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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(Dated: July 23, 2014)

By using a hydrostatic pressure, we have successfully tuned the ground state 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 ground state is switched from a semiconducting state 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 normal state reveals a weak semiconducting feature again. These results illustrate a non-trivial relationship between the normal state property and superconductivity in this newly discovered superconducting system.

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/phosphide, heavy fermion and organic superconductors, this may not be true. Recently, the recently discovered BiS$_2$ family, high pressure has been recognized as an important tool to enhance both the superconductivity volume and transition temperatures except for Bi$_4$O$_4$S$_3$ [22, 23]. In particular, the SC transition temperature of RE$_{1-x}$F$_x$BiS$_2$ (RE = La, Ce, Nd, Pr) [22] and Sr$_{1-x}$RE$_x$FBiS$_2$ (RE = La, Ce, Nd, Pr, Sm) 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 $\sim$ 10 K under 2 GPa [22]. In the Sr$_{1-x}$RE$_x$FBiS$_2$ (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 ($P21/m$) under pressures [27]. Very recently, a new superconductor LaO$_{0.5}$F$_{0.5}$BiSe$_2$ with the same structure as the 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 form of single crystals. Furthermore it is 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 as Sample-1 and Sample-2). By increasing pressure, the ground state is switched from a semiconducting state 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...
FIG. 1: (Color online) (a) X-ray diffraction pattern for a LaO$_{0.5}$F$_{0.5}$BiSe$_2$ 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 1.4 × 0.7 × 0.04 mm$^2$.

The LaO$_{0.5}$F$_{0.5}$BiSe$_2$ single crystals were grown by using flux method[18]. Powders of La$_2$O$_3$, LaF$_3$, Bi$_2$Se$_3$, 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 grounded 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 hours followed by cooling down at a rate of 3°C/hour 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$_\alpha$ radiation. DC magnetization measurements were carried out with a SQUID-VSM-7T (Quantum Design).

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 measured in zero-field-cooled (ZFC) and field-cooled (FC) modes. (b) and (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.

Measurements of resistivity under pressure were performed up to $\sim$ 2.3GPa on PPMS-16T (Quantum Design) by using HPC-33 Piston type pressure cell with the Quantum Design DC resistivity and AC transport options. The LaO$_{0.5}$F$_{0.5}$BiSe$_2$ single crystal with the standard four-probe method was immersed in pressure transmitting medium (Daphene 7373) in a Teflon cap.
Hydrostatic pressures were generated by a BeCu/NiCrAl clamped piston-cylinder cell. The pressure upon the sample was determined by measuring the pressure-dependent $T_c$ of a Sn sample with high purity.

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 (00l) 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 20kV and working distance of 10 millimeters by a scanning electron microscope (Hitachi Co.,Ltd.). One set of the EDS result on 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$_{0.48}$Bi$_{0.95}$Se$_{1.89}$. The atomic ratio is close to the nominal composition except for oxygen which can not be obtained accurately by the EDS measurement.

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 were not managed 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 a semiconducting behavior. This semiconducting behavior can be suppressed under a small pressure and turns to be a metallic one at about 0.54 GPa. With further increase of pressure, the metallic behavior maintains until the maximum pressure. This semiconducting to metallic transition with pressure has been noticed in Sr$_{1-x}$RE$_x$FBiS$_2$ (R= La, Ce, Nd, Pr, Sm) systems$^{22,28}$. 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 inter-atomic distances$^{22}$. 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$^{22}$, but the transition was not observed in previous reports of experiment$^{22,26}$. 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 semiconducting behavior under a very high pressure (18 GPa)$^{27}$.

In Fig. 2(b) and (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. As one can see, the variation of both the SC transition temperature and normal state resistivity upon the external pressure are non-monotonic. 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 $1.2 \pm 0.2$ 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 $1.2 \pm 0.2$ 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 $T_c$ monotonically increases with the pressure without showing the coexistence of two transient phases. This indicates the distinction between our present BiSe$_2$-based superconductors and the earlier studied BiS$_2$-based systems. For Sample-1 we were not managed 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 non-monotonic dependence on applied pressure. As shown in Fig. 2(b) and (c), the normal state resistivity just above the SC transition temperature gradually decreases with increasing pressure till 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 addressed below.

Fig. 3(a) and 3(b) present the phase diagram of $T_{c}^{onset}$ versus pressure and pressure-dependent resistivity (8K), respectively. Here, the pressure for the absence of the second transition (about 1.95 GPa) is defined as the critical one ($P_c$). Fig. 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 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 regime ($1.2 \pm 0.2$ to $\sim 1.95$ GPa). At a high pressure, all the phase becomes super-
FIG. 3: (Color online) (a) Phase diagram of $T_{c \text{onset}}$ versus pressure for the two LaO$_{0.5}$F$_{0.5}$BiSe$_2$ single crystals investigated here. The dark and red symbols represent the $T_{c \text{onset}}$ 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 $T_c$ phase with and without zero resistance, respectively.

In Fig. 4(a), we present the temperature dependent resistivity under magnetic field up to 14 T at 2.04 GPa ($T_c$ $\sim$ 6.5 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 ($T_c$ $\sim$ 6.3K). The upper critical field $H_{c2}$ versus $T_c$ is displayed in Fig. 4(b). We use different criterions of 90% $\rho_n$ and $T_{c \text{onset}}$ (determined using the crossing point shown in Fig. 2(c)) to determine the $H_{c2}$. The upper critical field at zero temperature can be estimated by using the Werthamer-Helfand-Hohenberg (WHH) formula:

$$H_{c2} = -0.69T_c \frac{dH_{c2}}{dT_c},$$

and the estimated $H_{c2}(0)$ is about 35 T for $T_{c \text{onset}}$. 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. That 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 semiconducting behavior re-emerges when the superconductivity is suppressed under a high magnetic field. A similar behavior was observed in NdO$_{0.5}$F$_{0.5}$BiSe$_2$ single crystals [18]. This phenomenon may be related to the semiconducting behavior of the sample at an ambient pressure, although it seems that the low $T_c$ phase does not show up here. The semiconducting ground states for either the low $T_c$ phase at 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 semiconducting behavior.

In summary, we have successfully tuned the ground state and superconductivity in LaO$_{0.5}$F$_{0.5}$BiSe$_2$ single crystals through a hydrostatic pressure. The ground
state is switched from a semiconducting state 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, a weak semiconducting behavior emerges when the superconductivity under a high pressure is suppressed under magnetic field. These results illustrate a non-trivial 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.

**ACKNOWLEDGMENTS**

We appreciate the kind help and discussions with Xiaojia Chen. We thank Xiang Ma and Dong Sheng for the assistance in SEM/EDS measurements. This work was supported by NSF of China, the Ministry of Science and Technology of China (973 projects: 2011CBA00102, 2012CB821403, 2010CB923002) and PAPD.

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