Degradation of topological surface state by nonmagnetic S doping in \( \text{Sr}_x\text{Bi}_2\text{Se}_3 \)

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Research on possible topological superconductivity has grown rapidly over the past several years, from fundamental studies to the development of next generation technologies. Recently, it has been reported that the \( \text{Sr}_x\text{Bi}_2\text{Se}_3 \) exhibits superconductivity with topological surface state, making this compound a promising candidate for investigating possible topological superconductivity. However, whether or not the topological surface state is robust against impurities is not clear in this system. Here we report a detailed investigation on the lattice structure, electronic and magnetic properties, as well as the topological superconducting properties of \( \text{Sr}_x\text{Bi}_2\text{Se}_3-y\text{S}_y \) samples. It is found that the superconducting transition temperature keeps nearly unchanged in all samples, despite of a gradual decrease of the superconducting shielding volume fraction with increasing S doping content. Meanwhile, the Shubnikov-de Hass oscillation results of the \( \text{Sr}_x\text{Bi}_2\text{Se}_3-y\text{S}_y \) samples reveal that the topological surface states are destroyed in S doped samples, suggesting the topological character is degraded by nonmagnetic dopants.

The topological quantum matter states have become one of the hottest research topics in condensed matter physics and materials science communities\(^1\)–\(^13\). The topological insulating state, the Dirac semimetal state, as well as the Weyl semimetal state have been theoretically proposed and experimentally proven in a variety of bulk materials in recent years\(^4\)–\(^10\). Besides the above mentioned topological quantum states, it has been proposed that a novel topological superconducting state may emerge at the boundary between a superconductor and a topological insulator\(^3\). The topological superconducting state is featured with a full pairing gap in the bulk and gapless surface states at the surfaces. The topological superconductor is believed to be an ideal platform for searching of Majorana Fermion, a long-sought yet elusive quasiparticle which has been extensively investigated in high-energy physics for many years.

The searching of topological superconducting state in a real material has been proven to be a big challenge. In the past decade, there are tremendous efforts aiming to realize the topological superconducting state\(^14\)–\(^17\). In particular, the discovery of superconductivity in Cu-intercalated \( \text{Bi}_2\text{Se}_3 \) topological insulator has attracted much attention, because large-size \( \text{Cu}_x\text{Bi}_2\text{Se}_3 \) superconducting single crystals can be grown. A lot of theoretical study and experimental work have been performed on this compound in order to realize possible topological superconductivity in bulk samples\(^18\)–\(^21\). However, whether or not the \( \text{Cu}_x\text{Bi}_2\text{Se}_3 \) is a topological superconductor is still controversial. For example, the point-contact spectroscopy measurements have clearly shown the presence of zero-bias conductance peaks from the Majorana bound states at the surface edges\(^19\). On the contrary, the scanning tunneling spectroscopy measurements reveal a fully-gapped feature in the density of states and there is no in-gap state, possibly suggesting that the superconducting state in the \( \text{Cu}_x\text{Bi}_2\text{Se}_3 \) samples is topologically trivial\(^21\). Thus it is of particular importance to investigate the properties of possible topological superconducting state in alternative compounds. Recently, it has been reported that by intercalation of alkaline earth element Sr into the \( \text{Bi}_2\text{Se}_3 \) topological insulator, superconductivity with large superconducting volume fraction can be realized in \( \text{Sr}_x\text{Bi}_2\text{Se}_3 \) system\(^22\). It has also been experimentally proven that the Dirac point and the topological surface states are well-preserved in the \( \text{Sr}_x\text{Bi}_2\text{Se}_3 \) samples\(^23\)–\(^26\). Furthermore, angle-dependent resistivity measurements on \( \text{Sr}_x\text{Bi}_2\text{Se}_3 \) single crystals by different groups have revealed apparent two-fold anisotropy, indicating rotational symmetry breaking in this compound\(^27\)–\(^28\). The nodeless and two-fold symmetric superconducting gap is consistent with

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the prediction of topologically nontrivial superconductivity in \( \text{Sr}_x \text{Bi}_2\text{Se}_3 \). These facts suggest that the \( \text{Sr}_x \text{Bi}_2\text{Se}_3 \) compound could serve as an important material platform for the investigation of topological superconductivity.

In this work, we perform a systematic investigation on the crystal lattice, the transport behavior, as well as the topological superconducting properties of a series of \( \text{Sr}_x \text{Bi}_2\text{Se}_{3-y} \text{S}_y \) single crystal samples. It is found that the isovalent S doping at the Se site does not lead to any noticeable change in the charge carrier density, which is of particular importance in identification of the intrinsic effects of S doping in a topological compound. The nonmagnetic S doping results in a gradual decrease of the superconducting shielding volume fraction of the \( \text{Sr}_x \text{Bi}_2\text{Se}_{3-y} \text{S}_y \) compound, while the onset of the superconducting transition temperature keeps nearly unchanged in all samples. Furthermore, the analysis of the Shubnikov-de Hass oscillation data reveals that the nonmagnetic S-doping can also destroy the topological surface states of the samples. These results demonstrate that the topological feature of the \( \text{Sr}_x \text{Bi}_2\text{Se}_3 \) system is sensitive to nonmagnetic impurities.

In order to know to what extent the nonmagnetic S ions are incorporated into the \( \text{Sr}_x \text{Bi}_2\text{Se}_3 \) lattice, we perform energy dispersive x-ray spectrometry analysis on the S-doped \( \text{Sr}_x \text{Bi}_2\text{Se}_3 \) samples. The comparison between nominal and real compositions of the \( \text{Sr}_x \text{Bi}_2\text{Se}_{3-y} \text{S}_y \) samples is listed in Table 1. It can be seen from Table 1 that the actual Sr contents in all samples are quite close to 0.066, consistent with previous reports. It is also clear that the actual S doping content in each sample is very close to the nominal doping content, meaning that the nonmagnetic S ions can easily substitute the Se ions.

![Figure 1. Powder x-ray diffraction patterns of the \( \text{Sr}_x \text{Bi}_2\text{Se}_{3-y} \text{S}_y \) samples. The bottom one is a representative single crystal x-ray diffraction pattern of the \( y = 0.2 \) sample.](image)

The temperature dependence of in-plane resistivity of the \( \text{Sr}_x \text{Bi}_2\text{Se}_{3-y} \text{S}_y \) samples is given in Fig. 2. For the samples with S doping level \( y \leq 0.3 \), they exhibit metallic-like behavior at the normal state. The normal state

| Nominal composition | Real composition | \( a \) (Å) | \( c \) (Å) |
|--------------------|-----------------|-------------|-------------|
| \( \text{Sr}_{1.16} \text{Bi}_2\text{Se}_3 \) | \( \text{Sr}_{1.066} \text{Bi}_2\text{Se}_3 \) | 4.1428 | 28.563 |
| \( \text{Sr}_{1.16} \text{Bi}_2\text{Se}_{3.15} \text{S}_{0.05} \) | \( \text{Sr}_{1.066} \text{Bi}_2\text{Se}_{3.15} \text{S}_{0.05} \) | 4.1418 | 28.56 |
| \( \text{Sr}_{1.16} \text{Bi}_2\text{Se}_{3.1} \text{S}_{0.05} \) | \( \text{Sr}_{1.066} \text{Bi}_2\text{Se}_{3.1} \text{S}_{0.05} \) | 4.1409 | 28.555 |
| \( \text{Sr}_{1.16} \text{Bi}_2\text{Se}_{3.2} \text{S}_{0.05} \) | \( \text{Sr}_{1.066} \text{Bi}_2\text{Se}_{3.2} \text{S}_{0.05} \) | 4.1381 | 28.541 |
| \( \text{Sr}_{1.16} \text{Bi}_2\text{Se}_{3.3} \text{S}_{0.05} \) | \( \text{Sr}_{1.066} \text{Bi}_2\text{Se}_{3.3} \text{S}_{0.05} \) | 4.1363 | 28.528 |
| \( \text{Sr}_{1.16} \text{Bi}_2\text{Se}_{3.4} \text{S}_{0.05} \) | \( \text{Sr}_{1.066} \text{Bi}_2\text{Se}_{3.4} \text{S}_{0.05} \) | 4.1342 | 28.517 |

Table 1. The comparison between nominal and real compositions of the \( \text{Sr}_x \text{Bi}_2\text{Se}_{3-y} \text{S}_y \) samples as well as the lattice parameters of the samples.
resistivity gradually increases with increasing S doping content, meaning that the isovalent S dopants introduce some random disorder which can scatter the motion of the charge carriers. The inset of Fig. 2 shows an enlarged view of the resistivity near the superconducting transition temperature. It can be seen that the onset temperature of the superconducting transition ($T_{c, onset}$) is about 2.9 K for the undoped Sr$_{0.066}$Bi$_2$Se$_3$ sample, which is consistent with previous reports$^{23–28}$. The width of the superconducting transition is less than 0.3 K, suggesting the high-quality of the single crystal sample. With the introducing of S dopants, it is found that the superconducting transition becomes weakened. For the samples with $y \geq 0.2$, the resistivity does not reach zero even when the temperature is down to 1.8 K. Despite of the fact of the gradual depression of superconductivity with increasing S dopants, it is interesting to notice that the $T_{c, onset}$ values of the S doped samples are all close to 2.9 K. In other words, the $T_{c, onset}$ value keeps nearly unchanged with increasing S doping.

In order to know whether or not the S doping leads to any change in the charge carrier concentration of the Sr$_{x}$Bi$_2$Se$_3$ compound, we determine the charge carrier density of the Sr$_{x}$Bi$_2$Se$_3$ parent sample and the S-doped samples which is derived from the Hall coefficient measurements. The variation of charge carrier concentration ($n_y$) as the function of temperature for the $y = 0$, 0.2, and 0.4 samples is given in the inset of Fig. 2. For the undoped Sr$_{0.066}$Bi$_2$Se$_3$ sample, the $n_y$ value is $2.14 \times 10^{19}$ cm$^{-3}$ at room temperature, which is consistent with previous reports$^{23–28}$. We notice that the introduction of S in the Sr$_{x}$Bi$_2$Se$_3$–$y$S compound does not lead to any significant change in the charge carrier concentration. For example, the $n_y$ value in the $y = 0.4$ sample is $2.03 \times 10^{19}$ cm$^{-3}$ at room temperature, which is comparable with that of the undoped sample. Thus it can be concluded that the suppression of superconductivity by S doping is not originated from the change in charge carrier concentration.

In order to see clearly how the nonmagnetic S doping suppresses the superconductivity of the Sr$_{x}$Bi$_2$Se$_3$ system, we perform the measurements of the temperature dependence of magnetic susceptibility ($\chi$) of the Sr$_{0.066}$Bi$_2$Se$_3$–$y$S samples. The results are shown in Fig. 3. The onset superconducting transition temperature determined from the $\chi$ – $T$ curve of the $y = 0$ sample is about 2.85 K. And the shielding volume fraction increases sharply with decreasing temperature, indicating a very good diamagnetic behavior. It can be seen that the shielding superconducting volume fraction of the undoped Sr$_{0.066}$Bi$_2$Se$_3$ sample is about 90.3% at 1.8 K, which is consistent with previous reports$^{23–25}$. With increasing S doping, the shielding volume fraction gradually decreases, suggesting the suppression of superconductivity. For the $y = 0.4$ sample, the shielding volume fraction is zero, meaning a completely depression of superconductivity. It is worth noticing that despite of the gradual decrease of shielding fraction, the onset superconducting transition temperature determined from the $\chi$ – $T$ curve keeps nearly unchanged at about 2.85 K for all samples. This fact suggests that the S dopants destroys the superconductivity of the Sr$_{x}$Bi$_2$Se$_3$ system locally. In other words, the superconductivity is completely destroyed in a small area near the S dopants, while the areas far away from the S dopants remain intact. The locally depression of superconductivity has also been discovered in some doped cuprate and iron-based superconductors$^{29,30}$. This locally destroyed superconductivity probably means an unconventional superconductivity.

In order to know whether or not the nonmagnetic S dopants destroy the topological surface state of the Sr$_{x}$Bi$_2$Se$_3$ system, we perform the Shubnikov–de Hass (SdH) oscillation measurements on both the undoped and the S-doped samples. The analysis of the quantum oscillation data under magnetic field has recently been widely employed in the investigating of topological materials$^{31–34}$. Figure 4(a–c) show the magnetic field dependence of resistivity of the $y = 0$, $y = 0.2$, and $y = 0.4$ samples, respectively. The temperature is kept at 2 K. For the undoped Sr$_{0.066}$Bi$_2$Se$_3$ sample, it can be seen that the superconductivity is rapidly killed with increasing external magnetic field. When the applied magnetic field is larger than 0.36 T, the transition from the superconducting state into normal state is finished. The Sr$_{0.066}$Bi$_2$Se$_3$ sample exhibits positive magnetoresistance. A profound oscillation appears when the magnetic field is higher than 7 T, suggesting the high-quality of the single crystal sample and the high mobility of the charge carriers. We analyze the oscillation signal by subtracting the background and plot the

![Figure 2. Temperature dependence of in-plane resistance of the Sr$_{x}$Bi$_2$Se$_3$–$y$S samples. The lower inset shows an enlarged view near the superconducting transition region. The upper inset gives the variation of charge carrier concentration as the function of temperature and S doping.](image-url)
oscillation data in Fig. 4(d). It can be seen that the oscillation is periodic against 1/B. The simple pattern shown in Fig. 4(d) gives a single frequency of $F = 142.5$ T. For the S doped samples, as can be seen from Fig. 4(b,c), a clear SdH oscillation signal appears when the applied magnetic field is higher than 8 T. The oscillation frequencies in the $y = 0.2$ and $y = 0.4$ samples are $F = 150.2$ T and $F = 148.1$ T, respectively. It can be seen that the introduction of S hardly affects the oscillation frequency, meaning that the S dopants does not alter the Fermi surface topology of the Sr$_{x}$Bi$_2$Se$_3$ compound.

In a solid state material, any closed cyclotron orbit is quantized under an external magnetic field $B$, according to the Lifshitz-Onsager quantization rule

$$A_n \frac{\hbar}{eB} = 2\pi (n + \gamma)$$

where $A_n$ is the extremal cross-sectional area of the Fermi surface (FS) related to the Landau level (LL) $n$ and $\gamma$ represents an additional Berry's phase. The additional Berry's phase ($\gamma$) in a non-topological material is zero. For an ideal topological quantum material with surface states, the additional Berry's phase $\gamma$ should be close to 1/2. We analyzed the SdH oscillations of the Sr$_{x}$Bi$_2$Se$_3$ samples by plotting the Landau index versus the inverse of the magnetic field (1/B). The results are given in Fig. 5. For the undoped Sr$_{0.066}$Bi$_2$Se$_3$, all the data fall into a straight.
line and the linear extrapolation gives an intercept at γ = 0.53 (Fig. 5(a)). The existence of a nontrivial Berry's phase (γ = 0.53) suggests the existence of surface states in the SrBi₂Se₃S system, which is consistent with previous SdH oscillation and angle-resolved photoemission spectroscopy results. For the y = 0.2 sample, the obtained γ value is 0.21, which is neither close to 1/2 nor close to 0. Thus it is difficult to claim whether or not there are surface states in the y = 0.2 sample. As can be seen from Fig. 5(c), the obtained γ value is zero in the y = 0.4 sample, meaning the completely disappearance of surface states in this sample. These results suggest that the topological features in the SrBi₂Se₃ compound is gradually destroyed with nonmagnetic S doping. Thus the present study reveals that the topological character can be sensitive to nonmagnetic S dopants in SrBi₂Se₃ compound.

A recent study reveals that the incorporation of S in the middle layer of the quintuple-layer crystal lattice of Bi₁₀Sn₀₂Sb₀₉Te₂S decreases the absolute energy of the valence band and makes the Dirac point isolated in energy from the bulk states. In Bi₁₀Sn₀₂Sb₀₉Te₂S system, the topological surface state is robust against S incorporation. The fact of the degradation of surface state in S-doped SrBi₂Se₃ compound is interesting and needs further investigation. A systematic angle resolved photoemission spectroscopy study would probably reveal the physical reason.

In conclusion, we perform a systematical investigation on the superconductivity and topological surface states of the SrBi₂Se₃ compound with nonmagnetic S doping. The superconducting volume fraction is gradually decreased with increasing S doping concentration while the onset superconducting transition temperature keeps nearly unchanged, suggesting that the nonmagnetic S dopants destroy the superconductivity locally. Interestingly, we find that the nonmagnetic S dopants destroys the topological surface states of the SrₓBi₂Se₃ system.

**Methods**

Single crystal of a series of S-doped SrBi₂Se₃ were prepared using self-flux method as reported previously. Stoichiometric mixtures of Bi powder, Sr piece, Se powder and S powder were sealed in evacuated quartz tubes. In order to achieve a reliable conclusion, we keep the nominal Sr content at x = 0.16 in all S-doped samples. The tubes were heated at 850°C for 48 h, followed by a slow cooling to 600°C at a rate of 2.5°C/h. After that, the furnace was shut down and the samples were cooled down with furnace. The chemical compositions of the obtained
crystals were examined using energy dispersive x-ray spectrometry (EDX) analysis, which was performed using Oxford SWIFT3000 spectroscopy equipped with a Si detector. For each sample, about twenty different points were randomly selected in the EDX measurements and the average was defined as the real composition. The obtained crystals were characterized by powder x-ray diffraction (XRD) and x-ray single crystal diffraction with Cu Kα radiation at room temperature. The temperature dependence of resistivity and Hall coefficient were measured in a commercial Quantum Design physical property measurement system (PPMS-14 T) system. In order to get reliable results, we mount the y = 0, y = 0.2, and y = 0.4 samples in one sample holder in the Shubnikov-de Hass oscillation experiments. The [001] crystal axis have been carefully aligned to ensure that angle between the applied magnetic field and the [001] crystal axis is identical for the three samples during the experiments. Magnetic properties were performed using a superconducting quantum interference device magnetometer (SQUID). The applied magnetic for both zero-field cooling process and field-cooling process is 2 Oe.

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Author Contributions
H.H. conceived and designed the project; H.H., Q.W., P.J., and X.H. performed the experiments; H.H., M.T., J.G., and Q.W. analyzed the data; H.H. wrote the paper with contributions from other authors.
Additional Information

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