Observation of anomalous temperature dependence of the critical current in Pb/Sr_2RuO_4/Pb junctions

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We have studied the temperature dependence of the critical current I_c of bilayer Pb/Sr_2RuO_4/Pb junctions prepared by using a submicron-diameter filament as a shadow mask. Sr_2RuO_4, a layered perovskite isostructural with La_2CuO_4, has a superconducting transition temperature (T_c) lower than that of Pb. Below the T_c of Pb, the critical current I_c of the junction was found to increase initially with decreasing temperature. As the temperature was lowered to below the T_c of Sr_2RuO_4, however, a sharp drop in I_c was observed. This downturn in I_c suggests that superconductivity in Sr_2RuO_4 actually suppresses the Josephson coupling between the two Pb electrodes, which are conventional *s*-wave superconductors. The implications of this unexpected behavior will be explored, in particularly, in the context of the pairing symmetry in Sr_2RuO_4.
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Recently, it has been suggested[1] that Sr_2RuO_4, the only known Cu-free layered perovskite superconductor,[2] may have an odd-parity (*p*-wave) pairing state. Although Sr_2RuO_4 and high-T_c cuprate superconductor $(La,Sr)_2CuO_4$ share the same crystalline structure,[3] their properties are rather different.[4] In particular, the normal-state spin fluctuations in Sr_2RuO_4 are predominantly ferromagnetic[5,6] rather than antiferromagnetic as in the case of the cuprates. In fact, $SrRuO_3$, the three-dimensional analog to Sr_2RuO_4, is a ferromagnetic metal.[7] In addition, the enhancements of the effective mass and the Pauli spin susceptibility of Sr_2RuO_4 are comparable with those found in ^3He, known to form a *p*-wave pairing state. All these seem to favor an odd-parity pairing state in Sr_2RuO_4. Experimentally, superconductivity in Sr_2RuO_4 has been found to be extremely sensitive to the presence of nonmagnetic impurities in the way expected for an unconventional *p*-wave superconductor.[8] Results of specific heat,[9] NMR and NQR (Ref. 10) measurements have shown a large residual density of states below the T_c of Sr_2RuO_4. More recently, muon spin-relaxation (μSR) measurements on superconducting Sr_2RuO_4 have revealed the spontaneous appearance of an internal magnetic field, indicating the breaking of time-reversal symmetry in this material.[11] All these observations support the non-*s*-wave-pairing picture for Sr_2RuO_4. However, so far, no direct experimental evidence for such pairing has been obtained.

In order to obtain direct information on the pairing symmetry in Sr_2RuO_4, we have carried out experiments on bilayer superconductor-normal metal-superconductor ($SN'S$) junctions, where S is Pb ($T_c = T_{cs} = 7.2\,K$) and N' is Sr_2RuO_4 ($T_c = T_{cn} < T_{cs}$). Since none of the films grown so far show superconductivity,[12] the experiments one can perform to directly probe the pairing symmetry of Sr_2RuO_4 are limited. We have developed a technique to prepare $SN'S$ junctions on single crytalline Sr_2RuO_4 using a submicron-diameter filament as a shadow mask. A schematic of a junction is shown in Fig. 1(a). The idea is to study the influence of a superconducting Sr_2RuO_4 interlayer on the Josephson coupling between two *s*-wave superconductors (Pb). If Sr_2RuO_4 does have a non-*s*-wave pairing symmetry, the Josephson coupling strength, quantified by the magnitude of the critical current I_c, will be suppressed when Sr_2RuO_4 becomes superconducting at $T_c = T_{cn}$.

Single crystals of Sr_2RuO_4 were grown by a floating-zone method using an image furnace. To compensate the high evaporation rate of Ru during the crystal growth, a Ru-rich ceramic feed rod was used. When the Ru content or the speed of the crystal growth is particularly high, Ru lamellas will form in a single-crystalline Sr_2RuO_4 matrix. According to Ref. 13, these Ru lamellas have an essentially identical thickness ($\approx 1\ \mu m$), but varying width ($\geq 1\ \mu m$) and length ($> 10\ \mu m$). The Sr_2RuO_4 crystals with high density of Ru lamellas were found to exhibit, quite surprisingly, a broad superconducting transition around 2–3 K.[13] Although the bulk Sr_2RuO_4 becomes superconducting around 1.3–1.5 K, superconductivity first occurs near 3 K in the Ru lamellas and their surrounding regions of Sr_2RuO_4.[13] Single-crystalline Sr_2RuO_4 used to prepare Pb/Sr_2RuO_4/Pb junctions (junctions *A* and *B*) were taken from one single-crystal rod. The value of their T_c was determined by ac magnetic susceptibility measurements on a crystal taken from this rod. A sharp transition at $T_c = 1.35\,K$, typical for a Ru-free Sr_2RuO_4 single crystal, was found. However, x-ray diffraction revealed the presence of pure Ru in the crystals used in the present study. Therefore, we expect that these crystals contain additional superconducting components with T_c

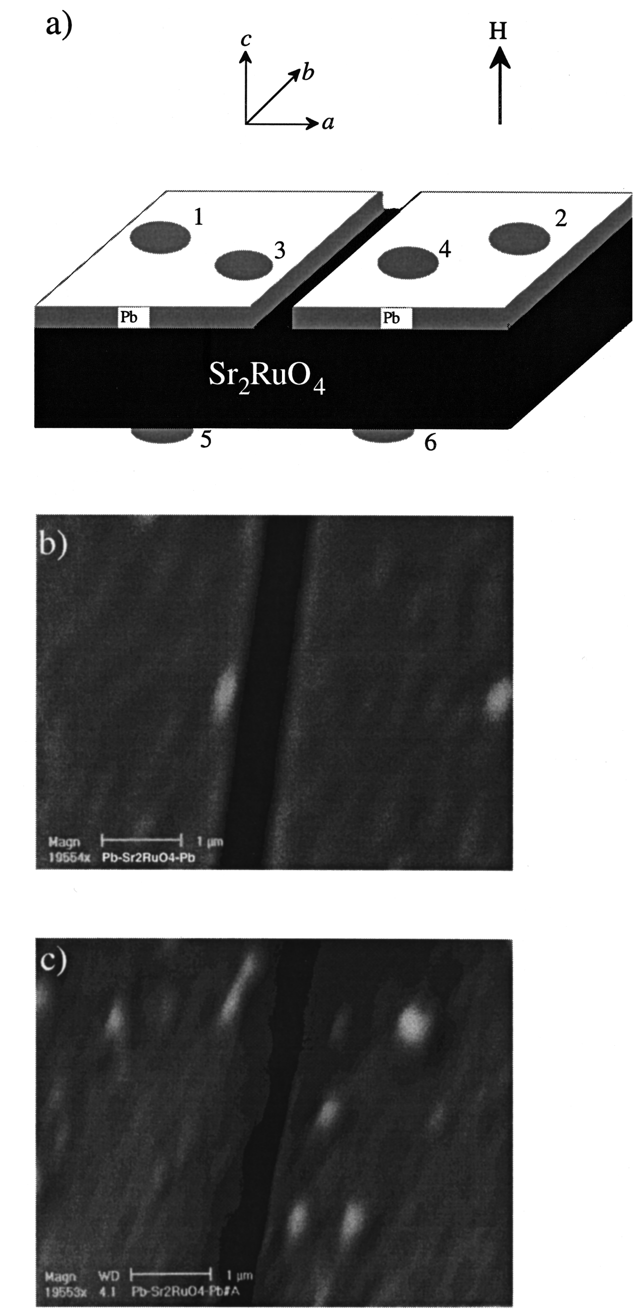

FIG. 1. (a) Schematic of a $Pb/Sr_2RuO_4/Pb$ junction and the electrical contacts for I-V curve measurements. The I-V characteristic of the junction at a fixed T was obtained by measuring the voltage V via leads 3 and 4 while sweeping the dc current I applied via leads 1 and 2. (b) A scanning electron microscope (SEM) picture of a $Pb/Sr_2RuO_4/Pb$ junction. (c) A SEM picture of part of $Pb/Sr_2RuO_4/Pb$ junction (junction A).

>1.35 K due to the presence of Ru lamellas. Although Sr_2RuO_4 single crystals with and without Ru lamellas were both used to prepare $Pb/Sr_2RuO_4/Pb$ junctions, so far only ones with Ru lamellas have yielded nonzero I_c (see below).

To prepare a $Pb/Sr_2RuO_4/Pb$ junction, a submicron filament was placed onto the ab-plane surface of a Sr_2RuO_4 single crystal ($\sim 1.2\times 1.0\times 0.2\,\mathrm{mm}^3$) and used as a shadow mask. Pb of 99.9999% purity was then deposited onto the crystal surface, forming two 0.25 μm thick electrodes separated by a gap. In Fig. 1(b), a scanning electron microscope (SEM) picture of a $Pb/Sr_2RuO_4/Pb$ junction is shown. Although this technique is capable of producing junctions with a well-defined gap width as shown in Fig. 1(b), part of edges of the junction can be less smooth, resulting in a gap with a varying width as shown in Fig. 1(c). Electrical contacts were made by attaching gold wires to the Pb electrodes with silver epoxy [see Fig. 1(a)]. All electrical measurements were carried out in a ^{3}He cryostat with a base temperature of 0.3 K. No magnetic shielding was installed in the cryostat. The I-V characteristic of a $Pb/Sr_2RuO_4/Pb$ junction at a fixed temperature was obtained by measuring the voltage (V) across the junction via leads 3 and 4 while sweeping the dc current (I) applied via leads 1 and 2 [Fig. 1(a)]. All electrical leads entering the sample enclosure in the cryostat were filtered by RF filters with insertion loss of 10 dB at 10 MHz 30 dB at 100 MHz, and 50 dB at 300 MHz.

In order to obtain a nonzero proximity-effect-induced supercurrent in a $Pb/Sr_2RuO_4/Pb$ junction, at least two conditions have to be met: the interface between Pb and Sr_2RuO_4 must be reasonably transparent, and the gap between two Pb electrodes has to be sufficiently small. The normal coherence length for an isotropic N' layer is given by[14]

$$\xi_n(T)=\frac{\hbar \nu_n}{2\pi k_B T} \tag{1}$$

for $T \gg T_{cn}$, where ν_n is the Fermi velocity. Using the value of ν_n given in Ref. 5, we estimate that $\xi_n(4\,\mathrm{K})\approx 73$ nm and $\xi_n(6\,\mathrm{K})\approx 49$ nm for Sr_2RuO_4 in the in-plane direction. Since the in-plane mean free path l_{ab} is around 150 nm,[15] we have $\xi_n < l_{ab}$, indicating that our bilayer $Pb/Sr_2RuO_4/Pb$ junctions are in the clean limit. Assuming a perfectly transparent interface, for a trilayer *SNS* sandwich geometry, the critical current density, in the clean limit, has the form[16]

$$J_c(T)=\frac{2eN_n\nu_n\Delta^2}{\pi k_B T_{cs}}\,\frac{\xi_n(T)}{L}\,e^{-L/\xi_n(T)} \tag{2}$$

for $T \lesssim T_{cs}$. Here L is the width of the gap, N_n is the density of states of N interlayer on the Fermi surface, and Δ is the superconducting energy gap of the S metal. Although no corresponding expression for a $SN'S$ junction with an anisotropic N' layer has been worked out, we may use the above formula to roughly estimate J_c for our $Pb/Sr_2RuO_4/Pb$ junctions. Inserting values of N_n and ν_n for Sr_2RuO_4 (Ref. 5) into Eq. (2) and using $\Delta = 3.06 k_B T_{cs}(1-T/T_{cs})^{1/2}$ (BCS result), we obtain

$$J_c(T)=3.3\left(1-\frac{T}{T_{cs}}\right)\frac{\xi_n(T)}{L}\,e^{-L/\xi_n(T)} \tag{3}$$

in the unit of $\mathrm{A}/\mu\mathrm{m}^2$ for $T \lesssim T_{cs}$. Therefore, it appears that a detectable J_c would require L to be no more than a few times ξ_n.

The real challenge, however, is to obtain a good metallic interface between Pb and Sr_2RuO_4. We have prepared many $Pb/Sr_2RuO_4/Pb$ junctions under varying conditions using the ab-plane surface of Sr_2RuO_4 single crystals without Ru lamellas. So far no finite supercurrent has been observed in any of these junctions. The typical I-V characteristic for these junctions is shown in Fig. 2. Although a finite jump is

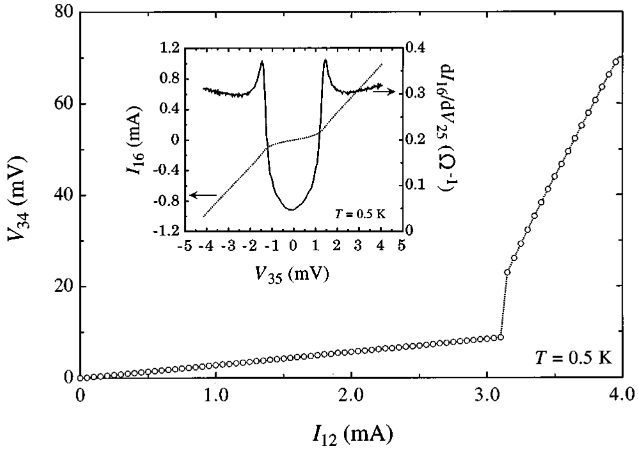

FIG. 2. Typical I-V characteristic for $Pb/Sr_2RuO_4/Pb$ junctions, where Ru-lamella-free Sr_2RuO_4 was used. The current was applied via leads 1 and 2 and voltage was detected via leads 3 and 4. Inset shows I-V characteristic for Pb/Sr_2RuO_4 along the c axis at $T = 0.5$ K, where I was applied via leads 1 and 6 and voltage was detected via leads 3 and 5 (see text).

seen in the I-V curve, no true zero-voltage supercurrent was detected. The physical origin of the absence of supercurrent appears to be related to the interface between Pb and Sr_2RuO_4. We have studied the I-V characteristics of the interface between Pb and Sr_2RuO_4 by measuring the voltage via contacts 3 and 5 while applying the bias current via contacts 1 and 6 [see Fig. 1(a)]. The I-V curves were found to exhibit tunneling characteristics as shown in the inset of Fig. 2. The detailed analysis of the tunneling results between Pb and Sr_2RuO_4 along the c axis and its implications on the symmetry of the pairing state will be presented elsewhere.[17] The tunneling feature between Pb and Sr_2RuO_4 indicates that an insulating barrier (I), probably resulting from oxidation of Pb was present at the interface. For these $SIN'IS$ junctions, the smallest gap (<0.3 μm) did not appear to warrant a Josephson coupling down to 0.3 K. Interestingly, Sr_2RuO_4 single crystals with Ru lamellas can help bypass this difficulty. In this case, small parts of Pb electrodes may form contacts with Ru lamellas, and then with Sr_2RuO_4. Since both Ru and Pb are conventional metals involving no oxygen, a metallic interface between them is expected. The interface between Ru lamellas and Sr_2RuO_4 should be reasonably good because it is naturally formed. A good $SN'S$ junction may be achieved through these Ru lamellas "windows" even though the rest part of the interface between Pb and Sr_2RuO_4 remains insulating.

The central result of the present work is that a finite zero-voltage critical current I_c was obtained and an anomalous temperature dependence of $I_c(T)$ was found for two $Pb/Sr_2RuO_4/Pb$ junctions. In Fig. 3(a) the temperature dependences of the critical current I_c in various magnetic fields (H) applied perpendicular to the ab plane of the Sr_2RuO_4 single crystal ($H \perp ab$) are shown for junction A. The inset of Fig. 3(a) shows the I-V characteristic at $T = 0.9$, 1.3, 2 K. At low currents, the voltage measured across the junction is zero within the experimental accuracy. Because of the unavoidable rounding at finite T, the values of I_c were determined by extrapolating the linear region of the I-V curves to $V = 0$ as indicated in the inset of Fig. 3(a). Defining I_c as the maximum current for $V = 0$ results in the same features in $I_c(T)$. At the lowest magnetic field $H = H_s$, which includes the Earth's and other stray magnetic fields in the order of 1 Oe in our cryostat, I_c was found to initially increase with decreasing T down to about 1.3 K with a concave curvature. Remarkably, beginning at the T_c of Sr_2RuO_4, a downturn was observed, resulting in an anomalous peak in $I_c(T)$. At $T \approx 0.9$ K, I_c reduced to about 50% of $I_c(T_{cn})$. As T was further lowered, I_c increased again. The peak was suppressed and shifted to a lower temperature for $H = 0.5$ kOe applied along the c axis of Sr_2RuO_4 and vanished completely for $H = 1.0$ kOe. [The value of H_{c2} for Sr_2RuO_4 with $T_c \approx 1.35$ K was found to be around 0.53 kOe (Ref. 18).] Since the H_{c2} of Pb thick films (≈ 0.4 μm) can be as high as 1.3 kOe,[19] the Pb electrodes should remain superconducting even at 1 kOe. As can be seen in Fig. 3(b) essentially identical behavior was observed in $Pb/Sr_2RuO_4/Pb$ junction B.

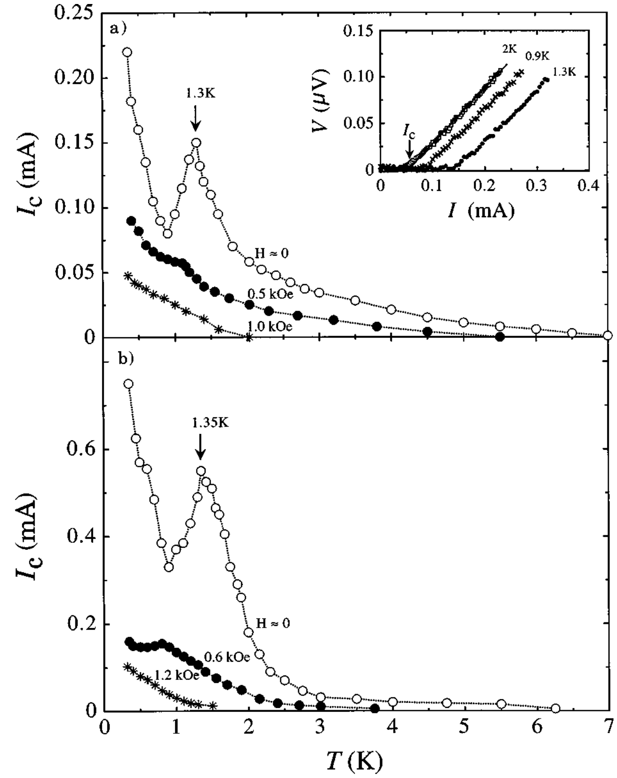

FIG. 3. (a) Critical current $I_c(T)$ for $Pb/Sr_2RuO_4/Pb$ junction (junction A) in various magnetic fields. The inset shows the I-V characteristics at $T = 0.9$, 1.3, 2 K. (b) Critical current $I_c(T)$ for junction B in the indicated magnetic fields.

In order to identify the physical origin of this downturn, it is important to make sure that the supercurrent observed in junctions A and B is due to the proximity Josephson coupling. A characteristic feature of an SNS junction is that its $I_c(T)$ curve exhibits a concave curvature because of the exponential dependence of I_c on $\xi_n(T)$. Although no detailed data fitting could be carried out because the $I_c(T)$ formula for our junctions (highly anisotropic SNS junctions in the clean limit) is not available in the literature, the concave curvature [see Figs. 3(a) and 3(b)] suggests that our junctions are SNS in character for $T > T_{cn}$. Furthermore, we may roughly estimate the value of I_c using Eq. (3). As can be seen in Fig. 1(c) the smallest gap size is ~ 0.3 μm. Assuming

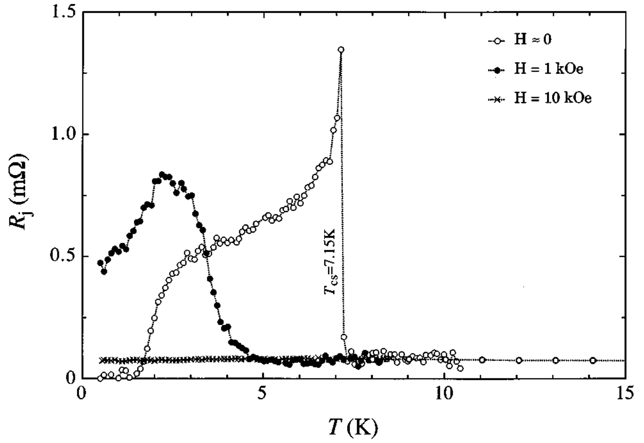

FIG. 4. Junction resistance $R_j(T)$ for junction *A* between 0.3 and 15.0 K in various magnetic fields. The applied dc current was 0.1 mA.

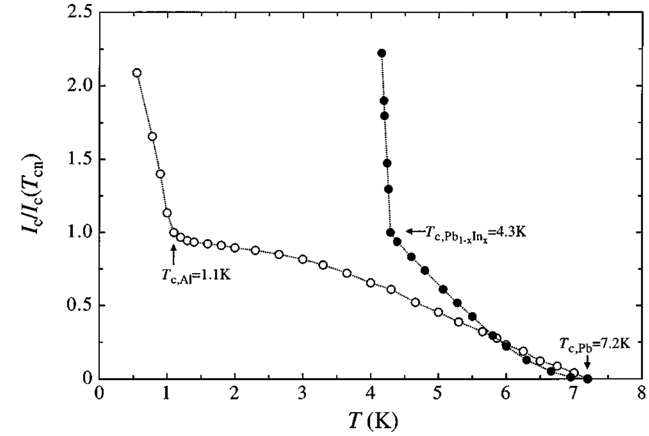

FIG. 5. Temperature dependence of normalized critical current $I_c/I_c(T)$ for Pb/Al/Pb (hollow circles) and Pb/$Pb_{1-x}In_x$/Pb (solid circles) junctions at $H = H_s$ (see text).

that the measured I_c was mainly due to the contribution of a junction formed by two 1 μm^2 Ru-lamella ''windows'' (see below) separated by $L \approx 0.5\ \mu$m (taking into account the penetration depth of Pb at 6 K), we obtain $I_c(6\text{ K}) \approx 2\ \mu$A for junction *A*. As shown in Fig. 3(a) the measured I_c is about 6 μA at 6 K, reasonably close to the estimated value.

The behavior of the junction resistance $R_j(T)$ can provide additional information concerning the nature of our junctions. In Fig. 4, $R_j(T)$ (measured at 0.1 mA dc current) in various magnetic fields for junction *A* is shown. At the T_c of Pb, 7.15 K, a sharp jump in R_j was observed. Similar features were also seen in junction *B*. R_j started to drop rapidly below 3 K and reached zero around 1.4 K, consistent with the ac magnetic susceptibility results. The peak with a suppressed height and a broadened width shifted to a lower *T* at $H = 1$ kOe, vanishing completely in sufficiently high fields. This suggests that the peak was due to the onset of superconductivity in Pb. Similar features were seen in Al,[20,21] where *SNS* junctions were involved. It was found that the narrower and/or cleaner the *N* region, the sharper and higher the resistance peak was due to the charge imbalance[22] at the *SN* interface. Therefore, the presence of the large resistance anomaly in our junctions suggests that they are of $SN'S$ type with a narrow N' layer. In such junctions, a Josephson coupling with nonzero critical current is expected.

The effective junction area may be estimated from the junction resistance and the magnetic-field dependence of I_c. Using the resistivity values for thick Pb films ($\approx 1 \times 10^{-5}\Omega$ cm) and Sr_2RuO_4 ($\rho_{ab} = 1.6 \times 10^{-6}\Omega$ cm, $\rho_c = 2.3 \times 10^{-3}\Omega$ cm), we estimate $R_j < 1 \times 10^{-5}\Omega$, much smaller than observed $R_j(\approx 1 \times 10^{-4}\Omega)$ above T_{cs}. R_j will be even smaller if the effect of Ru lamellas with lower resistivity ($\rho = 7.4 \times 10^{-6}\Omega$ cm at 295 K) is considered. The above implies that the observed Josephson current between two Pb electrodes was mainly through few Ru lamellas ''windows'' that happened to locate at the narrowest part of the gap. The Josephson current of an *SNS* junction should vanish once the total flux enclosed in the effective area of the *N* layer reaches a few flux quanta. Figure 3(a) shows that I_c is nonzero for $H = 0.5$ kOe at 5 K. As mentioned above [also see Fig. 1(c)], the narrowest part of the gap for Junction *A* is around 0.3 μm. This suggests that the width for Ru lamella ''windows'' is in the order of 1 μm. Since I_c decays exponentially with the increase of the gap width, the rest part of the junction should not contribute to I_c. It is possible that Pb/Ru/Pb junctions may also be present in the sample. However, no Pb short across the gap is expected, since no supercurrent in any junction prepared in the same way on Ru-lamella-free crystals has ever been observed.

Why did I_c drop in Pb/Sr_2RuO_4/Pb junctions when bulk Sr_2RuO_4 became superconducting? It is possible that this downturn was due to some subtle effects such as expelling of the (residual) magnetic flux from the bulk Sr_2RuO_4 below T_{cn}. Since our cryostat was not magnetically shielded, the Earth's and possibly other stray fields in the order of 1 Oe did exist in the experimental space. If the Ru lamellas were normal at $T_{cn} = 1.35$ K and connected to both Pb electrodes, the expelled flux would essentially create a higher external magnetic field at these Pb/Ru/Pb microbridges, suppressing the Josephson coupling across the junction so that $I_c(T)$ decreases. However, since the Ru lamellas and their surrounding Sr_2RuO_4 regions became superconducting around 3 K, the residual magnetic flux should not have entered the Pb/Ru/Pb microbridges at 1.35 K even if they were present in the sample.

To further understand the physical origin of the drop in I_c for Pb/Sr_2RuO_4/Pb junctions, a control experiment was carried out. We prepared Pb/Al/Pb and Pb/$Pb_{1-x}In_x$/Pb junctions following the same procedure as that for Pb/Sr_2RuO_4/Pb junctions. Sr_2RuO_4 was replaced by 4000 Å thick Al and 1000 Å thick $Pb_{1-x}In_x$ films, respectively. In Fig. 5, values of I_c for Pb/Al/Pb (hollow circles) and Pb/$Pb_{1-x}In_x$/Pb (solid circles) junctions at $H = H_s$ are plotted as $I_c/I_c(T_{cn})$ against *T*. Below T_c of Pb, I_c is nonzero and increases with decreasing *T*, rising sharply as *T* is lowered to below $T = T_{cn} = 1.1$ K for Al or 4.35 K for $Pb_{1-x}In_x$. These observations suggest that the downturn found in $I_c(T)$ for our Pb/Sr_2RuO_4/Pb junctions is related to the particular properties of Sr_2RuO_4.

The observation that superconductivity in Sr_2RuO_4 actually suppresses the Josephson coupling between two Pb electrodes may be understood most naturally if the superconducting pairing symmetry in Sr_2RuO_4 is qualitatively different from that in Pb (*s* wave). This unconventional pairing may be of either odd or even parity, or perhaps a combination of both. If it were of non-*s*-wave even-parity, say, *d* wave, no significant suppression in I_c should be expected below T_{cn} as the *s*- and the *d*-wave pairings do not exclude each other. Assuming the pairing in Sr_2RuO_4 were of odd-parity (*p*-wave), the non-zero-*s*-wave order parameter can still be induced in Sr_2RuO_4 by the proximity effect, if the spin-orbit scattering and/or the gradient of the order parameter at the interface are considered.[23] However, the *s*- and *p*-wave order parameters exclude each other, which will lead to a strong suppression in I_c below T_{cn}.[24] If Pb/Ru/Pb microbridges were present in our junctions, they could not result in a downturn in I_c at 1.3 K because Ru lamellas were superconducting already at this temperature. Nevertheless, they may be responsible, at least in part, for the upturn of I_c seen at low temperatures, which, on the other hand, could also have an intrinsic physical origin.[23,25]

Recently, two groups[23,25] have independently carried out calculations of $I_c(T)$ for our particular experimental configuration assuming a *p*-wave pairing state in Sr_2RuO_4. Both calculations have reproduced the features of $I_c(T)$ seen in our experiment. In particular, Ref. 23 suggests that the drop in $I_c(T)$ was the result of two competing terms in the Josephson coupling: one derived from the conventional proximity effect and the other from the effect of the *p*-wave superconducting order parameter in Sr_2RuO_4. The latter has a negative contribution which favors a nonzero phase shift between two Pb electrodes, leading to a ''back-flow'' Josephson current. In this picture, the intrinsic contribution from the *s*-wave proximity coupling will lead to the upturn in I_c at lower temperatures.

The ''backflow'' Josephson current scenario can be tested experimentally. We have carried out an experiment involving pressing a 99.9999% pure In wire onto the *ab* plane of a Sr_2RuO_4 single crystal with Ru lamellas taken from the same crystal rod as that for junctions *A* and *B*. As shown in Fig. 6, nonzero I_c was observed below $T=3$ K for In/Sr_2RuO_4 along the *c* axis. Since the bulk Sr_2RuO_4 is not superconducting above 1.35 K, the observed supercurrent can only be carried by Ru lamellas and the surrounding Sr_2RuO_4 regions. No suppression of I_c was seen at $T_{cn}=1.35$ K. Since only one In electrode was present in the sample, no ''backflow''

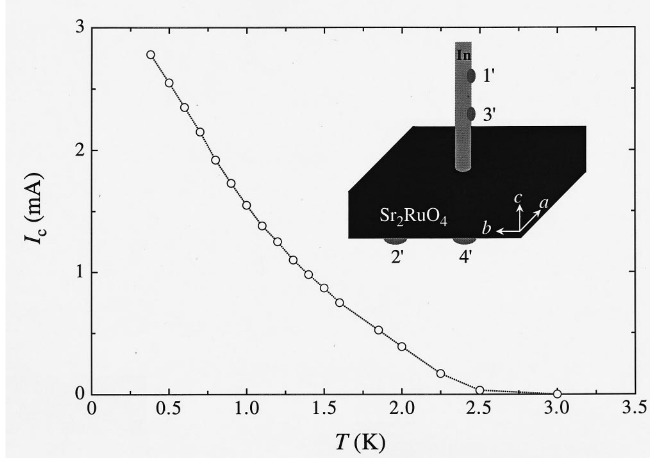

FIG. 6. Temperature dependence of critical current $I_c(T)$ for a sample of In wire pressed on Sr_2RuO_4 single crystal (with Ru lamellas) between 0.3 and 3.5 K. The inset shows a schematic of the sample and the electrical contacts. The current was applied through 1′ and 2′ and the voltage was measured via 3′ and 4′.

of the Josephson current could occur, supporting the model advanced in Ref. 23. This result also suggests that the magnetic focusing, which would be relevant in this experimental configuration, was not responsible for the downturn observed in the Pb/Sr_2RuO_4/Pb junctions.

In summary, we have performed experiments on Pb/Sr_2RuO_4/Pb junctions and observed a reduction in the critical current when temperature is lowered below the T_c of Sr_2RuO_4. This finding seems to provide additional evidence for an unconventional pairing state in Sr_2RuO_4. In order to fully resolve the pairing symmetry issue for this material, more phase-sensitive experiments on Ru-lamella-free Sr_2RuO_4 need to be carried out.

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