi - 45)\} + \cos \{4(\phi - 45)\} + C$ with positive coefficients. In this analysis, the phases of cosine functions are chosen for [100] or [110] directions. In the nematic state, the [100] direction is different from [010] as well as the relation between [110] and [−110]. The plotted data for $T = 3.0$ K will be discussed later since the data were fitted by different phase parameters. The estimated amplitude constants A, B, and C are 12 ± 5, 39 ± 5, and 245 ± 4 m$^2$ cm at $T = 2.5$ K and 8 ± 4, 18 ± 4, and 439 ± 3 m$^2$ cm at $T = 2.7$ K, respectively. The coefficient B, which is related to four-fold symmetric oscillation, is larger than A, which is related to two-fold symmetric one. The result indicates that the four-fold symmetric component is more dominant than that for two-fold symmetric oscillation at $T = 2.5$ and 2.7 K. The phase parameters exhibit directions of the minimum value of $\rho_c^{B|ab}$. When a four-fold-symmetric component is dominant, the phase parameters of 0° and 45° present that the direction of the minimum $\rho_c^{B|ab}$ is along [110] and [100], respectively. When a two-fold-symmetric component is dominant, the phase parameters of 0° or 90° and 45° or 135° present that the direction of the minimum $\rho_c^{B|ab}$ is along [010] or [001] and [110] or [−110], respectively. Four-fold components of $\cos \{4(\phi - 45)\}$ and $\cos \{4(\phi - 45)\}$ exhibit [110] and [100] directions, respectively. The two-fold-component of $\cos \{2(\phi - 90)\}$ and $\cos \{2(\phi - 90)\}$ exhibit the [100] or [010] and the [110] or [−110] directions, respectively. Therefore, we conclude that the direction of minimum $\rho_c^{B|ab}$ in the superconducting states is along [100]. As a result, four-fold symmetry of the $\rho_c^{B|ab}$ was observed in the superconducting states at $T = 2.5$ and 2.7 K, which is normally expected.
because the $ab$-plane of NdO₀.₇F₀.₃BiS₂ has a tetragonal (fourfold-symmetric) Bi–S plane. The result suggests that rotational symmetry in the crystal lattice is preserved when we see the in-plane anisotropy of the superconducting properties in NdO₀.₇F₀.₃BiS₂. We discuss the observed four-fold symmetry in the superconducting states by comparing that with the previous reports on superconducting states of NdO₀.₇F₀.₃BiS₂. Nodeless superconductivity was suggested for NdO₀.₇F₀.₃BiS₂ from magnetic penetration depth measurements and thermal conductivity measurements.³⁰,³¹ In contrast, the ARPES study suggested that superconducting gap are strongly anisotropic and have nodelike minima.¹³ In addition, the authors of Ref. 13 suggested that these results, which seems inconsistent between magnetic penetration depth and ARPES results, do not conflict if superconducting gap symmetry of NdO₀.₇F₀.₃BiS₂ is $s$-wave and the superconducting gap nodes, which were observed by ARPES, are accidental ones. Our present results (four-fold symmetry in the superconducting states) are also reasonable if the anisotropic $s$-wave scenario ($s$-wave with accidental nodes) is essential in NdO₀.₇F₀.₃BiS₂.

The results are, however, clearly different from the two-fold symmetric MR observed in the superconducting states of LaO₁₋ₓFₓBiSₓSe single crystals²⁴,²⁵ and suggest that the superconducting states for LaO₁₋ₓFₓBiSₓSe and NdO₀.₇F₀.₃BiS₂ are totally different. We show the data for in-plane anisotropy of the MR in the superconducting states for LaO₀.₅F₀.₅BiSₓSe in Fig. 2 (d).²³ Although we cannot clarify the origins of the different behavior of in-plane anisotropy of MR in these two systems, we here briefly discuss the possible explanations for the different behaviors of the in-plane anisotropy for NdO₀.₇F₀.₃BiS₂ and LaO₁₋ₓFₓBiSₓSe. First, in LaO₁₋ₓFₓBiSₓSe, two-fold-symmetric behavior was observed for $x = 0.1$ and 0.5. Since the carrier concentration and the Fermi surface topology could be different for those samples with different F concentrations ($x = 0.1$ and 0.5), one can assume that the emergence of two-fold-symmetric behavior is not directly affected by the carrier concentration. Then, we compare the constituent elements in the superconducting layers in NdO₀.₇F₀.₃BiS₂ and LaO₁₋ₓFₓBiSₓSe. NdO₀.₇F₀.₃BiS₂ contains Bi–S superconducting planes, while LaO₁₋ₓFₓBiSₓSe contains Bi–Se superconducting planes, according to the structural analysis of LaO₁₋ₓFₓBiSₓSe.³² The Se substitution for S at the superconducting plane can enhance spin–orbit coupling (SOC), and the SOC has been suggested as an important parameter to understand the electronic states and superconducting characteristics in the BiCh₂-based systems.³³–³⁵ Rashba–Dresselhaus SOC has been predicted and indeed observed by high-resolution spin- and angle-resolved photoemission spectroscopy measurement for BiCh₂-based superconductor.³³,³⁴ Furthermore, a large in-plane $H_||$ was proposed to be originated from the spin-momentum locked superconductivity due to the Rashba–Dresselhaus SOC in LaO₀.₅F₀.₅BiSₓSe.³⁵ Since the spin-singlet and triplet superconducting states can be mixed by Rashba–Dresselhaus SOC, we consider that the different in-plane anisotropy in NdO₀.₇F₀.₃BiS₂ and LaO₁₋ₓFₓBiSₓSe would be related to the different magnitude of SOC. More detailed studies are needed to clarify the mechanisms of the emergence of nematic superconductivity in BiCh₂-based superconductors.

For the analysis of the data at 3.0 K, which is near $T_c$, a function of $\cos(2 \phi) + \cos(4 \phi - 45) + C$, whose phase parameter is different from that used for $T = 2.5$ and 2.7 K, was used for fitting since the fitting with the function was better than other phase parameters in [100] and [110] directions. The constants $A$, $B$, and $C$ are estimated as $6 ± 3$, $5 ± 3$, and $570 ± 2$ m$\Omega$ cm; the large errors are due to the difficult fitting at this temperature. The $A$ related to two-fold symmetry is comparable to $B$ related to four-fold symmetry at $T = 3.0$ K. As well, the normal-state properties, shown in Fig. 2(a), were fitted by the function of $\cos(2 \phi - 135) + \cos(4 \phi) + C$. The estimated amplitude constants $A$, $B$, and $C$ are $5.9 ± 0.5$, $0.7 ± 0.5$, and $650.1 ± 0.3$ (3) m$\Omega$ cm at $T = 5.0$ K and $4.8 ± 0.4$, $0.5 ± 0.4$, and $625.8 ± 0.3$ at $T = 10.0$ K, respectively. The constant $A$ much larger than $B$ indicates that the two-fold-symmetric in-plane anisotropy of the MR was observed in the normal states. Two-fold symmetry was observed in the normal states, while four-fold symmetry was observed for superconducting states. Although the two-fold-symmetric behavior in the normal states is similar to the electronic nematicity observed in Fe-based superconductors, we need to investigate the phenomenon...
with various experimental and theoretical probes to clarify the origin of it. The minimum of the $\rho_{\phi}^{B|ab}$ in the normal states for Sample A is along [110] since the phase parameter of two-fold-symmetric component is 135°.

For comparison, the normal-state MR anisotropy for LaO$_{0.5}$F$_{0.5}$BiSSe is shown in Fig. 3(b), in which four-fold symmetry is clearly dominant. Note that the amplitude of those signals in the normal states is very small as shown in symmetry is clearly dominant. Note that the amplitude of those signals in the normal states is very small as shown in Figs. 2(c) and 2(d). However, there are differences in normal-state characteristics in those two systems. The two-fold symmetry in the normal states of NdO$_{0.7}$F$_{0.3}$BiS$_2$ cannot be concluded with current results only but may be related to the checkerboard stripe-type electronic-ordered (local charge-ordered) states observed in scanning tunneling microscopy measurements in comparable single crystals. The checkerboard stripe electronic state can originate from nonequivalent nature between the Bi$_6$-$p_x$ and Bi$_6$-$p_y$ bands which constitute conduction bands near the $E_F$. Two-fold symmetry in the normal states may emerge by the one-dimensional stripe structure. The local electronic ordered states should be related to the structural instability in the conducting plane. Although both phases have a tetragonal structure, there is in-plane local disorder and that is larger in Nd(O,F)BiS$_2$ than La(O,F)BiSSe. Although the scenario seems reasonable to understand both superconducting- and normal-state in-plane anisotropy of MR in Nd(O,F)BiS$_2$ and La(O,F)BiSSe, further experimental investigations on in-plane anisotropy and theoretical investigation for other BiCh$_2$-based systems are needed to understand the origins of variable in-plane anisotropy.

In Supplemental data, the set of experimental data of in-plane anisotropy of MR examined on another crystal obtained from the different batch (Sample B) is shown. The same trends were observed in two different crystals. However, the direction of the minimum of the $\rho_{\phi}^{B|ab}$ in the normal states for the Sample B is different from Sample A while the direction in the superconducting states for the Sample B is equal to Sample A. The minimum of the $\rho_{\phi}^{B|ab}$ in the normal states for Sample B is along [100] since the phase parameter of two-fold-symmetric component is 90°. [See supplementary information (available online at stacks.iop.org/JJAP/60/020907/mmedia).] At present, the origin for the sample dependence in the normal-state anisotropy is an open question.

To clearly show the evolutions of four-fold-symmetric and two-fold-symmetric components from the superconducting states to the normal states, we estimated the ratio of the amplitude for fitting function in Figs. 2(c) and 3. Figure 4 shows the temperature dependence of the B/A and C. The B/A below 1 indicates that two-fold-symmetric component is dominant. We determined the boundary between superconducting and normal states (indicated by the gray line) as the temperature at which $C(T)$ becomes the half of $C (T = 5.0 \text{K})$ and showed in Fig. 4. The temperature dependence of $C$ is close to resistivity at the mid-point $T_c$ in the temperature dependence of resistivity. The switching between two-fold symmetry and four-fold symmetry with decreasing temperature (decreasing $C$) is visually clear, and the boundary temperature is located near $T_s$.

4. Conclusion

We have investigated the in-plane anisotropy of the $c$-axis MR in both superconducting and normal states for single crystals of BiCh$_2$-based NdO$_{0.5}$F$_{0.5}$BiS$_2$ under in-plane magnetic fields. In the superconducting states, four-fold-symmetric in-plane anisotropy of MR was observed below $T_s$. Since the crystal structure of NdO$_{0.7}$F$_{0.3}$BiS$_2$ is tetragonal, the results suggest no symmetry breaking in the in-plane anisotropy of MR in the superconducting states. We consider that the four-fold symmetry of the MR in the superconducting states is consistent with previous studies. The four-fold-symmetric in-plane anisotropy in the superconducting states is clearly different from that observed for LaO$_{1-x}$F$_x$BiSSe single crystals, in which two-fold-symmetric anisotropy of MR was observed in its superconducting states. The constituent elements in the conducting layer might lead to the difference.

Fig. 3. (Color online) (a) $\phi$ angle dependences of the normal-state MR for NdO$_{0.5}$F$_{0.5}$BiS$_2$ measured at $B =$14 T in normal states at $T =$5.0–10.0 K for Sample A. The plotted data were fitted by the function of $A \cos(2(\phi - 135)) + B \cos(4\phi) + C$ (blue broken line). (b) $\phi$ angle dependences of the normal-state MR for LaO$_{0.5}$F$_{0.5}$BiSSe measured at $B =$15 T and at $T =$5.0 K. The plotted data were fitted by the function of $A \cos(2(\phi - 90)) + B \cos(4(\phi - 45)) + C$ (blue broken line).
Fig. 4. (Color online) Temperature dependence of $B/A$ (red circles) and $C$ (blue circles) obtained from the fitting for Sample A. The $B/A$ parameter larger than 1 indicates that the four-fold-symmetric component is dominant, and that smaller than 1 indicates that the two-fold-symmetric component is dominant. The gray line shows the boundary between superconducting states and normal states defined as the half of the $C$ at $T = 5.0$ K. $T_c$ is defined as the temperature at which $C(T)$ becomes the half of $C (T = 5.0$ K).

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