Enhancement of Ising Superconductivity in $Pb_{1-x}Bi_x$ Ultrathin Films

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Ising superconductivity has garnered much attention in recent years due to its extremely high in-plane upper critical field (B_{c2}). Here, we fabricated 14 multilayer $Pb_{1-x}Bi_x$ ($0\% \leq x \leq 40\%$) thin films on Si (111)-7 × 7 reconstructed surface by molecular beam epitaxy. Large B_{c2} beyond the Pauli limit is observed in all the $Pb_{1-x}Bi_x$ films, indicating that they may exhibit characteristics of Ising superconductivity. Moreover, the introduction of Bi doping can significantly enhance and effectively tune the in-plane B_{c2} of $Pb_{1-x}Bi_x$ films, which will help us better understand Ising superconductivity and provide a new platform for the development of tunable Ising superconductors.

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1. Introduction. Crystalline two-dimensional (2D) superconductors have attracted much research attention because of their exotic quantum phenomena,[1] such as quantum oscillations of the superconductivity of Pb films,[2–5] quantum metallic state in ion-gated ZrNCl and $NbSe_2$ bilayer superconductors,[6,7] and quantum Griffiths singularity of superconductor–metal transition in Ga thin films,[8] etc. Specifically, a large in-plane B_{c2} is observed in some highly crystalline 2D superconductors, which is attributed to Ising superconductivity. Generally, in 2D superconductivity, the in-plane B_{c2} is mainly determined by the spin pairing breaking, which is called the Pauli limit B_p [$B_p = 1.86\,T_c(0)$].[9,10] Under an external magnetic field, the spin alignment of Cooper pairs in a superconductor undergoes reorientation, thereby elevating the system energy. Hence, it is difficult for conventional superconductors to exceed the Pauli paramagnetic limit or Clogston-Chandrasekhar limit.[10,11] However, in 2015, Lu *et al.* first reported that an in-plane B_{c2} of up to 32.5 T was observed in MoS_2 monolayer on ionic-gated MoS_2 transistors, far surpassing B_p,[12] which indicated the emergence of so-called Ising superconductivity. Subsequent investigations have revealed that some 2D materials, mainly transition metal dichalcogenides (TMDs),[13–19] show in-plane B_{c2} that exceeds the Pauli limit by many times. These outstanding outcomes can be attributed to various mechanisms, including spin-orbit coupling (SOC),[12–19] spin-orbit scattering (SOS),[20–24] and Rashba-type spin-orbit interactions (SOIs).[25] Nevertheless, it is worth noting that SOS and Rashba-type SOIs tend to only have a minor impact on the in-plane B_{c2},[12,25] and conversely, SOC is considered to play a more important role in elevating in-plane B_{c2} far beyond the Pauli limit.[12–19] In their study, the origin of Ising superconductivity is ascribed to a robust SOC effect caused by the broken central inversion symmetry of the system, which generates a Zeeman-like effective field, polarizing the Cooper pair to an out-of-plane direction and thus leading to a substantially upgraded in-plane B_{c2}. However, in 2020, Falson *et al.* presented a notable observation of Ising superconductivity regarding the in-plane B_{c2} surpassing the B_p within a few-layer stanene possessing both central inversion symmetry and multiple degenerate orbitals.[26] Subsequently, Liu *et al.* reported a significant discovery in ultrathin $PdTe_2$ films: despite central inversion symmetry, the in-plane B_{c2} surpasses the B_p by over seven times, attributed to the film's multiple rotational symmetry.[27] Therefore, this type of superconductor, which possesses central inversion symmetry yet remains superconducting under a high in-plane magnetic field far beyond the Pauli limit, is designated as the type-Ⅱ

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Ising superconductor.

To date, despite extensive research on TMD Ising superconductivity, few studies have been conducted on other systems. What's more, there have been no reports on the intrinsic tuning of the Ising superconductivity of a 2D superconductor. The non-centrosymmetric structure of a metal Pb film possesses a strong SOC throughout the Brillouin zone,[28] and recently, it has been reported that ultrathin pure Pb films with a 4–6 multilayer (ML) thickness exhibit the characteristics of Ising superconductors.[29] It has also been reported that the SOC of pure Pb thin film systems can be enhanced with the Bi dopant, but the addition of Bi can lead to a reduction in the metallic state of the systems.[30] In addition, the doping of Bi can also tune the superconductivity of the material.[31–33] In particular, in low bismuth-doped systems, significant enhancement of superconductivity has been observed,[31,33] while superconductivity has been suppressed in highly bismuth-doped systems.[33] By considering these factors, there exists an optimal doping concentration that can maximize the in-plane B_{c2} of the system. From the above, the ability to enhance the Ising superconductivity by intrinsically tuning the doping ratio reveals the mechanism of changes in B_{c2}.

Here, we have successfully grown 14 ML $Pb_{1-x}Bi_x$ ($0\% \leq x \leq 40\%$) thin films with varying ratios x on Si (111) via molecular beam epitaxy (MBE). We then characterized their morphology by scanning tunneling microscope (STM) and measured their transport properties. Our experimental results reveal that Bi doping results in the enhancement of T_c and the out-of-plane B_{c2}. Moreover, under in-plane magnetic fields, all the samples exhibited in-plane B_{c2} that exceeded their corresponding Pauli limit. More importantly, our findings also demonstrate that B_{c2} of the system can be tuned and substantially enhanced by varying the Bi doping concentration, suggesting immense potential for tuning Ising superconductivity via doping.

2. Methods. All experimental procedures were conducted in an ultrahigh-vacuum (UHV) low-temperature STM system (home-built with an RHK R9 controller) with a base pressure of approximately 3×10^{-10} Torr. The system is also equipped with an MBE chamber for *in situ* growth of thin films. An n-type Si (111) wafer with a resistivity of 0.001 Ω·cm and a miscut angle of 0.1° was used as the substrate. The clean Si (111)-7 × 7 reconstruction was prepared by a standard flash process, heating to 1150 ℃ with direct current and slowly cooling to room temperature. Subsequently, various 14 ML $Pb_{1-x}Bi_x$ films were deposited on Si (111) reconstruction surface using an *in situ* MBE technique. By varying the deposition temperature of Bi and the co-deposition time, diverse ratios of 14 ML $Pb_{1-x}Bi_x$ films could be fabricated. Finally, a few nanometers of Ge were epitaxially grown on top of the $Pb_{1-x}Bi_x$ films as a capping layer. The growth rates of Pb, Bi, and Ge were calibrated by deposition on Si (111) substrates separately. STM and scanning tunneling spectroscopy measurements were conducted using electrochemically etched tungsten (W) tips.

For the *ex situ* transport measurements, all the $Pb_{1-x}Bi_x$ ($0\% \leq x \leq 40\%$) films were protected by Ge-capped layers. The resistance and magnetoresistance were measured using the standard four-probe method, and silver paint formed electrical contacts between electrodes and films. The steady high magnetic field measurements were implemented using the standard ac lock-in technique (13.7 Hz) with a He-3 fridge and a water-cooled magnet capable of reaching up to 28 T at the National High Magnetic Field Laboratory of China in Hefei.

3. Results and Discussion. Given its excellent stability and 2D characteristics, the 14 ML $Pb_{1-x}Bi_x$ film serves as an ideal system for our subsequent transport measurements. Thus, we selected it as the primary focus of our study. Figure 1(a) shows a typical STM image of a 14 ML $Pb_{0.6}Bi_{0.4}$ film grown on a Si (111) substrate by the *in situ* MBE method under UHV ($\sim 2 \times 10^{10}$ Torr) at 440 ℃ and 410 ℃ (effusion cell temperature) for Pb and Bi, respectively. The morphology of the atomically thin 2D crystalline film is notably flat and uniform, with less than 2% voids. To prevent oxidation of the thin film, it is necessary to deposit a capping layer on the film to protect it prior to the *ex situ* transport measurements. Based on the researches of Liu and Nam *et al.*,[29,34] Si and Ge have been identified as suitable candidates for use as protective layers due to their insulating properties at low temperatures, having no significant impact on the transport measurements of thin films. Therefore, we grew Ge on the $Pb_{1-x}Bi_x$ films by MBE as the protective layer [in Fig. 1(b)]. Based on a previous study conducted by Eom *et al.*,[3] the quantum well states (QWS) induced by the Pb film under the quantum size effect can vary as a function of the film thickness. Our study shows the QWS of the pure Pb films and the $Pb_{0.87}Bi_{0.13}$ films to determine their thickness as 14 ML, as shown in Figs. 1(c) and 1(d), respectively. It is evident that the QWS exhibited by the $Pb_{0.87}Bi_{0.13}$ film experiences a slight shift in comparison to the QWS observed in the pure Pb film. However, the observed trend remains consistent, by which it can be inferred that the QWS in the $Pb_{1-x}Bi_x$ system is primarily governed by Pb atoms at low Bi doping concentrations.

By convention, we define the temperature when the resistance drops to 50% of the normal state resistance (R_n) as the T_c of the superconducting transition. In Fig. 1(e), all samples show a superconducting transition from the normal state [a positive correlation was observed between the system R_n and the concentration of Bi in the samples, and more details are shown in Fig. S1 of the Supplementary Materials (SM)] to zero resistance (at zero field). Figure 1(f) displays the dependence of the T_c on the Bi concentration for the 14 ML $Pb_{1-x}Bi_x$ films ($0 \leq x \leq 40\%$), showing that T_c exhibits a monotonic rise trend with increasing Bi concentration, and the T_c of all the samples with Bi doping exceeds that of the bulk of pure Pb (7.23 K, shown by the red dashed line), which indicates the enhancement of superconductivity with Bi doping. We believe that the reason is that Pb has one fewer outermost

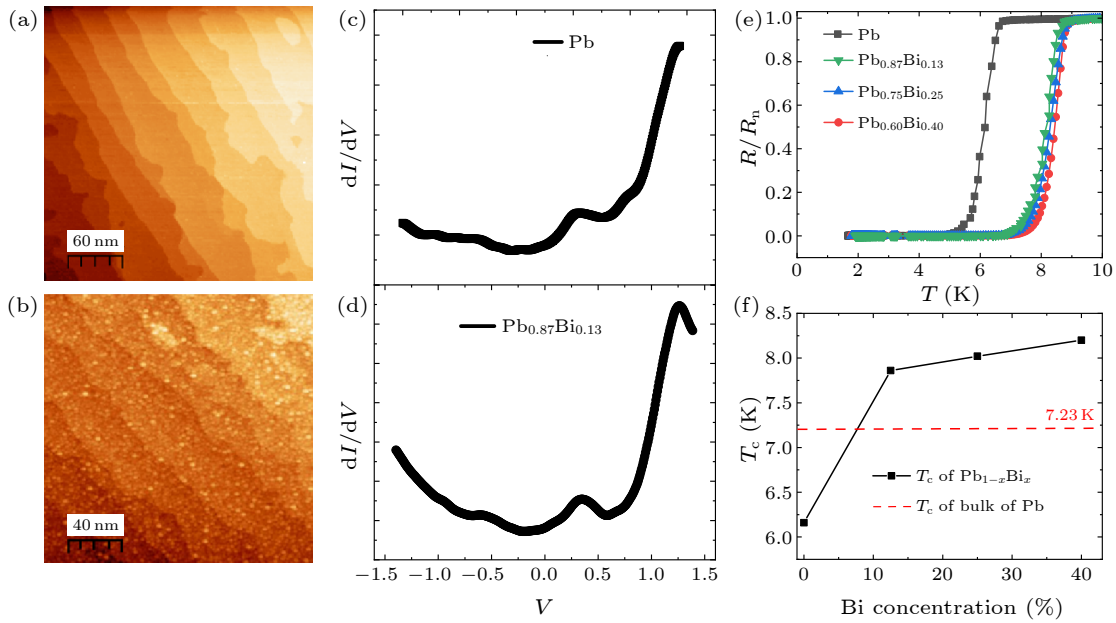

Fig. 1. (a) A typical STM image of a 14 ML $Pb_{0.6}Bi_{0.4}$ film (300 × 300 nm, sample bias 1.5 V, tunneling current 50 pA). (b) STM image of 14 ML $Pb_{1-x}Bi_x$ film after 3.6 nm Ge capping (200 × 200 nm, sample bias 2.0 V, tunneling current 50 pA). (c) and (d) dI/dV–V curves of 14 ML pure Pb film and $Pb_{0.87}Bi_{0.13}$ film, respectively. The QWS exhibited by the $Pb_{0.87}Bi_{0.13}$ film align closely with the QWS trend observed in the pure Pb film, with a slight shift, indicating that the QWS of the $Pb_{0.87}Bi_{0.13}$ film are predominantly influenced by Pb atoms. (e) Temperature dependence of R/R_n for 14 ML $Pb_{1-x}Bi_x$ ($0\% \leq x \leq 40\%$) film around the superconducting transition (at zero field). (f) The T_c as a function of samples with different Bi concentrations; the red dashed line is the T_c (7.23 K) of the bulk of pure Pb. When Bi concentration $x \leq 40\%$, T_c exhibits an increase with an increase in x.

electron than Bi, which indicates that the introduction of Bi is similar to n-type doping in semiconductors.[32] The so-called n-type doping offers extra electrons to enhance Fermi surface nesting along the (111) direction, thereby promoting superconductivity.[33,35] Given that the T_c of a monocrystal bulk Bi is situated at approximately 0.53 mK, signifying a notably weak superconductivity,[36] we speculate that the superconductivity of the system will be suppressed when Bi is likely to become the predominant element in the film under higher doping.[33] Our experimental results show that $Pb_{0.6}Bi_{0.4}$ has the largest T_c value in the 14 ML $Pb_{1-x}Bi_x$ system around $x = 40\%$, which is different from the 2 ML $Pb_{1-x}Bi_x$ system.[33] One possibility is that the 2 ML Pb system possesses only a single quantum channel capable of supporting the formation of Cooper pairs,[4] which suggests that the superconductivity of the 2 ML Pb system is more susceptible to inhibition by Bi. Here, we would like to emphasize that despite our observation of the highest T_c in the $Pb_{0.6}Bi_{0.4}$ thin film, it is difficult to accurately determine the Bi ratio of the 14 ML $Pb_{1-x}Bi_x$ films with the maximum value of T_c due to uncertainties arising from errors in the calibration of the growth rate.

$B_{c2}(T)$ is typically defined as the magnetic field strength at which a superconductor undergoes a transition from the superconducting state to the normal state. More precisely, for both out-of-plane and in-plane magnetic fields, it is determined at a specific temperature where the resistance of the superconductor reaches 50% of R_n. In the presence of an out-of-plane magnetic field, the superconducting state of a material is susceptible to disruption. It is evident from the results depicted in Fig. 2(a) that the out-of-plane B_{c2} of the 14 ML $Pb_{0.75}Bi_{0.25}$ thin film is observed to be markedly suppressed with the application of an increasing magnetic field at a certain temperature. Additionally, the out-of-plane B_{c2} of $Pb_{0.75}Bi_{0.25}$ exhibits a monotonic increase as the temperature decreases. For out-of-plane B_{c2} at 0 K, $B_{c2}(0)$ can be obtained by fitting the 2D Ginzburg–Landau (GL) theoretical formula of $B_{c2,\perp}(T) \propto 1 - T/T_c$ (out-of-plane B_{c2}),[17,18,37,38] and the Pauli limit for our samples can be calculated by the simplified formula $B_p = 1.86T_c(0)$,[12] where $T_c(0)$ is the zero-field superconducting transition temperature (in units of K). The fitted curve of $B_{c2,\perp}(T)$ vs T for 14 ML $Pb_{0.75}Bi_{0.25}$ at 0 K is shown in Fig. 2(b) (the fitting curves of all samples are shown in Fig. S3 of SM), and the fitting result shows that the out-of-plane B_{c2} at 0 K is around 2.25 T, which is notably lower than the corresponding Pauli limit for 14 ML $Pb_{0.75}Bi_{0.25}$ of 14.9 T. It is observed that the $B_{c2,\perp}(T)$ of all samples at 0 K exhibits a monotonically increasing trend with the increase of Bi concentration [Fig. 2(c)]. What is more, it is noteworthy that the $B_{c2,\perp}(0)$ for each sample is significantly lower than its corresponding Pauli limit (more details in Fig. S4 of SM), which indicates that the Zeeman-like orthogonal field is unable to maintain the pairing of Cooper pairs under large out-of-plane magnetic fields. According to BCS theory, the out-of-plane B_{c2} is expected to be

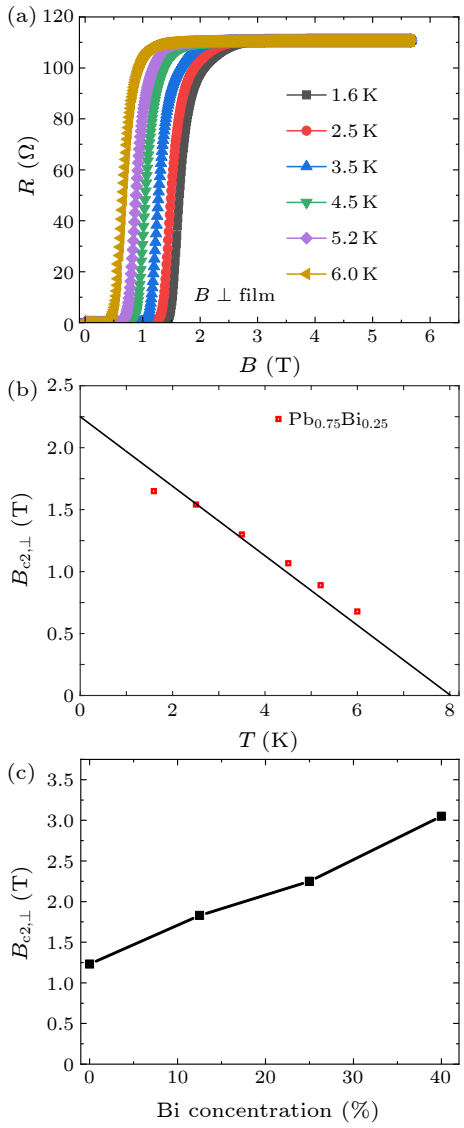

Fig. 2. Out-of-plane B_{c2} transport properties of 14 ML $Pb_{1-x}Bi_x$ ($0\% \leq x \leq 40\%$) film. (a) For B (out-of-plane) dependence of R of 14 ML $Pb_{0.75}Bi_{0.25}$ at different temperatures. (b) Fitting curve of $B_{c2,\perp}(T)$ vs T under $B_{c2,\perp}(T) \propto 1 - T/T_c$ fitting formula of 14 ML $Pb_{0.75}Bi_{0.25}$. (c) Different $B_{c2,\perp}(T)$ of different Bi concentrations for all the samples at 0 K.

proportional to the square of the T_c. However, our experimental results show that with Bi doping, the enhancement of the out-of-plane B_{c2} exceeds that expected from the squared increase of T_c. This significant deviation from the BCS expectation suggests the involvement of additional mechanisms in our system. We believe that the answer might lie in the fact that the source of the continuous augmentation in out-of-plane B_{c2} can be attributed to the Rashba-type SOIs, which are capable of locking the spin to the in-plane direction in a system of superconductors with central inversion symmetry breaking.[12,39] This effect has been shown to enhance the out-of-plane B_{c2} of the system, and it has been reported that the strength of Rashba-type SOIs in $Pb_{1-x}Bi_x$ thin films increases with increasing Bi content.[30] Moreover, although orbital effect generally suppresses the out-of-plane B_{c2} by generating screening currents under a perpendicular magnetic field, which break Cooper pairs and weaken superconductivity,[38] we still observe an overall increase in out-of-plane B_{c2}. This indicates that the combined influence of the conventional BCS mechanism and enhanced Rashba-type SOIs outweighs the suppression of orbital effect, resulting in a net increase in the out-of-plane B_{c2}.

In order to study the in-plane B_{c2}, a high magnetic field up to 28 T is applied to our samples. The representative in-plane magnetic field measurements are summarized in Fig. 3. Figures 3(a) and 3(b) present the R–B (in-plane) curves for the 14 ML Pb film and the 14 ML $Pb_{0.75}Bi_{0.25}$ film at varying temperatures, respectively. The results depicted in these figures indicate that the in-plane B_{c2} rises as the temperature decreases, which is consistent with the trend of the out-of-plane B_{c2}. According to the 2D GL theory, $B_{c2,\parallel}(T)$ can be effectively determined by fitting the expression $B_{c2,\parallel}(T) \propto (1 - T/T_c)^{1/2}$, enabling the extraction of the corresponding value (0 K).[17,18,37,38] In Fig. S6 of SM, the GL fitting curves for temperature versus in-plane B_{c2} are presented for all samples. The fitting results demonstrate that the $B_{c2,\parallel}$ of several films at 0 K exceed their corresponding Pauli limits. In particular, the $B_{c2,\parallel}$ of the $Pb_{0.75}Bi_{0.25}$ film at 0 K exceeds that of pure Pb by around 10 T in our fitting, indicative of a significant enhancement in the in-plane B_{c2} resulting from Bi doping of the pure Pb film. Figure 3(c) shows the $B_{c2,\parallel}$–T/T_c fitting curves for several $Pb_{1-x}Bi_x$ films that were doped with varying concentrations. We can observe that the in-plane B_{c2} of every $Pb_{1-x}Bi_x$ ($0\% < x \leq 40\%$) film surpasses the in-plane B_{c2} of the pure Pb film, which shows the significant potential for enhancing the in-plane B_{c2} of the system by changing the relative proportions of its constituent elements.

Figure 3(d) displays the variation of in-plane B_{c2} and B_p shown at 0 K in relation to the Bi doping concentration for all the samples, denoted by the black and red solid lines, respectively. Firstly, it is observed that the in-plane B_{c2} increases with Bi concentration. Interestingly, in our measurements, in-plane B_{c2} decreases with increasing Bi concentration at high doping. We then discuss the possible origin of this dome-like trend exhibited by in-plane B_{c2} and concentration x. For the first stage, one possibility that in-plane B_{c2} monotonically increases with x can be attributed to the SOC strength exhibited by Bi atoms exceeding that of Pb atoms, manifested as an elevation of in-plane B_{c2}. However, as previously indicated, the Rashba-type SOIs of the $Pb_{1-x}Bi_x$ system will experience enhancement with the introduction of Bi.[30] It is inferred that the Rashba-type SOIs induce a preferential alignment of the spin in the in-plane orientation rather than the out-of-plane direction, which introduces a competitive interaction with the influence of SOC. Based on the finding that Yi *et al.* reported that Rashba-type SOIs suppressed Ising superconductivity in Bi_2Se_3/monolayer $NbSe_2$ heterostructures,[40] we suppose that a similar mechanism is at play in our $Pb_{1-x}Bi_x$

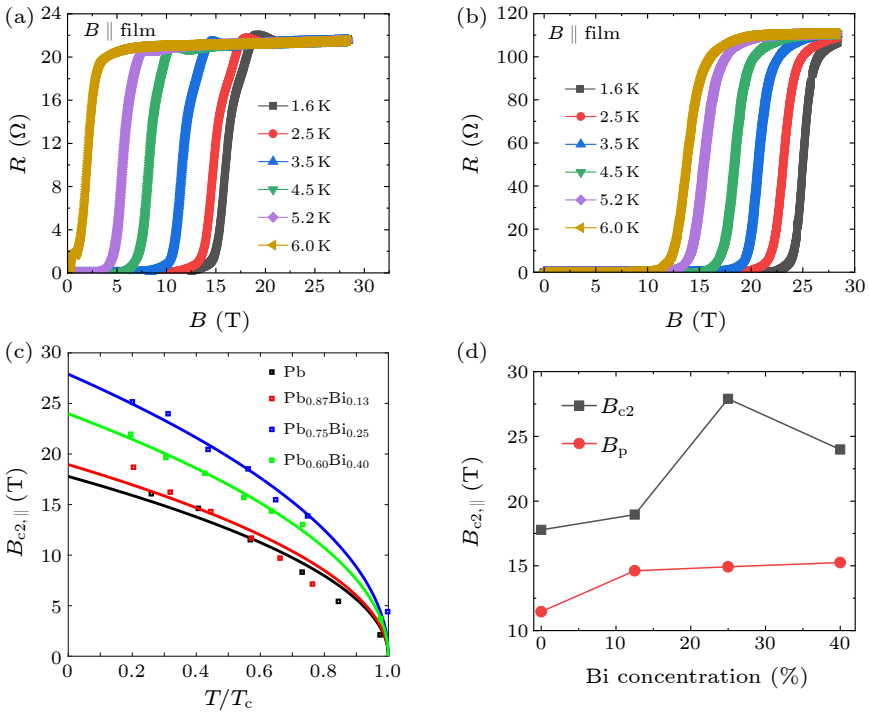

Fig. 3. In-plane B_{c2} transport properties of 14 ML $Pb_{1-x}Bi_x$ ($0\% \leq x \leq 40\%$) film. (a) and (b) For B (in-plane) dependence of R of 14 ML Pb film and $Pb_{0.75}Bi_{0.25}$ film at different temperatures, respectively. (c) The fitting curve of $B_{c2,\parallel}(T)$–T/T_c for 14 ML $Pb_{1-x}Bi_x$ ($0\% \leq x \leq 40\%$), and the squares represent measured data. Here, the $B_{c2,\parallel}(T) \propto (1 - T/T_c)^{1/2}$ fitting formula was used to calculate the B_{c2} at 0 K for different samples. (d) The variation of the in-plane B_{c2} (black curve) under the fitting calculation and B_p (red curve) with Bi concentration for different samples at 0 K.

alloy system. In our experimental observations, when x is of small magnitude, the influence of SOC is notably stronger than that of Rashba-type SOIs, leading to an enhancement in B_{c2}. We speculate that in the stage of decreasing in-plane B_{c2}, the influence of the Rashba-type SOIs might become dominant and suppress the possible existence of Ising superconductivity, resulting in a decline of in-plane B_{c2}. By comparing the results shown in Figs. 2(c) and 3(d), we find that the GL anisotropy factor, $\gamma = B_{c2,\parallel}/B_{c2,\perp} \gg 1$, confirms that the orbital effect is significantly suppressed under in-plane magnetic fields.[24] Therefore, we conclude that SOC and Rashba-type SOIs play dominant roles in determining the in-plane B_{c2}, rather than the orbital effect. In addition, a clear comparison is made between the in-plane B_{c2} at 0 K and the corresponding B_p for all samples with varying concentrations in Fig. 3(d). Notably, the in-plane B_{c2} values for all samples surpass their respective B_p values. Furthermore, it is observable that the introduction of Bi doping significantly enhances the in-plane B_{c2} of pure Pb films, which highlights the tunable effect of Bi concentration on Ising superconductivity. Our measurements at high magnetic fields up to 28 T and low temperatures down to 1.6 K provide more reliable evidence that Bi doping can enhance the in-plane B_{c2} of pure Pb films to a great extent, and we believe that $Pb_{1-x}Bi_x$ thin film is also a type of Ising superconductor.

Conclusions. In conclusion, we have systematically studied the transport properties of $Pb_{1-x}Bi_x$ thin films at low temperatures and high magnetic fields. Our findings demonstrate that the incorporation of Bi into Pb thin films can effectively tune the B_{c2} in the system, showing a significant improvement in the in-plane B_{c2}. Moreover, all the $Pb_{1-x}Bi_x$ ($0\% \leq x \leq 40\%$) samples in our study exceeded their respective Pauli limits for the in-plane B_{c2}. Our results suggest that Bi doping can be utilized as a means of enhancing the in-plane B_{c2} in these systems. Our findings will provide inspiration and an effective methodology for future investigations into the tuning of in-plane B_{c2} in Ising superconductors.

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