Pressure-tuned enhancement of superconductivity and change of ground state properties in $LaO_{0.5}F_{0.5}BiSe_2$ single crystals

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By using a hydrostatic pressure, we have successfully tuned the normal state property and superconductivity in $LaO_{0.5}F_{0.5}BiSe_2$ single crystals. It is found that, with the increase of pressure, the original superconducting phase with $T_c \sim 3.5$ K can be tuned to a state with lower T_c, and then a new superconducting phase with $T_c \sim 6.5$ K emerges. Accompanied by this crossover, the normal state is switched from that with a low temperature resistivity upturning to a metallic one. Accordingly, the normal state resistivity also shows a nonmonotonic change with the external pressure. Furthermore, by applying a magnetic field, the new superconducting state under pressure with $T_c \sim 6.5$ K is suppressed, and the recovered normal state reveals a resistivity-upturning feature again. These results illustrate a nontrivial relationship between the normal state property and superconductivity in this newly discovered superconducting system.

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I. INTRODUCTION

According to the theory of Bardeen-Cooper-Schrieffer (BCS), superconductivity is achieved by the quantum condensation of Cooper pairs which are formed by the electrons with opposite momentum near the Fermi surface. The ground state when superconductivity is removed is thus naturally believed to be metallic. In some unconventional superconductors, such as cuprate, iron pnictide/chalcogenide, heavy fermion, and organic superconductors, this may not be true. Recently, the BiS_2-based superconductors whose structures are similar to the cuprates [1] and iron pnictides [2] have been discovered and formed a new superconducting (SC) family. Many new SC compounds with the BiS_2 layer have been found, including $Bi_4O_4S_3$ [3–5], $RO_{1-x}F_xBiS_2$ ($R =$ La, Nd, Ce, Pr, and Yb) [6–10], $Sr_{1-x}La_xFBiS_2$ [11], and $La_{1-x}M_xOBiS_2$ ($M =$ Ti, Zr, Hf, and Th) [12], etc. Among these compounds, the high pressure synthesized $LaO_{0.5}F_{0.5}BiS_2$ was reported to have an optimal $T_c \sim 10.6$ K [6]. The basic band structure obtained by first principle calculations indicates the presence of strong Fermi surface nesting at the wave vector (π,π) [13–15] when the doping is close to $x = 0.5$ in, for example, $LaO_{1-x}F_xBiS_2$. Quite often the superconductivity is accompanied by a normal state with a clear semiconductinglike behavior with unknown reasons [6–12]. In addition, possible triplet paring and weak topological superconductivity were suggested based on renormalization-group numerical calculation [16], but this mechanism is still much debated. Moreover, the experiments on the $NdO_{0.5}F_{0.5}BiS_2$ single crystals also reveal interesting discoveries concerning the SC mechanisms in this new system [17–21].

Through adjusting the lattice parameters and intimately the electronic band structure, high pressure has served as a very effective method, which can tune both the SC and normal state of superconductors. In this newly found BiS_2 family, high pressure has been recognized as an important tool to enhance both the superconductivity volume and transition temperatures except for $Bi_4O_4S_3$ [22–28]. In particular, the SC transition temperature of $RO_{1-x}F_xBiS_2$ ($R =$ La, Ce, Nd, Pr) [22–24] and $Sr_{1-x}R_xFBiS_2$ ($R =$ La, Ce, Nd, Pr, Sm) [25,28] systems was enhanced tremendously by applying the hydrostatic pressure. Taking $LaO_{0.5}F_{0.5}BiS_2$ as an example, the T_c of the sample can be increased from about 2 K under ambient pressure to ∼10 K under 2 GPa [22]. And in the $Sr_{1-x}R_xFBiS_2$ ($R =$ Ce, Nd, Pr, Sm) system, the non-SC sample at ambient pressure can also be tuned to become a SC one with $T_c \sim 10$ K under a pressure of 2.5 GPa [28]. To understand the role of high pressure, x-ray diffraction measurements under pressures have been performed on $LaO_{0.5}F_{0.5}BiS_2$ system and suggest a structural phase transition from a tetragonal phase ($P4/nmm$) to a monoclinic phase ($P2_1/m$) under pressures [27]. Very recently, a new superconductor $LaO_{0.5}F_{0.5}BiSe_2$ with the same structure as $LaO_{0.5}F_{0.5}BiS_2$ was discovered with $T_c \sim 3.5$ K [29–31]. It was reported that the electronic structure and Fermi surface in these two compounds are quite similar [32]. Since the system now is selenium based, it is highly desired to do investigations on $BiSe_2$-based materials, better in the form of single crystals. Furthermore, we are curious to know how the high pressure influences the superconductivity and the ground state property in the $BiSe_2$-based superconductors.

Here, we report the successful growth of the $LaO_{0.5}F_{0.5}BiSe_2$ single crystals, and a systematic high-pressure study on two single crystals (hereafter named sample 1 and sample 2). By increasing pressure, the ground state is switched from a state with a low temperature resistivity upturning to a metallic one; simultaneously the original SC $T_c \sim 3.5$ K (at ambient pressure) initially drops down to about 2 K and finally increases with pressure. As the pressure reaches about 1.2 ± 0.2 GPa, a new SC phase with higher T_c appears. At about 2.17 GPa, the T_c of the new SC phase reaches about 6.5 K. Accompanied with the change of SC transition temperatures, the normal state resistivity (ρ_n) decreases first and then increases with pressure. This nonmonotonic pressure dependence of T_c and the normal state resistivity in the present $BiSe_2$-based system are very different from the BiS_2-based family. Furthermore, the SC phase with

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higher T_c can be suppressed by applying a magnetic field, and a weak resistivity-upturning feature in the normal state emerges again when superconductivity is suppressed. All these results show the competing feature between superconductivity and the underlying ground state associated with the low temperature resistivity-upturning behavior.

II. EXPERIMENTAL DETAILS

The $LaO_{0.5}F_{0.5}BiSe_2$ single crystals were grown by using flux method [18]. Powders of La_2O_3, LaF_3, Bi_2Se_3, Se, and La scraps (all 99.9% purity) were mixed in stoichiometry as the formula of $LaO_{0.5}F_{0.5}BiSe_2$. The mixed powder was ground together with CsCl/KCl powder (molar ratio CsCl : KCl : $LaO_{0.5}F_{0.5}BiSe_2 = 12 : 8 : 1$) and sealed in an evacuated quartz tube. Then it was heated up to 800 °C for 50 h followed by cooling down at a rate of 3 °C/h to 600 °C. Single crystals with lateral sizes of about 1 mm were obtained by washing with water. X-ray diffraction (XRD) measurements were performed on a Bruker D8 Advanced diffractometer with the Cu-K_α radiation. dc magnetization measurements were carried out with a SQUID-VSM-7T (Quantum Design).

Measurements of resistivity under pressure were performed up to ~ 2.3 GPa on PPMS-16T (Quantum Design) by using HPC-33 Piston type pressure cell with the Quantum Design dc resistivity and ac transport options. For the resistive measurements, silver leads with a diameter of 50 μm were glued to the $LaO_{0.5}F_{0.5}BiSe_2$ single crystal with dimensions of about $1.3 \times 0.7 \times 0.05$ mm^3 in a standard four-probe method by using silver epoxy, and the crystal was immersed in pressure transmitting medium (Daphne 7373) in a Teflon cap with a diameter of 4 mm. Hydrostatic pressures were generated by a BeCu/NiCrAl clamped piston-cylinder cell. When the pressure cell is installed to the PPMS for measurements, the applied field is approximately parallel to *ab* plane of the single crystal. The pressure upon the sample was calibrated with the shift in T_c of a Sn sample with high purity by measuring the temperature dependence of resistivity.

III. RESULTS AND DISCUSSION

In Fig. 1(a) we present the x-ray diffraction (XRD) pattern for the $LaO_{0.5}F_{0.5}BiSe_2$ single crystal. It's clear that only (00*l*) reflections can be observed yielding a *c*-axis lattice constant $c = 14.05 \pm 0.03$ Å. The inset of Fig. 1(a) shows the Laue diffraction pattern of the $LaO_{0.5}F_{0.5}BiSe_2$ single crystal. Bright and symmetric spots can be clearly observed, indicating a good crystallinity. Energy dispersive x-ray spectrum (EDS) measurements were performed at an accelerating voltage of 20 kV and working distance of 10 mm by a scanning electron microscope (Hitachi Co., Ltd.). One set of the EDS result on a nominally formulated $LaO_{0.5}F_{0.5}BiSe_2$ single crystal is shown in Fig. 1(b), and the composition of the single crystal can be roughly expressed as $LaO_yF_{0.48}Bi_{0.95}Se_{1.89}$ through the EDS analysis. The atomic ratio is close to the nominal composition except for oxygen which cannot be obtained accurately by the EDS measurement. To clarify the precise ratio of O/F, future measurement on electron probe microanalysis (EPMA) may be needed.

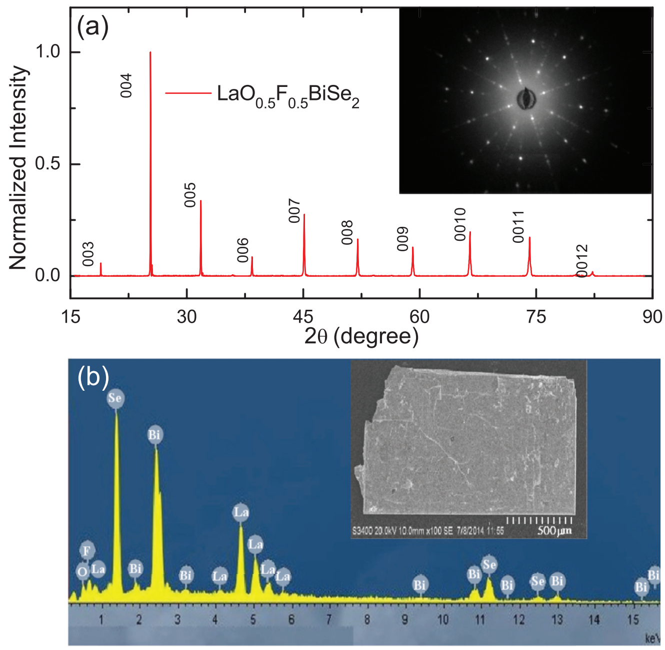

FIG. 1. (Color online) (a) X-ray diffraction pattern for a $LaO_{0.5}F_{0.5}BiSe_2$ (nominal composition) single crystal. The inset shows the back Laue x-ray diffraction pattern of a $LaO_{0.5}F_{0.5}BiSe_2$ single crystal. (b) Energy dispersive x-ray microanalysis spectrum taken on a $LaO_{0.5}F_{0.5}BiSe_2$ single crystal. The inset shows the SEM photograph of the crystal with typical dimensions of about $1.5 \times 0.75 \times 0.04$ mm^3.

The temperature dependence of resistivity for the $LaO_{0.5}F_{0.5}BiSe_2$ single crystal (sample 1) at various pressures with temperature ranging from 2 K to 300 K is illustrated in Fig. 2(a). The inset of Fig. 2(a) shows the temperature dependent magnetic susceptibility at ambient pressure under a magnetic field of 10 Oe, and a sharp SC transition is observed at about 3.5 K. An estimate on the Meissner screening volume through the magnetic susceptibility measured in the zero-field-cooled (ZFC) mode reveals a high superconducting volume. For sample 1, we did not manage to measure the sample at a pressure higher than 2.04 GPa. As shown in Fig. 2(a), at ambient pressure the normal state resistivity shows an upturning behavior in the low temperature region. Similar behavior was observed in sample 2. The reason for this low temperature resistivity upturning remains puzzling, which might be induced by the disorder effect, especially when the normal state is a bad metal. Interestingly, this feature can be suppressed under a small pressure and changes to a metallic one at about 0.54 GPa. With further increase of pressure, the metallic behavior maintains until the maximum pressure. This semiconductinglike to metallic transition with pressure has been noticed in $Sr_{1-x}R_xFBiS_2$ (R = La, Ce, Nd, Pr, Sm) systems [25,28]. In the case of $Sr_{0.5}La_{0.5}FBiS_2$ polycrystalline sample, the semiconductor-metal transition was considered as coming from the change of F-Sr/La-F bond angle along with interatomic distances [25]. Interestingly, the semiconductor-metal transition under pressure for $CeO_{0.5}F_{0.5}BiS_2$ system has been proposed according to the first-principle calculations [33], but the transition was not observed in previous

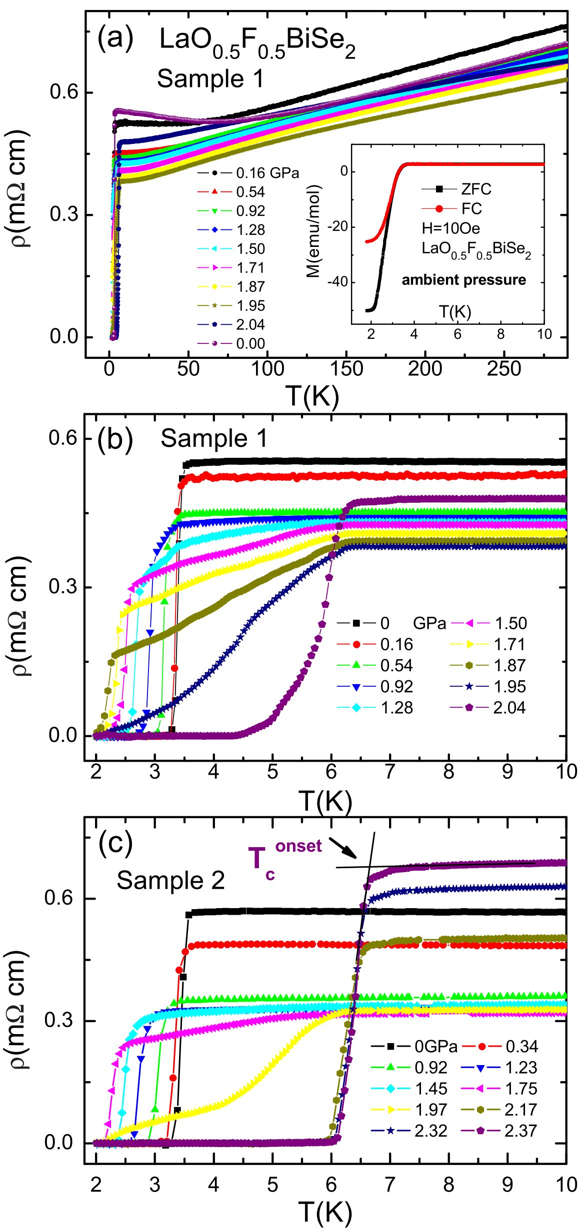

FIG. 2. (Color online) (a) Temperature dependence of resistivity for sample 1 at various pressures in the temperature range 2 K to 300 K. The inset shows the magnetic susceptibility of a $LaO_{0.5}F_{0.5}BiSe_2$ single crystal in an applied field of 10 Oe ($\parallel c$ axis) under the ambient pressure. Both the magnetic susceptibility measured in zero-field-cooled (ZFC) and field-cooled (FC) modes are shown. (b), (c) Enlarged views of the resistive transition in the temperature range 2 K to 10 K at various pressures for sample 1 and sample 2, respectively. The superconducting transitions are rather sharp at ambient and high pressures.

reports of experiment [23,26]. In particular, for $LaO_{0.5}F_{0.5}BiS_2$ polycrystalline samples, the normal state resistivity decreases monotonically with increasing pressure, but it still exhibits semiconductinglike behavior under a very high pressure (18 GPa) [27].

In Figs. 2(b) and 2(c), we present enlarged views of SC transitions at low temperatures under various pressures for sample 1 and sample 2, respectively. Both samples exhibit very similar behavior. Here the T_c was determined by the crossing point of a linear extrapolated line of the normal state and the transition curve, as illustrated in Fig. 2(c). As one can see, the variation of both the SC transition temperature and normal state resistivity upon the external pressure are nonmonotonic. The original $T_c \sim 3.5$ K (at ambient pressure) gradually drops down with increasing pressure and becomes below 2 K at about 1.95 GPa. At the same time, a high T_c phase gradually emerges starting from about 1.2 ± 0.2 GPa and enhances continuously with increasing pressure. It seems that the high T_c phase with $T_c = 6.5$ K coexists with the low T_c phase in the range from 1.2 ± 0.2 GPa to about 1.95 GPa. With further increase of pressure, zero resistance corresponding to the high T_c phase appears above 2 K and the SC transition becomes sharper at higher pressures. A similar behavior under pressure has been observed in some strongly correlated electronic systems, such as heavy fermion [34], organic systems [35], and iron chalcogenides [36]. In previous high pressure studies on BiS_2-based superconductors, the coexistence of two transient phases is not observed clearly. This indicates the distinction between our present $BiSe_2$-based superconductors and the earlier studied BiS_2-based systems. For sample 1 we did not manage to measure the resistivity beyond 2.04 GPa. Two samples are from the same batch. One can see that the resistive transitions below 2.04 GPa are quite similar to each other.

It is worth noting that the normal state resistivity presents a nonmonotonic dependence on applied pressure. As shown in Figs. 2(b) and 2(c), the normal state resistivity just above the SC transition temperature gradually decreases with increasing pressure until about 1.95 GPa. Surprisingly, above the threshold pressure, the normal state resistivity begins to increase remarkably with increasing pressure. It is clear that this qualitative behavior is closely related to the pressure-dependent T_c, as we will address below.

Figures 3(a) and 3(b) present the phase diagram of T_c^{onset} versus pressure and pressure-dependent resistivity (8 K), respectively. Here, the pressure for the absence of the second transition (about 1.95 GPa) is defined as the critical one (P_c). In the high temperature region, $\rho(T)$ shows a parallel shift with pressure and a turning point at the critical pressure P_c is also observed as illustrated for data measured at 8 K in Fig. 3(b). This implies that the transition associated with the turning point at the critical pressure P_c of resistivity is an intrinsic effect. Figures 3(a) and 3(b) clearly reveal two distinct SC phases: the low T_c SC phase below P_c and the high T_c SC phase above P_c. In the low T_c SC phase region, both T_c and the normal state resistivity are suppressed with increasing pressure. On the contrary, in the high T_c SC phase, T_c is slightly enhanced and the normal state resistivity increases remarkably with raising pressure. In $LaO_{0.5}F_{0.5}BiS_2$ polycrystalline samples, a structural phase transition from a tetragonal phase ($P4/nmm$) to a monoclinic phase ($P2_1/m$) has been suggested by high-pressure x-ray diffraction measurements. And a high T_c value of 10.7 K in the high-pressure regime appears in the monoclinic structure [27]. Therefore, considering the very weak change of the transition temperature of the high T_c phase, and taking a comparison between $LaO_{0.5}F_{0.5}BiS_2$ and $LaO_{0.5}F_{0.5}BiSe_2$, we believe that there are two distinct SC phases in our $LaO_{0.5}F_{0.5}BiSe_2$ single crystals in the intermediate pressure

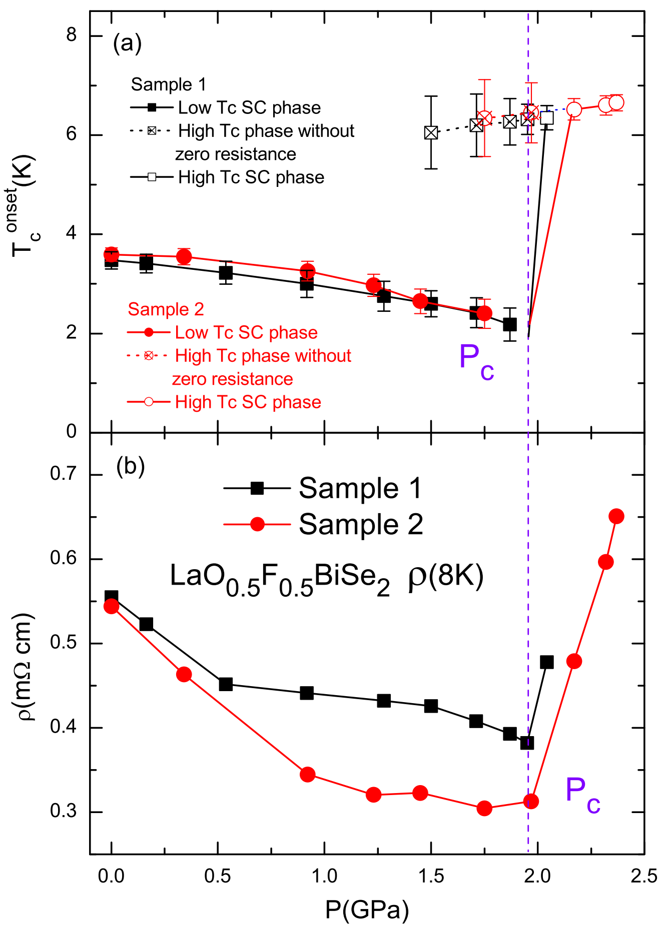

FIG. 3. (Color online) (a) Phase diagram of $T_c^{\rm onset}$ versus pressure for the two $LaO_{0.5}F_{0.5}BiSe_2$ single crystals investigated here. The dark and red symbols represent the $T_c^{\rm onset}$ of sample 1 and sample 2, respectively. The filled symbols stand for the low T_c phase; the open and crossed symbols stand for the high T_c phase with and without zero resistance, respectively. (b) Resistivity at 8 K in the normal state at various pressures for sample 1 (filled squares) and sample 2 (filled circles).

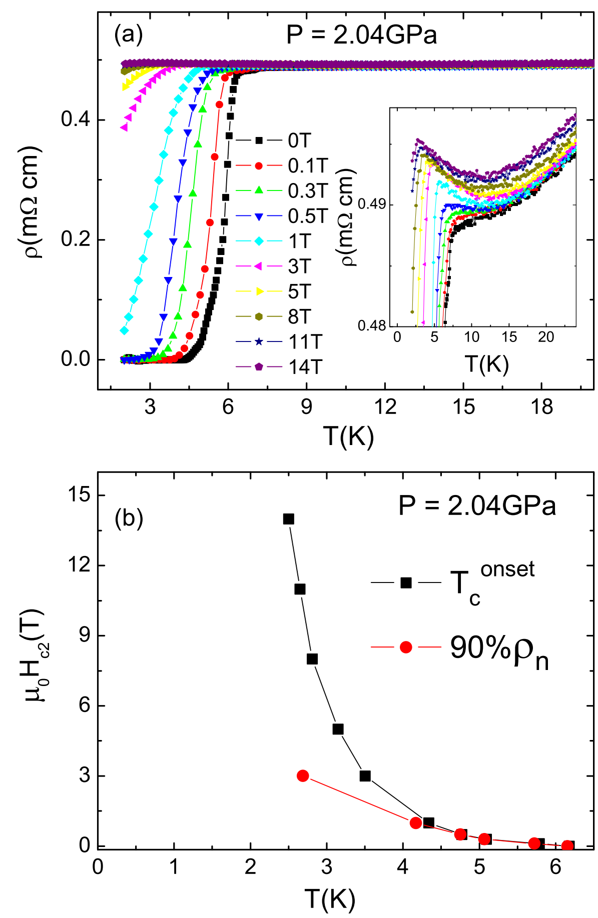

FIG. 4. (Color online) (a) Temperature dependence of resistivity for sample 1 under a pressure of 2.04 GPa at various magnetic fields. The inset shows the enlarged view of a weak resistivity-upturning feature in the normal state under high magnetic fields. (b) Upper critical field determined by $T_c^{\rm onset}$ and 90% normal state resistivity ρ_n.

region (1.2 ± 0.2 to $\sim$1.95 GPa). At a high pressure, all the phase becomes superconductive with $T_c \sim 6.5$ K. The transition from the low T_c phase to the high T_c one could be induced by the structural transition, which needs to be further checked.

In Fig. 4(a), we present the temperature dependent resistivity under magnetic field up to 14 T at 2.04 GPa ($T_c \sim$ 6.3 K). The upper critical field H_{c2} versus T is displayed in Fig. 4(b). We use different criterions of 90%ρ_n and $T_c^{\rm onset}$ to determine the $H_{c2}(T)$. One can see that the $H_{c2}(T)$ exhibits an extremely concave curved shape, leading to a very small slope $-dH_{c2}(T)/dT$ near T_c. The residual resistivity ratio RRR $= \rho(300\text{ K})/\rho(8\text{ K})$ is less than 2, indicating a strong impurity scattering. Usually the Werthamer-Helfand-Hohenberg (WHH) [37] formula $H_{c2}(0) = -0.69T_c dH_{c2}(T)/dT|_{T_c}$ can be used to determine the upper critical field in the dirty limit. However, as shown in Fig. 4(b), $H_{c2}(T)$ exhibits an extremely curved feature. Similar effect was observed in the iron based superconductor $LaFeAsO_{0.89}F_{0.11}$ [38], which was explained as a consequence of multiband effect. Therefore, here the Werthamer-Helfand-Hohenberg (WHH) formula may not be appropriate for estimating the upper critical field $H_{c2}(0)$ at zero temperature. The inset of Fig. 4(a) shows the enlarged view of superconducting transitions as in the main panel. As shown in the inset, the SC is very robust and keeps presence above 2 K when the field is up to 14 T. This could be induced by the fact that the applied field was approximately parallel to *ab* plane of the single crystal in the pressure cell during the measurement, and a large anisotropy has been discovered in $LaO_{0.5}F_{0.5}BiSe_2$ single crystals [30]. An interesting phenomenon is that a weak resistivity-upturning behavior reemerges when the superconductivity is suppressed under a high magnetic field. A similar behavior was observed in $NdO_{0.5}F_{0.5}BiS_2$ single crystals [18]. This phenomenon may be related to the resistivity-upturning behavior of the sample at an ambient pressure, although it seems that the low T_c phase does not show up here. The semiconductinglike ground states for either the low T_c phase at an ambient pressure, or the one with high T_c superconductivity under a high pressure but suppressed with a high magnetic field, may be caused by the same reason: both point to the competition of superconductivity with a tendency which underlines the low temperature resistivity-upturning behavior.

IV. CONCLUSION

In summary, we have successfully tuned the normal state property and superconductivity in $LaO_{0.5}F_{0.5}BiSe_2$ single crystals through a hydrostatic pressure. The ground state is switched from that with a low temperature resistivity upturning to a metallic one with increasing pressure. Moreover, the original SC phase with $T_c \sim 3.5$ K can be tuned to a new SC state with $T_c \sim 6.5$ K. In the low T_c SC phase region, both T_c and the normal state resistivity are suppressed with increasing pressure. On the contrary, in the high T_c SC phase, superconductivity is enhanced and the normal state resistivity increases remarkably with increasing pressure. Moreover, the weak resistivity-upturning behavior reemerges when the superconductivity under a high pressure is suppressed by a high magnetic field. These results illustrate a nontrivial relationship between the normal state property and superconductivity. Further theoretical and detailed structure investigations are highly desired to clarify the new high T_c SC phase under a high pressure.

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