1. Introduction

Recently, nematic superconductivity (NSC), which is typically characterized by spontaneous rotational symmetry breaking in the amplitude of the superconducting gap, has been observed in superconducting states of a topological superconductor system $A_x Bi_y Se_z$ ($A = \text{Cu, Sr, Nb}$).\textsuperscript{[11–17]} Although $A_x Bi_y Se_z$ has a trigonal structure, the observed in-plane anisotropy of superconducting properties in these superconductors exhibits twofold symmetry.\textsuperscript{[2–46]} This behavior indicates rotational symmetry breaking in the superconducting states and is called NSC. Because the systems in which NSC has been observed are limited, a new system that shows NSC is desired to obtain further knowledge about the emergence of NSC states in layered materials.

Very recently, in single crystals of LaO$_{1-x}$F$_x$BiS$_2$ ($x = 0.1$ and 0.5), which is a BiCh$_2$-based layered superconductor (Ch: S, Se),\textsuperscript{[8–10]} twofold symmetric in-plane anisotropy of magnetoresistance (MR) was observed in the superconducting states,\textsuperscript{[11,12]} while those phases have tetragonal symmetry.\textsuperscript{[13]} These experimental results proposed BiCh$_2$-based superconductors as a new platform to study NSC. As both samples with different electron-doping concentrations ($x = 0.1$ and 0.5) exhibit NSC, the Fermi surface topology does not seem to affect the emergence/disappearance of NSC in LaO$_{1-x}$F$_x$BiS$_2$. However, in another BiCh$_2$-based superconductor, NdO$_{0.7}$F$_{0.3}$BiS$_2$, no features of NSC were observed in its superconducting states. The MR in the superconducting states exhibited fourfold symmetric in-plane anisotropy.\textsuperscript{[14]} According to theoretical investigations of upper critical field ($B_c$) in tetragonal systems with a multicomponent order parameter, fourfold symmetric anisotropy of $B_c$ was suggested.\textsuperscript{[15,16]} From the viewpoint, fourfold symmetric character observed in NdO$_{0.7}$F$_{0.3}$BiS$_2$ is consistent with the observation of anisotropic superconducting gap in angle-resolved photoemission spectroscopy for Nd(O,F) BiS$_2$.\textsuperscript{[17]} Then, the question arises as to what the origins of the switching behavior between twofold and fourfold characteristics are. Therefore, further examples which show twofold symmetric anisotropy (NSC) or fourfold symmetric anisotropy are needed to understand the factor, essential to the emergence/disappearance (switching) of NSC in the BiCh$_2$-based system REOBiCh$_2$ (RE: La, Ce, Pr, Nd, Sm). To investigate the origin of the switching phenomena, a comparison of the properties of the phases with different in-plane chemical pressure (CP) magnitudes should provide key information.\textsuperscript{[18]}

In general, REOBiCh$_2$ is a semiconductor with a bandgap, and electron-carrier doping is needed to induce superconductivity.\textsuperscript{[10]} However, some of the electron-doped systems, such as LaO$_{1-x}$F$_x$BiS$_2$, do not exhibit bulk superconductivity. In BiCh$_2$-based compounds, the superconducting properties are strongly affected by the presence of in-plane local disorder, which locally lowers the in-plane structural symmetry of the Bi-Ch$_1$ network (see Figure 1b for the definition of the Ch1 site).\textsuperscript{[18–21]} By increasing in-plane CP, local structural disorder could be suppressed, and bulk superconductivity is induced in electron-doped REOBiCh$_2$. $T_c$ also increases with increasing in-plane CP. There are two ways to apply positive CP in the plane of the REOBiCh$_2$ system: 1) using a smaller RE and 2) substituting Se for the S site. The substitution with a smaller RE compresses the lattice along the $a$ axis. In addition, if the REO blocking layer is fixed, the substitution of larger Se for the site of smaller S results in a positive in-plane CP, as discussed through synchrotron X-ray diffraction (XRD) analyses,\textsuperscript{[18]} and the Se substitution is more
effective in suppressing local in-plane disorder. In this respect, LaO$_{1-x}$F$_x$BiS$_3$ exhibits quite high in-plane CP because of nearly 100% occupancy of Se at the in-plane Ch1 site. For Nd(O,F)BiS$_3$, the in-plane CP is lower than that for LaO$_{1-x}$F$_x$BiS$_3$[26] whereas it is higher than other RE(O,F)BiS$_2$ with RE = La, Ce, and Pr and hence shows bulk superconductivity. Therefore, it is possible that the presence of weak local structural disorder suppresses the NSC states in BiCh$_2$-based systems. Hence, we need further examples of BiCh$_2$-based superconductors showing NSC to clarify the aforementioned scenario.

This study focused on the CeOBiS$_2$–S$_x$Se$_{1-x}$ system. Bulk superconductivity was observed in polycrystalline samples with $x = 0.4$ and 0.6 of CeOBiS$_2$–S$_x$Se$_{1-x}$, and its $T_c$ was $\approx 3$ K.[24] The crystal structure is composed of alternate stacks of a CeO blocking layer and a BiCh$_2$ conducting layer. Notably, external elemental substitution is not needed to generate electron carriers in BiCh$_2$ layers because carriers are self-doped via the mixed valence states of Ce.[24,25] This situation is clearly different from the cases of other RE(O,F)Bi$_2$Se$_3$ systems, in which elemental substitutions are required for electron doping.[9,10] In addition, the Se concentration in the Bi-Ch1 conducting plane is significantly lower than that in LaO$_{1-x}$F$_x$BiS$_3$. Hence, there are clear compositional differences in both blocking and conducting layers between LaO$_{1-x}$F$_x$BiS$_3$ ($x = 0.1$ and 0.5), NdO$_{1-x}$F$_x$Bi$_2$S$_3$, and CeOBiS$_2$–S$_x$Se$_{1-x}$. In this study, we investigated the in-plane anisotropy of MR in the superconducting states of CeOBiS$_2$–S$_x$Se$_{1-x}$ ($x = 0.3$) and observed NSC features.

### 2. Results

As shown in the inset of Figure 1a, plate-like crystals were obtained. To confirm the c-axis direction of the obtained sample, powder XRD was performed on the CeOBiS$_1.7$Se$_{0.3}$ single crystals; the plates of the crystal were loaded on a sample holder. As shown in Figure 1a, only 00l peaks were observed, which confirms that the ab plane is well developed. The average ratio of the constituent elements (except for O) was estimated to be Ce:Bi:S:Se = 1:1.00(1):1.74(2):0.30(1), where the Ce value was fixed to 1 for normalization. Considering the error in the energy-dispersive X-ray (EDX) spectroscopy analysis, we concluded that the chemical composition of the single crystals is close to the nominal composition, and hence the sample was called CeOBiS$_{1.7}$Se$_{0.3}$.

Single-crystal X-ray structural analysis was performed on a CeOBiS$_{1.7}$Se$_{0.3}$ crystal. Details of the analysis conditions and the refined structural parameters are summarized in Table 1 and 2.

Because the Se ions selectively occupy the in-plane Ch1 site,[26] the Se occupation ratio for the Ch2 site was fixed as 0. The ratios of S and Se are consistent with the EDX analysis results. The CeOBiS$_{1.7}$Se$_{0.3}$ single crystal crystallizes in the tetragonal space group $P4/mnm$ and has lattice parameters of $a = 4.0327(9)$ Å and $c = 13.603(4)$ Å, which agrees with a report on the polycrystalline samples.[24] As demonstrated by Sogabe et al.[27] the bond valence sum (BVS) for Ce site was calculated using the following parameters: $b_0 = 0.37$ Å, $R_0 = 2.15$ Å for Ce–O bond, 2.62 Å for Ce–S bond, and 2.74 Å for Ce–Se bond. The bond distances between Ce and the nine coordinating anions were determined by single-crystal X-ray structural analysis. The site occupancies at the chalcogen site were included in the calculation. The estimated valence of Ce is 3.25, which is consistent with the value obtained for the polycrystal and indicates that Ce has a mixed valence state.[24] Hence, the phase is a self-doped system with mixed-valence Ce.

Figure 2 shows the temperature dependence of the electrical resistivity for a CeOBiS$_{1.7}$Se$_{0.3}$ single crystal measured with a
current along the: a) \(ab\) plane (\(\rho_{ab}\)) and b) \(c\) axis (\(\rho_c\)). The onset temperature (\(T_c^{\text{onset}}\)) and zero-resistivity temperature (\(T_c^{\text{zero}}\)) were determined to be 3.6 and 3.3 K, respectively. The \(T_c^{\text{zero}}\) values are slightly higher than those observed for polycrystalline samples of CeOBiS\(_{1.7}\)Se\(_{0.3}\).[24] In the normal states, \(\rho_c\) is clearly higher than \(\rho_{ab}\), which is due to the structure composed of stacking of electrically conductive BiCh\(_2\) layers and insulating layers along the \(c\) axis. Similar behavior has been observed in various layered superconductors, such as Cu oxide Bi\(_2\)Sr\(_2\)CuO\(_y\).[28] Fe pnictide BaFe\(_2\)As\(_2\).[29] and BiCh\(_2\)-based compounds.[30]

**Figure 3** shows the temperature dependence of the magnetization after zero-field cooling (ZFC) and field cooling (FC) with an applied field of 10 Oe parallel to the \(c\) axis for the CeOBiS\(_{1.7}\)Se\(_{0.3}\) single crystal. A large diamagnetic signal corresponding to superconductivity was observed, indicating that the observed superconducting states are bulk in nature, as reported in polycrystalline samples.[24] \(T_c\) was estimated to be 3.3 K, which is consistent with the zero resistivity states in the \(\rho-T\) data.

**Figure 4a** shows a schematic image of the rotation angles for the MR measurements. Figure 4b shows the \(\theta\) dependence of \(\rho_{ab}\) at \(T=2.1\) K and various magnetic fields ranging from 0.3 to 2.0 T. Anisotropy was clearly observed, which is a typical trend for layered superconductors. Figure 4c shows the \(\theta\) dependence of \(\rho_{ab}\) at \(B=1.0\) T, \(T=2.1\) K, and \(\phi=0^\circ\). The minimum \(\rho_{ab}\) was observed at \(\theta=98^\circ\), in which the magnetic field was applied parallel to the \(ab\) plane. Therefore, we defined that the magnetic field is parallel to the \(ab\) plane when \(\theta=98^\circ\).

**Figure 5** shows the temperature dependence of \(\rho_{ab}\) under various magnetic fields of: a) \(B//ab\) and b) \(B//c\). \(T_c\) decreases with increasing magnetic field in both directions. The suppression of \(T_c\) under a magnetic field parallel to the \(c\) axis is more significant than that under a magnetic field parallel to the \(ab\) plane. A \(B_c-T\) phase diagram is shown in Figure 5c, in which the temperature at which the resistivity becomes 90% of the normal-state value under various applied magnetic fields. We calculated \(B_c(0)\) for \(B//ab\) and \(B//c\) using the conventional one-band Werthamer–Helfand–Hohenberg (WHH) model for type-II superconductors in a dirty limit[31] which gives \(B_c(0)=-0.693T_c(dB_c/dT)_{T=T_c}\). The \(B_c(0)\) values for \(B//ab\) and \(B//c\) were estimated to be \(B_c(0)//ab=3.3\) T and \(B_c(0)//c=0.46\) T, respectively. The anisotropic parameter for \(B_c(0)\), \(\gamma=B_c(0)//ab/B_c(0)//c\), is determined to be 7.3. This value is lower than that of other BiCh\(_2\)-based superconductors.[30] Because the anisotropy parameter depends on its fluorine concentration in the case of RE(O,F)BiS\(_2\) with RE = La, Ce, Pr, and Nd[30,32] the lack of fluorine may affect the \(\gamma\) in BiCh\(_2\)-based systems and may result in relatively small \(\gamma\) for CeOBiS\(_{1.7}\)Se\(_{0.3}\).

**Figure 6a** shows the \(\phi\) dependences of \(\rho_{ab}\) at \(B=0.5\) T and at various temperatures ranging from 2.4 to 8.0 K. Zero resistivity was observed under the conditions of \(B=0.5\) T, \(T=2.7\) K, and \(\phi=90^\circ\) and 270°. In Figure 5a, zero resistivity was also observed under the conditions of \(B=0.5\) T and \(T=2.7\) K. Therefore, the superconducting characteristics of the samples used for the measurements shown in Figure 5 and 6 are comparable. Below 3.0 K, the \(\phi\) dependence of \(\rho_{ab}\) shows twofold-symmetric in-plane anisotropy. This trend is not simply expected from tetragonal structural symmetry (fourfold symmetry) as introduced in the introduction part. The breaking of the fourfold symmetry in the superconducting states is the trend of NSC, which has been observed in LaO\(_{1-x}\)F\(_x\)BiSe\(_2,[31,32]\). Above 4.0 K, \(\rho_{ab}\) is independent of \(\phi\), which suggests that the observed twofold symmetry of MR appears in superconducting states only. To investigate the reproducibility and the effect of the magnitude
Figure 4. a) Schematic image of the rotation angles. b) $\theta$ dependence of $\rho_{ab}$ plane resistivity ($\rho_{ab}$) for a CeOBiS$_{1.7}$Se$_{0.3}$ single crystal at $T = 2.1$ K and various magnetic fields ranging 0.30–2.0 T. c) $\theta$ dependence of $\rho_{ab}$ for a CeOBiS$_{1.7}$Se$_{0.3}$ single crystal at $B = 1.0$ T, $T = 2.1$ K.

Figure 5. a,b) Temperature dependences of the $\rho_{ab}$ plane resistivity ($\rho_{ab}$) for a CeOBiS$_{1.7}$Se$_{0.3}$ single crystal under various magnetic fields along $B$/$ab$ (a) and $B$/$c$ (b). c) Temperature dependence of upper critical field $B_{c2}(T)$ for a CeOBiS$_{1.7}$Se$_{0.3}$ single crystal. The dashed curves indicate the WHH-model fitting.
of magnetic fields on the observed twofold symmetric in-plane anisotropy, we performed in-plane anisotropy measurements under various magnetic fields in a different sample as well.

Figure 6b shows the dependence of $\rho_{ab}$ for a CeOBiS$_{1.7}$Se$_{0.3}$ single crystal at $T = 2.1$ K and at various magnetic fields ranging 0.50–2.0 T. In this experimental setup, uncertainty of the in-plane angle (about 10°) is expected. Therefore, the error bars of the angular $\phi$ are added.

We found that the directions of the nematic behavior (minimum resistivity) between experimental data in Figure 6a,b are slightly different. We cannot explain the origins of the phenomena, but we assume that the difference may be affected by anisotropic pinning direction of nematicity or the presence of superconducting domains. To clarify the origins, further experiments with different probes are needed.

3. Discussion

As there are no theoretical studies regarding the explanation or prediction of the emergence of twofold symmetric in-plane anisotropy of the superconducting states in BiCh$_2$-based compounds, the origin of the observed twofold symmetry in the superconducting states has not been revealed. Herein, we briefly describe the possible origins of the observed twofold symmetry of the in-plane anisotropy of MR in the superconducting states of CeOBiS$_{1.7}$Se$_{0.3}$.

The first possible scenario is the lowering of structural symmetry at low temperatures. The room-temperature structure of LaOBiS$_2$ is monoclinic ($P2_1/m$), and that for CeOBiS$_2$ is tetragonal ($P4/nmm$).[34] This fact could be explained by the difference in interlayer mismatching in REOBiS$_2$. Because the composition of CeOBiS$_{1.7}$Se$_{0.3}$ is close to CeOBiS$_2$, the high stability of the tetragonal structure is expected for CeOBiS$_{1.7}$Se$_{0.3}$. In addition, it was reported that LaOBiSSe undergoes a structural transition from tetragonal (high-$T$ phase: $P4/nmm$) to monoclinic (low-$T$ phase: $P2_1/m$) at 300–400 K.[13,35] However, this transition is rapidly suppressed by 3% F substitution in LaO$_{0.97}$F$_{0.03}$BiSSe.[13] The trend in which electron carrier doping suppresses the structural transition and stabilizes the tetragonal structure is commonly observed in a REOBiCh$_2$-type structure,[10] and a theoretical calculation also suggested the stability of the tetragonal structure after electron doping.[36] The expected amount of electron carriers doped in CeOBiS$_{1.7}$Se$_{0.3}$ is 0.25 per Bi because of the Ce valence of 3.25. Having considered the amount of doped electrons in the present phase and structural trend of the REOBiCh$_2$-type compounds, we consider that the low-temperature structure is tetragonal. In addition, we did not observe an anomaly which indicates a structural transition in $\rho-T$ and $M-T$ measurements. However, we cannot exclude lowering of structural symmetry with the data shown in this article. This issue should be studied further using more sophisticated tools such as low-temperature high-resolution XRD.

Figure 6. a) Dependences of the $ab$ plane resistivity ($\rho_{ab}$) for a CeOBiS$_{1.7}$Se$_{0.3}$ single crystal at $B = 0.5$ T and various temperatures in the range 2.4–8.0 K. b) Dependences of the $ab$ plane resistivity ($\rho_{ab}$) for a CeOBiS$_{1.7}$Se$_{0.3}$ single crystal at $T = 2.1$ K and at various magnetic fields ranging 0.50–2.0 T. In this experimental setup, uncertainty of the in-plane angle (about 10°) is expected. Therefore, the error bars of the angular $\phi$ are added. c) Schematic image of the terminal configuration and the definition of angular $\phi$ and the crystal plane.
Another possibility is that the measuring current itself caused the twofold symmetry of the in-plane MR. Because the current one-dimensionally flows in the ab plane, it naturally breaks the symmetry under rotating fields in the ab plane. When the magnetic field is perpendicular to the current, the Lorentz force may cause flux lines to detach from the pinning centers, which leads to a finite resistance. Although this effect has been observed for MgB$_2$, the observed twofold anisotropy is small for a current density of 30 A cm$^{-2}$ [37]. As shown by Pan et al. [16] with a careful setup of the experiments, $\rho_{ab}$ can be used for investigating the in-plane anisotropy of superconducting states. In our experiments, the current densities were less than 0.7 A cm$^{-2}$, and the magnitude of the magnetic field was small (0.5 T in Figure 6a). In NdO$_1$-$xF_5$Bi$_2$ systems, twofold anisotropy due to the flux flow is observed under high magnetic fields [18]. However, we confirmed that a fourfold-symmetric behavior, which was observed in $\rho_{c}$ for NdO$_{0.7}$Fe$_{0.3}$Bi$_2$ [14], could be reproduced in the setup of $\rho_{ab}$ with a low current density and low magnetic fields. Furthermore, the minimum $\rho_{ab}$ of twofold symmetry is shown at different $\phi$ in Figure 6a,b; the minimum $\rho_{ab}$ is observed at $\phi \approx 90^\circ$ and $270^\circ$ in Figure 6a, and at $\phi \approx 135^\circ$ and $315^\circ$ in Figure 6b. This indicates that there is sample-to-sample variation in the direction where the nematicity aligns: along the (100) or (110) directions. In addition, pinning direction may affect the direction as described earlier. The selectivity of the direction in the nematicity alignment was reported in $\text{A}_2\text{Bi}_2\text{Se}_3$ [5,10]. Therefore, we can rule out the effect of flux flow.

In addition, there is possibility that the small component of magnetic field along the c-axis direction results in twofold in-plane anisotropy. Twofold symmetry behavior could be observed due to misorientation of the crystal, which means the case that in-plane field is not applied along the ab plane. However, in that case, superconducting states are rapidly suppressed and the systematic evolution of the twofold symmetric behavior as in Figure 6 could not be observed, which was experimentally confirmed with our sample. Therefore, we consider that our sample setup was almost ideal.

Herein, we briefly mention related works. It was theoretically suggested that a BiCh$_2$-based compound could be a weak topological superconductor. [40] Furthermore, hidden spin-polarized states by a local Rashba–Dresselhaus effect were proposed on the basis of the broken local inversion symmetry in each BiCh$_2$ bilayer. [41] The local Rashba–Dresselhaus spin polarization was experimentally observed in LaO$_{0.55}$Fe$_{1.45}$Bi$_2$ by high-resolution spin- and angle-resolved photoemission spectroscopy [42]. If the Rashba–Dresselhaus spin-orbit coupling affects the superconducting states of BiCh$_2$-based superconductors, spin-singlet and spin-triplet states can be mixed in its superconducting states. These possible exotic superconducting states are related to the emergence of twofold symmetric in-plane anisotropy of superconducting states of CeOBiS$_{1.7}$Se$_{0.3}$ and La(O,F)BiSe.

4. Conclusion

We synthesized CeOBiS$_{1.7}$Se$_{0.3}$ single crystals using a flux method. Single-crystal X-ray structural analysis revealed that the crystal has a tetragonal structure with a space group of P4/nmm. BVS calculations showed that the valence of Ce ions was 3.25, indicating a mixed-valence state of Ce. Bulk superconductivity with a transition temperature of 3.3 K was confirmed through electrical resistivity and magnetization measurements. The estimation of the anisotropy parameter $\gamma_{Bc2}$ revealed that the $\gamma$ for CeOBiS$_{1.7}$Se$_{0.3}$ is lower than that observed for RE(O,F) Bi$_2$ crystals. In the in-plane anisotropy measurements, a twofold symmetric in-plane anisotropy of MR in the superconducting states was observed in CeOBiS$_{1.7}$Se$_{0.3}$.

5. Experimental Section

CeOBiS$_{2-x}$Se$_x$ single crystals were grown using a high-temperature flux method in an evacuated quartz tube. Polycrystalline powders of CeOBiSe$_{2-x}$Se$_x$ were prepared by the solid-state reaction method as described by Kiyama et al. [24]. The mixture of the polycrystalline powders of CeOBiS$_{2-x}$Se$_x$ ($x=0.5$ g) and CsCl flux ($=3.8$ g) with a molar ratio of 1:20 was mixed and then sealed in an evacuated quartz tube. The tube was heated at 950 °C for 15 h and slowly cooled to 650 °C at a rate of $-2.0$ °C · h$^{-1}$. After furnace cooling to room temperature, the quartz tube was opened under an air atmosphere, and the product was filtered and washed with pure water.

The obtained samples were characterized by XRD with Cu-Kα radiation on a MiniFlex-600 (Rigaku) by the $\theta$–2$\theta$ method. The single crystals were analyzed by scanning electron microscopy (SEM), and their chemical composition was investigated by EDX spectroscopy. Single-crystal X-ray structural analysis was carried out at room temperature on a XtaLAB (Rigaku). The structural parameters were refined with the tetragonal (P4/mnm) structural model using the refinement program SHELXL [43]. A crystal structure image was depicted using VESTA software [44].

Electrical resistivity was measured using an in-plane four-terminal configuration. The terminals were fabricated using Au wire (25 μm) and Ag paste. In-plane anisotropy was investigated on a PPMS with a horizontal rotator (Quantum Design) and a sample holder designated for in-plane anisotropy measurements. Note that different crystals were used for out-of-plane anisotropy measurements and in-plane anisotropy measurements because of different sample-stage setups. The temperature dependence of the magnetic susceptibility was measured by a superconducting interface device (SQUID) magnetometer with an applied field of 10 Oe after ZFC and FC on an MPMS3 (Quantum Design).

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Research data are not shared.

Keywords

BiCh$_2$-based superconductors, magnetoresistance, nematic superconductivity, single crystal structure analyses
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