Two-fold symmetry of in-plane magnetoresistance anisotropy in the superconducting states of BiCh$_2$-based LaO$_{0.9}$F$_{0.1}$BiSSe single crystal

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Abstract

Recently, two-fold symmetric in-plane anisotropy of the superconducting properties have been observed in a single crystal of BiCh$_2$-based (Ch: S, Se) layered superconductor LaO$_{0.3}$F$_{0.5}$BiSSe having a tetragonal (four-fold-symmetric) in-plane structure; the phenomena are very similar to those observed in nematic superconductors. To explore the origin of the two-fold symmetric anisotropy in the BiCh$_2$-based system, we have investigated the electron-doping dependence on the anisotropy by examining the in-plane anisotropy of the magnetoresistance in the superconducting states for a single crystal of LaO$_{0.9}$F$_{0.1}$BiSSe under high magnetic fields up to 15 T. We observed a two-fold symmetry of in-plane anisotropy of magnetoresistance for LaO$_{0.9}$F$_{0.1}$BiSSe. The results obtained for LaO$_{0.9}$F$_{0.1}$BiSSe are quite similar to those observed for LaO$_{0.5}$F$_{0.5}$BiSSe, which has a higher electron doping concentration than LaO$_{0.9}$F$_{0.1}$BiSSe. Our present finding suggests that the emergence of the in-plane symmetry breaking in the superconducting state is robust to the carrier concentration in the series of LaO$_{1-x}$F$_x$BiSSe.

1. Introduction

The BiCh$_2$-based (Ch: S, Se) superconductor family [1–3], discovered in 2012, has been extensively studied because of the crystal structure similar to the cuprate- and the iron-based high-transition-temperature (high-$T_c$) superconductors [4, 5]. Particularly, the crystal structure of the REOBiCh$_2$ (RE: rare earth) systems resembles that of the iron pnictide system REOFeAs [5]. The typical REOBiS$_2$ system has a layered crystal structure composed of the REO insulating layer and the BiCh$_2$ conducting layer, as shown in figure 1(a) [2, 3]. The F substitution for the O site generates electron carriers in the BiCh$_2$ layers, which results in the emergence of superconductivity. The highest $T_c$ among the BiCh$_2$-based superconductors is 11 K for LaO$_{0.5}$F$_{0.5}$BiS$_2$ prepared under high pressure [6, 7]. The pairing mechanisms of the superconductivity for the BiCh$_2$-based superconductors have not been clarified. Recent theoretical calculations and angle-resolved photoemission spectroscopy (ARPES) have suggested that the unconventional pairing mechanisms are essential for the superconductivity of the BiCh$_2$-based superconductors [8, 9]. Moreover, a study of selenium isotope effect for the BiCh$_2$-based superconductor LaO$_{0.6}$F$_{0.4}$BiSSe revealed that the isotope effect exponent ($\alpha_{Se}$) on $T_c$ was close to zero ($-0.04 < \alpha_{Se} < +0.04$). This result cannot be explained by the electron-phonon mechanism and supports unconventional pairing mechanisms [10].

Recently, particularly in the layered superconductors, nematicity has been a hot topic due to the possible relation to the unconventional superconducting states. Electronical nematicity has been observed in various
unconventional superconductors, such as the cuprate and the iron pnictide superconductors. Those superconductors show the rotational symmetry breaking while the structural symmetry has been maintained as above the $T_c$ \[11, 12\].

As a superconducting counterpart of normal-state electric nematicity, nematic superconductivity has been observed in A$_x$Bi$_2$Se$_3$ (A = Cu, Sr, Nb) \[13–17\]. The Cu$_x$Bi$_2$Se$_3$ superconductor is known as an odd-parity superconductor and can be categorized as a topological superconductor. In the superconducting state of A$_x$Bi$_2$Se$_3$, two-fold symmetry of the superconducting gap was observed while the crystal structure has a hexagonal superconducting plane with three-fold symmetry. The unconventional superconducting states in doped Bi$_2$Se$_3$ are related to the strong spin-orbit coupling and multi-orbital characteristics. The BiCh$_2$-based superconductors also possess a strong spin-orbit coupling due to Bi-6p electrons and local inversion symmetry breaking due to the van der Waals gap of the BiCh$_2$ bilayers \[18–21\]. In addition, a thin film of a related compound LaOsBiSe$_2$ was predicted as the Dirac material \[22\]. On the basis of these similarities to the A$_x$Bi$_2$Se$_3$ system, nematic or related exotic phenomena can be expected for the superconducting states of the BiCh$_2$-based compounds. Indeed, two-fold symmetric in-plane anisotropy was recently observed in the superconducting states of LaO$_{0.9}$F$_{0.1}$BiSSe, whose conducting plane has a four-fold tetragonal symmetry \[23\]. This observation suggests that the BiCh$_2$ superconductors are promising candidates of nematic superconductor. In this study, we show the in-plane anisotropy of magnetoresistance for LaO$_{0.9}$F$_{0.1}$BiSSe with a smaller electron doping concentration than LaO$_{0.5}$F$_{0.5}$BiSSe. As similar to the previous study, the magnetoresistance of the present sample also showed two-fold-symmetric behavior in the superconducting state, suggesting the universal behavior of nematic-supercconductivity-like phenomena is in the LaO$_{1-x}$F$_x$BiSe systems. This study shows that the BiCh$_2$-based compound family will be a useful platform to study the physics and chemistry of nematic superconductivity in layered materials.

2. Experimental details

LaO$_{0.9}$F$_{0.1}$BiSSe single crystals were grown by a high-temperature flux method in an evacuated quartz tube. Polycrystalline LaO$_{0.9}$F$_{0.1}$BiSSe was prepared using the solid-state-reaction method using powders of La$_2$O$_3$ (99.9%), La$_5$S$_3$ (99.9%), Bi$_2$O$_3$ (99.999%), and BiF$_3$ (99.9%) and grains of Bi (99.999%) and Se (99.999%) \[24\]. A mixture of the starting materials was mixed with a nominal ratio of LaO$_{0.9}$F$_{0.1}$BiSSe, pressed into a pellet and annealed at 700 °C for 20 h in an evacuated quartz tube. The polycrystalline LaO$_{0.9}$F$_{0.1}$BiSSe (0.62 g) were mixed with CsCl flux (2.2 g), and the mixture was sealed into an evacuated quartz tube. The tube was heated at 900 °C for 12 h, slowly cooled to 645 °C with a rate of 1.0 °C h$^{-1}$, and furnace-cooled to room temperature. After furnace cooling, the quartz tube was opened under air atmosphere, and the product was filtered and washed with pure water. The chemical composition of the obtained crystal was investigated using energy-dispersive x-ray spectroscopy (EDX) spectroscopy. The average compositional ratio of the constituent elements (except for O and F) was estimated to be La: Bi: S: Se = 1: 0.99: 0.93: 1.0, which was normalized by the La value. The analyzed
atomic ratio is almost consistent with the nominal composition LaO$_{0.9}$F$_{0.1}$BiSe. Considering the typical detection error in the EDX analysis, we regard the composition of the examined crystal as LaO$_{0.9}$F$_{0.1}$BiSe.

The single crystals were ground with quartz powders to get homogeneous powders for the synchrotron powder x-ray diffraction (SXRD) experiment [25]. The SXRD was performed at the beamline BL02B2 SPring-8 at a wavelength of 0.495 274 Å (proposal No. 2018B1246). The SXRD experiments were performed with a sample rotator system at room temperature; the diffraction data were collected using a high-resolution one-dimensional semiconductor detector (multiple MYTHEN system [26]) with a step size of 2θ = 0.006°. The crystal structure parameters were refined using the Rietveld method with the RIETAN-EP software [27]. The schematic image of the crystal structure refined by the Rietveld refinement was depicted using the VESTA software [28].

The resistive anisotropy was investigated under magnetic fields up to 15 T using a superconducting magnet at the Institute for Materials Research (IMR) of Tohoku University. To precisely investigate the anisotropy, a $^3$He probe equipped with an accurate two-axes rotator system was used. The electrodes were fabricated using Au wires and Ag pastes. DC current of 50 µA with polarity change to subtract thermoelectric voltage was used for the experiments.

3. Results and discussion

Figure 1(b) shows the SXRD pattern for the powdered sample of LaO$_{0.9}$F$_{0.1}$BiSe. The SXRD pattern was refined using the tetragonal (P4/mnm) structural model. The refined lattice parameters are $a = 4.108$ 49(9) Å and $c = 13.6851(4)$ Å. The lattice parameters $a$ and $c$ are slightly smaller than those obtained with a polycrystalline sample with $x = 0.1$ in [24], and the lattice constant $c$ for the crystal was comparable to that observed for $x = 0.3$. Since the lattice constant sometimes differs between single and polycrystalline samples in BiCh$_2$-based superconductors, we use the analysis result on the lattice constant for the confirmation that electron carrier has been doped in the crystal. As mentioned later, from the estimation of $T_c$, we could assume that the carrier doping amount is close to the starting nominal value of $x = 0.1$. In the Rietveld refinement, we assumed that the in-plane Ch1 site (see figure 1(a)) was fully occupied by Se, and the Ch2 site was fully occupied by S, on the basis of the EDX analysis result and previous structural analysis [24]. The obtained reliable factor $R_{wp}$ was 11.5%. In fact, the 00l peaks and others related to $c$-axis direction are broadened and have a shoulder (see supplemental information is available online at stacks.iop.org/JPCO/4/095028/mmedia), which should be resulting in a slightly high $R_{wp}$. This may be due to the strain introduced during the sample preparation by grinding with quartz powders and the sticky nature of the crystals. However, the higher angle fitting with the tetragonal model (see the inset of figure 1) is quite nice. In addition, we did not obtain better $R_{wp}$ with monoclinic model.

Although the LaO$_{1-x}$Fe$_x$BiSe phase undergoes a structural transition from tetragonal to monoclinic (P2$_1$/m), our recent study suggested that the transition is suppressed by 3% substitution of O by F [29]. Therefore, the crystal structure of the present crystal with nominal $x = 0.1$ can be regarded as tetragonal with four-fold symmetry in the conducting plane down to low temperature near $T_c$.

Figure 2 shows the temperature dependence of electrical resistivity for LaO$_{0.9}$F$_{0.1}$BiSe single crystal measured with current along the $c$-axis. The $T_c$ onset is 3.4 K, and the $T_c$ zero is 2.9 K. These $T_c$ values are comparable to those observed for polycrystalline samples with $x = 0.1$. Figure 3(a) shows the magnetic field dependence of resistivity where the magnetic field perpendicular to the $c$-axis was applied. The superconducting states are suppressed with increasing magnetic field. Figure 3(b) shows the temperature dependence of the in-plane upper critical field $\mu_0H_{c2}$, estimated from the midpoint $T_c$ ($T_c^{\text{mid}}$) in the superconducting transition, for LaO$_{0.9}$F$_{0.1}$BiSe. From the linear extrapolation of the data, $\mu_0H_{c2}$ reaches 20 T at 0 K. The $\mu_0H_{c2}(0)$ estimated from Werthamer-Helfand-Hohenberg model [30] was 13.8 T. The high $\mu_0H_{c2}$ with $H/ab$ is consistent with previous reports for BiCh$_2$-based superconductors [3, 21].

Figure 4(a) shows a schematic image of the terminal configuration for the in-plane anisotropy measurement. To investigate the in-plane anisotropy in the superconducting states, the crystal was rotated using two rotation angles. $\theta$ and $\phi$ are defined as shown in figure 4(b). $\theta$ is defined as the formed angle from the $c$-axis to $ab$-plane and $\phi$ is defined as the formed angle from the $a$-axis to the $b$-axis. The angle dependences of resistivity were investigated for the superconducting states in between the onset and the zero-resistance states by tuning the temperature and magnetic field based on the obtained field-temperature phase diagram (figure 3(b)).

Figure 4(c) shows the $\theta$ angle dependence of the electronic resistivity at $\phi = 180^\circ$, $\mu_0H = 4$ T, and $T = 2$ K. The $\rho_{\min}$ is defined as the minimum electronical resistivity, which was at $\theta = 90^\circ$ in the angle scan.

Figure 4(d) shows the $\phi$ angle dependence of $\rho_{\min}$ where the in-plane anisotropy of the magnetoresistance in the superconducting states (or upper critical field) can be investigated from the $\phi$ scan of $\rho_{\min}$. Although the crystal possessed a four-fold symmetry in its in-plane structure as discussed above, an oscillation of $\rho_{\min}$ with a period of about $180^\circ$ in figure 4(d). Furthermore, the plotted data was well fitted by the function of
A \cos \{2(\phi-\alpha)\} + B \cos \{4(\phi-\beta)\} + C. The estimated amplitude constants $A$, $B$, and $C$ were 12.8(9), $-2.9(9)$, and 44.9(6) m$\Omega$cm. Since the constant $A$ related to the two-fold symmetry oscillation is clearly larger than $B$ related to the four-fold symmetry, the appearance of the two-fold symmetry in the in-plane anisotropy of the magnetoresistance in the superconducting states should be essential. In contrast, almost $\phi$-independent behavior was observed in the normal states (see supplemental information). Therefore, the two-fold symmetric behavior in LaO$_{0.9}$F$_{0.1}$BiSe is the characteristics of the superconducting states. The phase factor $\alpha$ and $\beta$ are 28(2) and $-48(5)$ deg., respectively. In this experimental setup, uncertainty of the in-plane angle (about 10 deg.) is expected. Therefore, the error bars of the x-axis are added in figure 4(d). As shown in figure 4(d), the present results suggest that the direction of the minimum resistivity is observed between the $[\overline{1}00]$ and $[\overline{1}10]$ direction. However, it is difficult to determine the principle axis of the two-fold symmetry from the present data due to uncertainty of the in-plane angle. The roughly-estimated $\mu_0H_{c2}$ oscillation amplitude $\Delta\mu_0H_c \sim 2A/(d\rho_c/dH)$ at $T = 2$ K and 4 T is 1.2 T, which is comparable to Cu$_2$Bi$_2$Se$_3$ [14]. We have confirmed the emergence of the two-fold symmetric behavior in the superconducting states using a crystal taken from another batch of the sample (see supplemental information).

As described in the introduction, similar two-fold-symmetric in-plane anisotropy of magnetoresistance in the superconducting states has been observed for LaO$_{0.5}$F$_{0.5}$BiSe. Since the present experiments revealed that LaO$_{0.9}$F$_{0.1}$BiSe also exhibits the two-fold symmetry in the superconducting states, we conclude that the carrier

Figure 2. Temperature dependence of electrical resistance (measured with a current along the $c$-axis) of LaO$_{0.9}$F$_{0.1}$BiSe single crystal from 2 to 5 K at 0 T.

Figure 3. (a) In-plane field dependence of electronical resistance at 2.5 K, 2.0 K, 1.6 K, 1.0 K and 0.6 K, respectively. (b) The temperature dependence of $\mu_0H_{c2}$. 

Figure 4. (a) In-plane field dependence of electronical resistance at 2.5 K, 2.0 K, 1.6 K, 1.0 K and 0.6 K, respectively. (b) The temperature dependence of $\mu_0H_{c2}$. 

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concentration is not an essential parameter for the condition of the appearance of the phenomena in the LaO$_{1-x}$F$_x$BiSSe system. This fact surprised us because the Fermi surface topology is largely different between electron doping concentrations of $x = 0.1$ and $x = 0.5$ in the BiCh$_2$-based compounds [31]. However, it is possible that the direction of the minimum resistivity (nematicity) is different between $x = 0.5$ and 0.1 because the principle axis of the magnetoresistance plot in the superconducting state lies along the [100] direction for $x = 0.5$ [23], but that for $x = 0.1$ (present data) lies between the [100] and [110] direction. To clarify the relationship between the carrier concentration and the nematic superconducting states in LaO$_{1-x}$F$_x$BiSSe, further experiments are needed.

The phenomena shown here, which is similar to nematic superconductivity states in doped Bi$_2$Se$_3$ systems, will motivate further experiments in LaO$_{1-x}$F$_x$BiSSe and related systems. Although there are no theoretical studies predicting the emergence of nematic superconductivity in the BiCh$_2$-based compounds, a theoretical study predicted the possible topological superconductivity in BiCh$_2$-based systems [32]. Moreover, local Rashba-Dresselhaus effect has been proposed in BiCh$_2$-based systems due to the breaking of local inversion symmetry in each BiCh$_2$ bilayer in spite of possessing global inversion symmetry [33]. Actually, the local Rashba-Dresselhaus spin polarization has been experimentally observed in LaO$_{0.55}$F$_{0.45}$BiS$_2$ by high-resolution spin- and angle-resolved photoemission spectroscopy [34]. Furthermore, one group indicated that the Rashba-Dresselhaus spin-orbit coupling (SOC) can play an important role for high in-plane $H_{c2}$ for LaO$_{0.5}$F$_{0.5}$BiS$_2$ [21]. If the Rashba-Dresselhaus SOC significantly affects the superconducting states of BiCh$_2$-based superconductors, spin-singlet and spin-triplet states can be mixed in its superconducting states. Two-fold symmetric superconducting gap on the $ab$-plane can be observed when spin-triplet component such as $p_x$ and $p_y$-wave state is more dominant than spin-singlet. A theoretical study has proposed that BiCh$_2$-based superconductor may have a dominant triplet component [32]. Although there is still no experimental evidence of the triplet pairing in BiCh$_2$-based systems, the two-fold symmetry on the $ab$-plane in the superconducting states observed in LaO$_{1-x}$F$_x$BiSSe may be related to the possible presence of spin-triplet
component in the pairing states. Since the pairing mechanisms of the BiCh$_2$-based superconductors have not been concluded [31], further theoretical and experimental investigations are needed, and the present result on the in-plane anisotropy of the superconducting states in LaO$_{1-x}$F$_x$BiSSe should be one of the key information for the goal.

4. Conclusion

We have investigated the transport properties of a single crystal of BiCh$_2$-based superconductor LaO$_{0.9}$F$_{0.1}$BiSSe under high magnetic fields up to 15 T. From the $c$-axis electrical resistance (measured with a current along the $c$-axis), the upper critical field was determined. Also, the in-plane anisotropy of the electrical resistance was investigated using a $^3$He probe equipped with a two-axes rotator system to investigate the in-plane anisotropy of magnetoresistance. From the in-plane anisotropy measurements, we observed two-fold symmetry of magnetoresistance in the superconducting states within the $ab$ plane of LaO$_{0.9}$F$_{0.1}$BiSSe. Since the crystal possessed a tetragonal square plane with a tetragonal (four-fold) in-plane symmetry, the appearance of two-fold symmetry indicates the rotational symmetry breaking in the superconducting states. The phenomena are very similar to those observed for LaO$_{0.5}$F$_{0.5}$BiSSe with a higher electron doping concentration. Therefore, we conclude that the carrier doping concentration, which affects the Fermi surface topology, is not an essential parameter for the emergence of the nematic-superconductivity-like phenomena in LaO$_{1-x}$F$_x$BiSSe. We hope that the results shown here are useful for further investigation on superconductivity pairing mechanisms of the BiCh$_2$-based compounds and related studies on nematic superconductivity in layered systems.

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