Magnetic field effect on the pressure-induced superconducting state in the hole-doped two-leg ladder compound $Sr_2Ca_{12}Cu_{24}O_{41}$

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We report electrical resistivity on a single crystal of the hole-doped two-leg ladder compound $Sr_2Ca_{12}Cu_{24}O_{41}$, which becomes superconducting with $T_c \sim 5$ K only at pressures above ~3.0 GPa. Measurements were performed at nearly hydrostatic pressures up to 5.7 GPa and low temperatures down to 100 mK under static magnetic fields up to 20 T parallel to the *a* axis (along the ladder rungs) and up to 7 T parallel to both the *b* axis (perpendicular to the ladder plane) and the *c* axis (along the ladder legs). A clear difference in the resistive upper critical field $H_{c2}(T)$ is observed among these three directions, confirming that this system has a highly anisotropic superconducting ground state. Also, $H_{c2}(T)$ parallel to the ladder plane is found to exceed the Pauli limit by a factor of more than 2, suggesting either a strong spin-orbit scattering or spin-triplet pairing. Furthermore, it is implied, from measurements of resistivity versus angle of magnetic field in the *bc* plane, that another superconducting phase is stable below around 3 K only when the magnetic field is applied exactly along a certain direction that is ±35° from the ladder direction.

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I. INTRODUCTION

A concept of $S=\frac{1}{2}$ Heisenberg-type spin-ladder has offered predictions[1–3] of two features: the existence of a spin gap and superconductivity by doped hole-pairs with the spin gap. These are closely related to features of high-T_c cuprates (HTSC). The most important point in the predictions is that the pairing interaction is supposed to be caused by purely electronic origin, namely, magnetic interaction. At present, a compound $Sr_{14-x}Ca_xCu_{24}O_{41}$ (Refs. 4–6) is the only known candidate as a real system having the theoretically predicted properties of the doped spin-ladders as mentioned above, and also is the first nonsquare lattice superconducting cuprate.

This spin-ladder system becomes superconducting with $T_c \lesssim 12$ K only in a pressure P above ~3.0 GPa when $x \geqslant 10$ and the T_c shows the bell-shaped curve against applied pressure.[4–6] These features are quite similar to those of HTSC, having two-dimensional (2D) CuO_2 planes in which the spin gap behavior has been observed so far and the superconducting phase is stabilized only in a certain range of carrier doping. The essential difference is that this ladder system has conducting layers composed by weakly coupled one-dimensional (1D) Cu_2O_3 two-leg ladders with a spin gap and shows 1D charge transport along the ladder at low pressure below ~2 GPa.[7] Furthermore, the anisotropy ratio of the resistivity in this system is quite similar to that of a quasi-1D organic superconductor $(TMTSF)_2PF_6$ (Ref. 8), which has conducting layers composed by weakly coupled 1D chains. On the other hand, the anisotropic electrical resistivity measurements under high pressure[5] indicate that pressure causes a dimensional crossover from 1D to anisotropic 2D charge transport in the normal state and then superconductivity is suggested to occur in the Cu_2O_3 two-leg ladders as in the anisotropic 2D system. However, the superconducting property, especially under magnetic field, of this ladder system is not well understood yet. There are only few measurements concerning the upper critical field on this system, for example, ac susceptibility[9] at a pressure of 4.0 GPa up to 10 T and resistivity[10] at a pressure of 4.8 GPa up to 7 T.

In this paper, we present the results of precise magnetotransport measurements of a $Sr_2Ca_{12}Cu_{24}O_{41}$ single crystal at three different pressures where $dT_c/dP>0$, $dT_c/dP \simeq 0$, and $dT_c/dP<0$ in magnetic fields applied along all three crystallographic axes. In these measurements, the magnetic field range is extended up to 20 T parallel to the *a* axis (along the ladder rungs) and up to 7 T parallel to both the *b* axis (perpendicular to the ladder plane) and the *c* axis (along the ladder legs).

II. EXPERIMENT

A single crystal of $Sr_2Ca_{12}Cu_{24}O_{41}$ was grown by the traveling-solvent floating zone method using an infrared furnace under oxygen gas.[7] Using a newly developed self-clamped high-pressure cell[11] that employs Bridgman anvils with a Teflon capsule filled with a pressure transmitting liquid medium (a 1:1 mixture of Fluorinert FC 70 and FC 77), the absolute values of electrical resistivity by a four-probe technique were measured under nearly hydrostatic pressure in the temperature range down to around 100 mK by a dilution refrigerator. A sample with dimensions of 0.3 (*a* axis) $\times 0.2$ (*b* axis) $\times 0.7$ (*c* axis) mm^3 was used in the resistivity measurements with an ac current (10 or 100 μA at 15.9 Hz, ohmic response without self-heating effect was confirmed within this current range) along the *c* axis. We used two superconducting magnets: a Helmholtz-type horizontal rotatable 7 T parallel to the *bc* plane and a cylindrical 20 T parallel to the *a* axis. Although it was required to warm the cryostat with the pressure cell up to around 100 K in order to

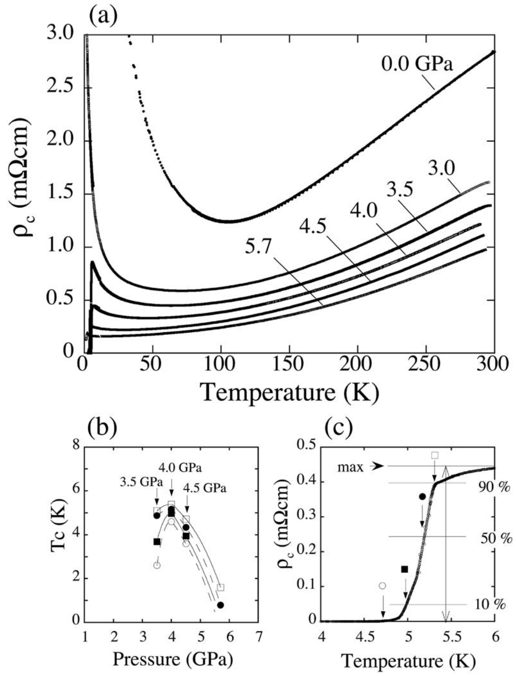

FIG. 1. (a) $\rho_c(T)$ of $Sr_2Ca_{12}Cu_{24}O_{41}$ at several pressures. (b) Pressure dependence of T_c that was determined by several criteria as illustrated in (c) for the case at 4.0 GPa as an example where we define T_c, which satisfies a condition that $\rho_c(T_c)$ is equal to a fixed percentage of the resistive maximal value before the transition. Lines in (b) are guides for the eyes.

change a magnet to another at a fixed pressure, this thermal cycle did not cause any change in the pressure because T_c did not change in this process. This ensured that these experiments were done under exactly the same pressures for the identical sample.

For pressure calibration at low temperatures in this pressure cell, we have measured the temperature dependence of ac susceptibility of lead to determine the superconducting transition temperature. In this measurement, at ambient pressure, the superconducting transition temperature T_c is observed at 7.2 K, with a transition width ΔT_c of 0.03 K. It is noted that the ΔT_c under each pressure still remains 0.03 K, which is at ambient pressure. This result clearly indicates that the generated pressures in this pressure cell are close to the hydrostatic one although the pressure transmitting medium is solidified in this temperature region. From this result, we construct the pressure calibration curve of this pressure cell by using the data of Bireckoven and Wittig.[12] A reproducibility of this curve is confirmed with a precision of ±0.1 GPa at 2.55 GPa by four different measurements which monitor the resistive transition of Bi due to the I-II structural phase transition at room temperature. More detailes are described elsewhere.[11]

III. RESULTS AND DISCUSSION

A. ρ_c under magnetic fields

Figure 1(a) shows the temperature dependence of resistivity ρ_c along the c axis of $Sr_2Ca_{12}Cu_{24}O_{41}$ up to 5.7 GPa. At ambient pressure, ρ_c is ~3 mΩ cm at room temperature and shows T-linear metallic dependences upon cooling. These facts indicate that this ρ_c clearly shows the transport properties of the c axis, namely, the ladder direction at ambient pressure. At room temperature, ρ_c decreases with pressure continuously. Above 3.5 GPa the sample becomes superconducting at around 5 K and the T_c exhibits the bell-shaped curve against pressure as shown in Fig. 1(b) where T_c is determined in several ways as shown in Fig. 1(c). These pressure effects on the transport and superconducting properties in the zero field are consistent with the previously reported results on the $x=11.5$ single crystal.[5]

Figure 2 shows the temperature dependence of ρ_c of $Sr_2Ca_{12}Cu_{24}O_{41}$ at 3.5, 4.0, and 4.5 GPa in various magnetic fields applied along the a axis up to 20 T and the b axis up to 7 T. It is clearly observed that, with increasing pressure from 3.5 to 4.5 GPa, the pressure influences the qualitative changes in the behavior of resistive-transition in the magnetic fields. For the fields up to 7 T along the b axis, at 3.5 and 4.0 GPa the superconductivity is not completely destroyed while at 4.5 GPa $\rho_c(T)$ at 5.5 and 7 T shows almost the same curve at least down to around 1 K. These qualitative changes in the behavior of resistive transition in the magnetic fields are also observed in field sweep measurements at fixed temperatures. The results are shown in Fig. 3, where the fields were applied along the a axis up to 18 T and the b axis up to 7 T at pressures of 4.0 and 4.5 GPa. As seen in Fig. 3, for the fields up to 7 T along the b axis, at 4.0 GPa the resistivity has not reached a constant value while at 4.5 GPa the resistivity has reached a constant value at 7 T. These facts indicate that normal state resistivity is recovered at 4.5 GPa under the magnetic fields along the b axis above 5.5 T at temperatures down to about 1 K. Thus the $\rho_c(T)$ curve at 4.5 GPa above 1 K under 7 T along the b axis refers to normal state resistivity.

B. Resistive upper critical field

The resistive upper critical field $H_{c2}(T)$ is defined as the magnetic field where the normal state resistivity is recovered in the measurement of field sweep at a fixed temperature. In this sense, we can determine $H_{c2}(T)$ only for data at 4.5 GPa where the normal state resistivity is recovered within the measured magnetic field range. However, due to the difficulty of applying this criterion to the data perfectly, we determined critical magnetic fields H^n where $\rho_c(T)$ is equal to a fixed percentage ($n=10\%$, 50%, and 90% and $n=$"$\rho=0$" for 0%) of the normal state resistivity, that is $\rho_c(T)$ curve above 1 K at 4.5 GPa and 7 T as denoted $\rho_c(H=7\mathrm{T}//b)$ as shown in Fig. 4. From the field dependence of the resistive transition as shown in Figs. 2 and 3, together with the results along the c axis not shown here, the critical fields at 4.5 GPa for all crystallographic axes are determined and summarized in Fig. 5. Figure 6 shows a summary of critical fields $H^{\rho=0}$ at several pressures. As we can see in Figs. 5 and 6, it is found that there is no significant discrepancy between the results from temperature sweep and field sweep data. Also, it is noted that $H_{c2}(T) > H^{90\%}(T)$ and $H^{\rho=0}(T)$ corresponds to the irreversibility line in the case of HTSC. As it has been observed and

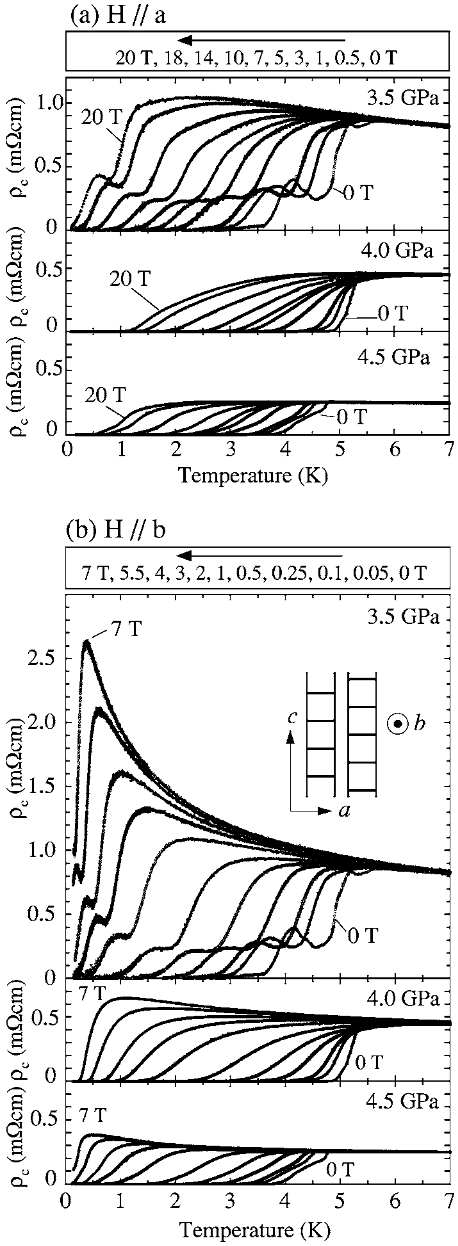

FIG. 2. $\rho_c(T)$ of $Sr_2Ca_{12}Cu_{24}O_{41}$ at 3.5, 4.0, and 4.5 GPa in various magnetic fields along (a) the *a* axis and (b) the *b* axis. Inset in (b) shows a schematic structure of the two-leg ladders viewed from the *b* axis.

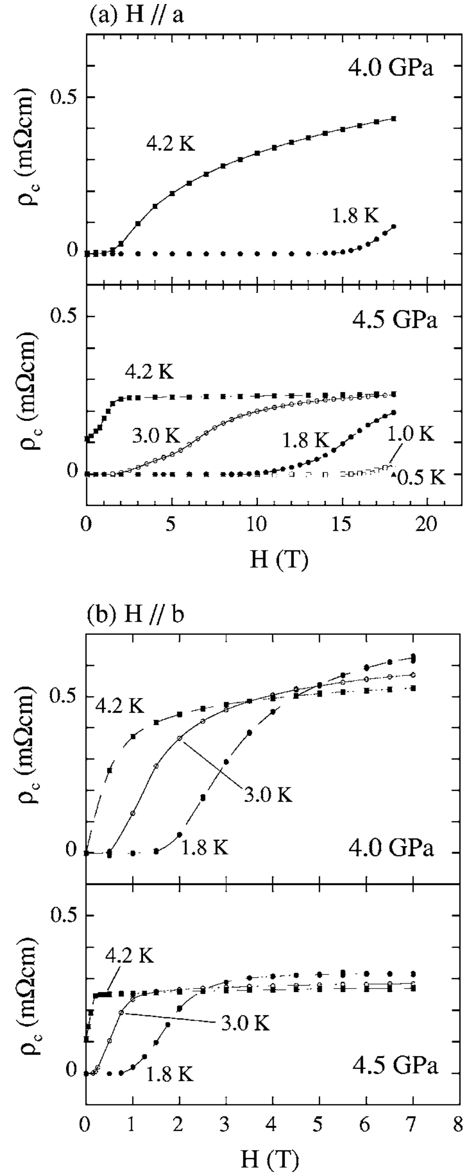

FIG. 3. Magnetic field dependence of ρ_c of $Sr_2Ca_{12}Cu_{24}O_{41}$ at 4.0 and 4.5 GPa and at several temperatures for the fields along (a) the *a* axis and (b) the *b* axis.

discussed in HTSC, it is difficult to interpret the resistive upper critical field because in some cases the motion of flux may affect the shape of the resistive transition. However, at present we cannot distinguish a difference between the resistive upper critical field and the thermodynamic upper critical field for the present ladder system because of the technical difficulty of performing thermodynamic experiments, such as specific heat, at hydrostatic pressures above 3 GPa under magnetic fields.

As observed in Fig. 5, there is the clear difference in the critical fields, also implied in $H_{c2}(T)$ as well, among these three directions. This fact clearly indicates that this system has a highly anisotropic superconducting ground state. It is seen in Fig. 6 that the anisotropy of $H^{\rho=0}(T)$ is unchanged at three different pressures where $dT_c/dP>0$, $dT_c/dP\simeq 0$, and $dT_c/dP<0$ within measured temperature and magnetic field range, implying that the anisotropy of $H_{c2}(T)$ is also unchanged in the whole pressure range. The most important fact seen in Fig. 5 is that H_{c2} along the *a* axis, parallel to the conducting ladder plane, exceeds the value of Pauli paramagnetic limit [as indicated by arrows in Fig. 5 for a criterion of $T_c(50\%)$] by a factor of more than 2, which is given by $H_p(T=0)=1.84\ T_c(H=0)$ for isotropic singlet *s*-wave pairing without spin-orbit scattering.[13]

As reported by the anisotropic electrical resistivity measurements under high pressure,[5] superconductivity is suggested to occur in the Cu_2O_3 two-leg ladders as in the anisotropic 2D system. This implies that coherence length along the *b* axis ξ_b becomes shorter than a distance between the

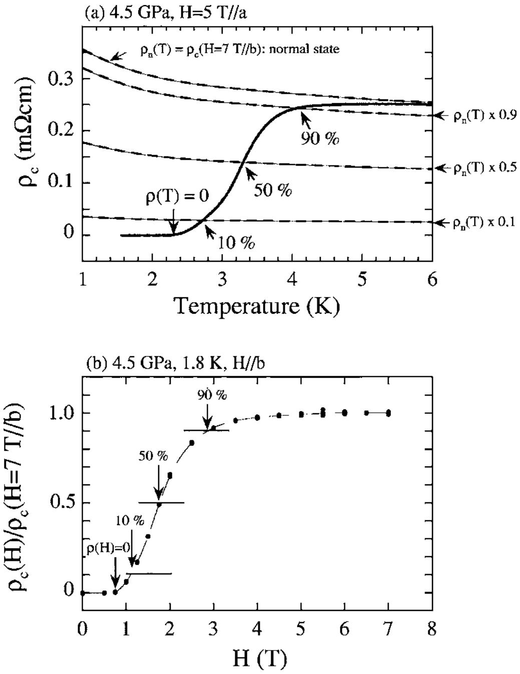

FIG. 4. Examples of criteria to define T_c, where we define T_c which satisfies a condition that $\rho_c(T_c, H^n)$ is equal to a fixed percentage n of the normal state value $\rho_n = \rho_c(H=7\ \mathrm{T} \| b)$ at 4.5 GPa for (a) the temperature sweep data and for (b) the field sweep data.

ladder plane. In fact, a value of $\rho_c \sim 0.5$ mΩ cm gives a mean free path along the c axis $l_c \sim 10-100$ Å by assuming a simple free electron model with a carrier density estimated from a Hall coefficient of $\sim 2\times10^{-3}$ cm^3/C.[14] We would

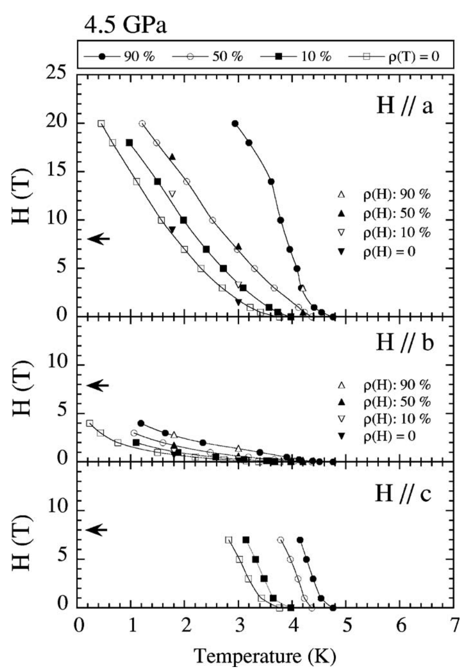

FIG. 5. Critical fields $H^{90\%}$, $H^{50\%}$, $H^{10\%}$, $H^{\rho=0}$ of $Sr_2Ca_{12}Cu_{24}O_{41}$ at 4.5 GPa for H$\|a$ axis, H$\|b$ axis, and H$\|c$ axis. Data for $\rho_c(H)$: 90%, 50%, and 10% and $\rho_c(H)=0$ are from the field sweep data (Fig. 3). Lines are guides for the eyes. Arrows indicate $H_p(T=0)$ for $T_c(50\%)$ (see text).

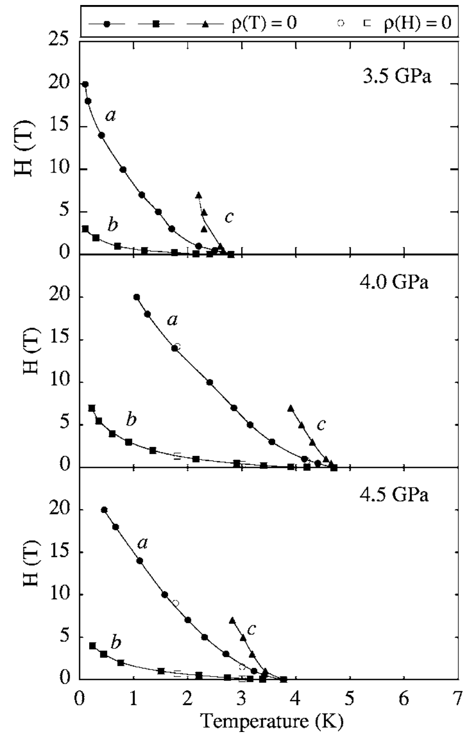

FIG. 6. Critical fields $H^{\rho=0}$ at 3.5, 4.0, and 4.5 GPa for H$\|a$ axis, H$\|b$ axis, and H$\|c$ axis. Lines are guides for the eyes.

expect ρ_b to be lower than ρ_c by two orders of magnitude,[7] this means l_b to be $\sim 0.1-1$ Å. Also, this system is supposed to be a dirty superconductor due to being a highly doped system; thus we expect that coherence length ξ is shorter than l; then ξ_b would be less than $\sim 0.1-1$ Å. This fact implies that ξ_b seems to be shorter than the distance between the ladder planes about 7 Å.[15] Therefore ξ_b is expected to become smaller than the ladder spacing at a certain temperature. Below this temperature orbital effects as a mechanism for the quenching of superconductivity might be suppressed, then H_{c2} parallel to the ladder plane would be infinite in the absence of both Pauli limit and spin-orbit scattering that reduces Pauli paramagnetic effect, as predicted for the upward curvature of $H_{c2}(T)$ for 2D layered superconductors.[16] Thus one possible explanation for the observed absence of Pauli limit is that there is a spin-orbit scattering that is strong enough for the quenching Pauli paramagnetic effect, as observed in layered superconductors $TaS_{2-x}Se_x$ ($x=0$ and 0.4).[17] Besides such a strong spin-orbit scattering, one may speculate that another possible explanation for the absence of Pauli limit might be due to spin-triplet pairing. In fact, very recently, Fujiwara *et al.*[18] have shown, from the high-pressure NMR experiment at 3.5 GPa, that the Knight shift does not change at T_c within their experimental accuracy. This fact indicates a possibility of triplet superconductivity in this system. Furthermore, they have also observed the peak of T_1^{-1} at T_c. These behaviors are quite similar to those of a quasi-1D molecular superconductor $(TMTSF)_2PF_6$ in which a large evolution of H_{c2} parallel to conducting layers at low temperatures, exceeding the Pauli limit, has been observed.[19] This might be due to spin-triplet pairing that has been shown recently.[20] In contrast, the Pauli limit is observed

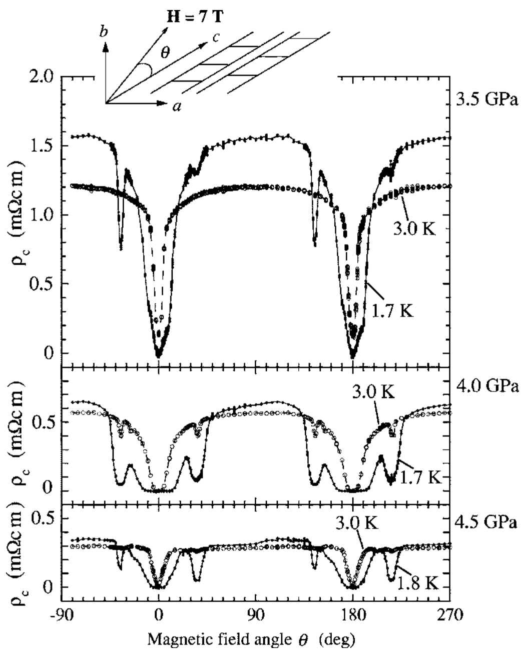

FIG. 7. ρ_c vs angle θ of a magnetic field $H=7$ T in the *bc* plane of $Sr_2Ca_{12}Cu_{24}O_{41}$ at pressures of 3.5, 4.0, and 4.5 GPa in the two different temperature regions of 1.7 to 1.8 (open circles) and 3 K (closed circles). Lines are guides for the eyes.

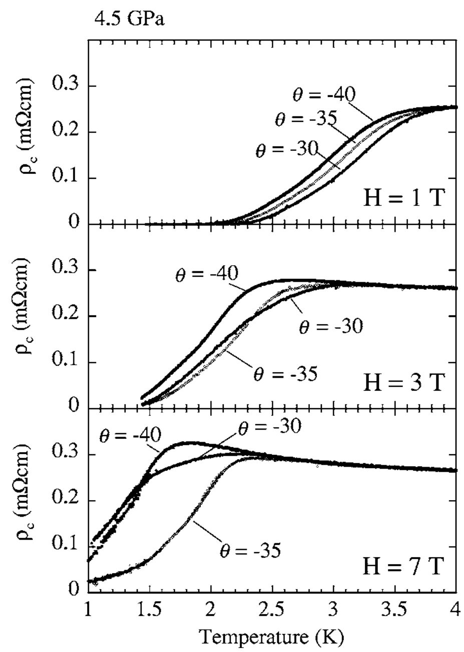

FIG. 8. $\rho_c(T)$ of $Sr_2Ca_{12}Cu_{24}O_{41}$ in fields $H=1$, 3, and 7 T in the *bc* plane with angles of $\theta=-30°$, $-35°$, and $-40°$ from *c* axis (ladder direction).

in $YBa_2Cu_3O_{7-\delta}$ (Ref. 21) for H_{c2} parallel to the conducting CuO_2 layer, in which a singlet *d*-wave superconducting state might be realized.

C. ρ_c vs angle of magnetic field in the *bc* plane

Figure 7 shows the results of ρ_c versus angle of magnetic field of 7 T in the *bc* plane in the two different temperature regions, 1.7 to 1.8 K and 3.0 K, at each pressure. As seen in the figure, apart from resistive drops at the ladder direction (*c* axis: $\theta=0°$ and 180°) due to the anisotropy of the critical field, other extra resistive drops are clearly observed at the angles of ±35° from the ladder direction symmetrically. This angle is independent of both current and magnetic field, which indicates that these resistive drops are not caused by the motion of flux due to the Lorentz force, which depends on both current and magnetic field. The angle is also independent of applied pressure. On the other hand, the sharpness of this resistive drops at ±35° seems to be similar to that at *c* axis, namely, $\theta=0$ and 180°. Therefore this resistive anomaly is supposed to be caused by superconductivity, possibly another superconducting phase. Also, it is difficult to explain that the symmetrical appearance of this additional superconducting phase at the angles of ±35° from the ladder direction would be caused by the inhomogeneity of a pressure-induced superconducting region in the sample. Figure 8 shows the results of $\rho_c(T)$ curves at 4.5 GPa under three different magnetic fields, 1, 3, and 7 T, applied along the directions with three different angles of −30°, −35°, and −40° from the ladder direction. At 1 T the shift of resistive transition in these curves seems to be in accordance with its anisotropy from −30° to −40°. With increasing fields, the shift of the transition goes to lower temperature; then, just after passing through around 2 to 3 K, the shift of the transition in the curve with an angle of −35° starts to deviate from the two curves with angles of −30° and −40°. This behavior is also observed in field sweep measurements at 1.8 K with different field angles, as shown in Fig. 9. These facts indicate that this additional superconducting phase becomes to develop below around 3 K only when the magnetic field is applied exactly along a certain direction that is ±35° from the ladder direction. Although, at present, a physical meaning of ±35° from the ladder direction is unknown, we might say at least that the appearance of this additional superconducting phase clearly depends on both the temperature and the direction of the applied magnetic field. Also, it is noted that this addi-

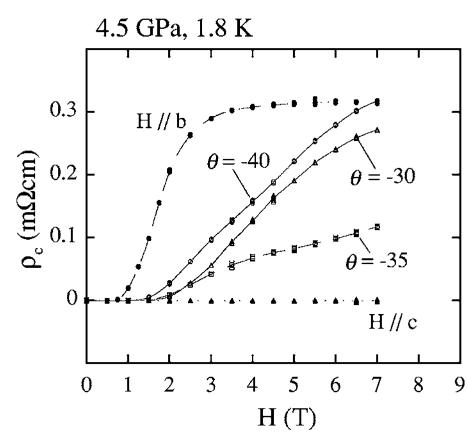

FIG. 9. Magnetic field dependence of $\rho_c(H)$ of $Sr_2Ca_{12}Cu_{24}O_{41}$ at 4.5 GPa and 1.8 K for the fields in the *bc* plane with angles of $\theta=-30°$, $-35°$, $-40°$, 90° ($H\|b$), and 0° ($H\|c$). Lines are guides for the eyes.

tional superconducting phase appears at three different pressures. Additionally, we would like to point out a possibility that a physical meaning of ±35° is related to a mathematical relation: $\tan^{-1}(\frac{7}{10})=34.99°$. However, in order to confirm whether or not the additional superconducting phase exists in the pressure-induced superconducting state in the $Sr_2Ca_{12}Cu_{24}O_{41}$, precise specific heat measurements at pressures above 3 GPa under magnetic fields are required for future study.

IV. CONCLUSION

In summary, we report the results of precise magnetotransport measurements of a hole-doped two-leg ladder $Sr_2Ca_{12}Cu_{24}O_{41}$ single crystal in fields applied along all three crystallographic axes. The clear difference in the resistive upper critical field $H_{c2}(T)$ is observed among these three directions, indicating that this system has a highly anisotropic superconducting ground state. Also, the absence of Pauli limit in H_{c2} parallel to the conducting ladder plane is found, which suggests either a strong spin-orbit scattering or spin-triplet pairing. Furthermore, it is implied, from measurements of resistivity versus angle of magnetic field in the *bc* plane, that an additional superconducting phase is stable below around 3 K only when the magnetic field is applied exactly along a certain direction that is ±35° from the ladder direction.

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