Electronic and magnetic properties of the electron-doped superconductor $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$

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Temperature-dependent magnetization $[M(T)]$ and specific heat $[C_p(T)]$ measurements were carried out on single crystal $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ ($T_c=16.5$ K). The magnetic anisotropy in the static susceptibility $\chi \equiv M/H$ is apparent not only in its magnitude but also in its temperature dependence, with $\chi_\perp$ for $\mathbf{H}\perp\mathbf{c}$ larger than $\chi_\parallel$ for $\mathbf{H}\parallel\mathbf{c}$. For both field orientations, χ does not follow the Curie-Weiss behavior due to the small energy gap of the $J=7/2$ multiplet above the $J=5/2$ ground-state multiplet. With increasing temperature, however, $\chi_\parallel(T)$ exhibits a broad minimum near 100 K and then a slow increase while $\chi_\perp(T)$ shows a monotonic decrease. A sharp peak in $C_p(T)$ at 4.7 K manifests an antiferromagnetic ordering. The electronic contribution, γ, to $C_p(T)$ is estimated to be $\gamma=103.2(7)$ mJ/mole Sm K^2. The entropy associated with the magnetic ordering is much smaller than $R\ln 2$, where R is the gas constant, which is usually expected for the doublet ground state of Sm^{+3}. The unusual magnetic and electronic properties evident in $M(T)$ and $C_p(T)$ are probably due to a strong anisotropic interaction between conduction electrons and localized electrons at Sm^{+3} sites.

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I. INTRODUCTION

After the discovery of high-temperature superconductivity in copper-oxide compounds,[1] a new class of superconducting compounds was found with the formula $Ln_{2-x}M_xCuO_4$ where Ln stands for Pr, Nd, Sm, and Eu, and M for Ce and Th.[2] These compounds have become a subject of intense study due to their peculiar physical properties, which are different from those of the other high-temperature cuprate superconductors. The Ln_2CuO_4 parent compound crystallizes in a tetragonal "T'-phase" structure containing CuO_2 planes in which the copper ions are surrounded by a square planar arrangement of oxygen ions, in contrast to the La_2CuO_4 parent compound which forms an orthorhombic "T-phase" structure at low temperature (below ≈ 500 K) containing CuO_2 planes in which copper ions are surrounded by an octahedral arrangement of oxygen ions.

The $Ln_{2-x}M_xCuO_4$ ($M=$Ce or Th) compounds have electrons as a charge carrier (n type) in forming superconducting Cooper pairs, in contrast to the related $La_{2-x}M_xCuO_4$ ($M=$Sr or Ba) compounds containing holes as a charge carrier (p type). The electron-doped compounds have the pressure dependence of T_c variation with negative $d\ln T_c/dP$, where P is pressure, while the hole-doped ones have positive $d\ln T_c/dP$.[3] Antiferromagnetic (AFM) ordering of the rare-earth ions in Ln_2CuO_4 has been found for Ln$=$Nd ($T_N\approx 1.7$ K), Sm ($T_N\approx 5.9$ K), and Gd ($T_N \approx 6.6$ K) while no magnetic ordering was observed for Ln $=$Pr and Eu.

The AFM ordering temperatures for Ln$=$Nd and Sm are lowered by substituting electron donor element (Ce^{+4} or Th^{+4} ions) for Ln^{+3} ions. The superconductivity appears within a narrow range of electron doping near $x=0.15$ with $T_c>T_N$ and coexists with the AFM state below T_N. The nature of the AFM transition was studied in terms of magnetization and specific heat measurements.[4,5]

The estimation of the entropy associated with magnetic ordering in Sm_2CuO_4 confirms the doublet ground state, expected by crystalline electric field splitting.[6] The electronic contribution to the specific heat, $\gamma\approx 82$ mJ/mole Sm K^2, in Sm_2CuO_4, however, is found to be much larger than those of the other T'-phase compounds. The large value of γ is suspected to be due to the existence of magnetic correlation much above T_N, making the evaluation of γ uncertain, but is not understood clearly yet.

For superconductivity, experimental determination of the order-parameter symmetry of n-type cuprate superconductors is critical in establishing a comprehensive understanding of the mechanism of superconducting pairing in cuprates. Recent experiments suggest that the dominant symmetry of the order parameter is of d-wave type.[7–9] In addition, the role of rare-earth magnetic moments interacting with a d-wave superconducting system of electrons opens up a new area of theoretical and experimental studies. So far only $Nd_{2-x}Ce_xCuO_{4-\delta}$ was extensively studied in which relatively weak moments strongly influence the temperature dependence of the penetration depth,[7,8] which helps to identify the order-parameter symmetry. It is a relevant, prerequisite research objective to investigate the electronic and magnetic properties in the normal state of electron-doped $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ in a single crystalline form. In this paper, the specific heat and magnetization data are presented for a study of the normal state properties of superconducting $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ compounds.

II. EXPERIMETAL DETAILS

Superconducting single crystals of $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ have been grown by a flux-based technique. A batch of about 40 g is prepared by mixing and grinding powders of Sm_2O_3 (99.9%), CeO_2 (99.99%), and CuO (99.99%) in the molar ratio of $(2-x):(2x):(7.2\sim13.4)$, respectively. The powders were pre-baked at 800−950 °C (for Sm_2O_3 and CeO_2), or at 400−600 °C (for CuO), to remove some volatile impurities. The mixed batch needs to be sintered at 900 °C and ground several times. It was soaked at 1000 °C for 10−20 h and heated to 1210 °C in air (300 °C/h). After a short soak for 1–3 h, the temperature was lowered to 1000 °C at a rate of 5–12 °C/h, and then to room temperature. As-grown crystals with typical size of $\sim1.5\times1\times0.03$ mm^3 were synthesized by this procedure. Superconductivity was induced by annealing and quenching in an inert gas; the initial raising rate of temperature was 5–10 °C/min (300–600 °C/h), and the soak time at 880 °C was 16 h. The quenching needs to be done within 30 min to preserve the high-temperature structure.

The grown single crystals of $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ are confirmed to be of the single phase of the Sm_2CuO_4 structure by measurements of powder x-ray diffraction of pulverized single crystals. The impurity phases of Cu_2O and Sm_2O_3, which are often found in polycrystalline samples, are not detected in the diffraction pattern. Temperature dependent static magnetization was measured by using a 7-T Quantum Design superconducting quantum interference device magnetometer (SQUID). In order to minimize the background signal from the sample holder a straw was used to hold the crystal. The typical background signal ($\approx10^{-7}$ emu) is found to be negligible compared with the sample signal ($\approx10^{-5}$ emu). The field-cooled (FCW) and zero-field-cooled (ZFC) data in the superconducting state were obtained on warming after the magnet was quenched. The specific heat measurements down to 1.2 K were made on the grown single crystal, using a time constant method (relaxation method) described in detail elsewhere.[10]

III. RESULTS AND DISCUSSION

The magnetization versus temperature $[M(T)]$ data in Figs. 1(a) and 1(b) show the flux expulsion (FCW) and magnetic shielding (ZFC) effects for $\mathbf{H}\parallel\mathbf{c}$ and $\mathbf{H}\perp\mathbf{c}$ in a $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ crystal for an external magnetic field $H=10$ G, respectively. The plots show typical superconducting diamagnetic signal for both field orientations, indicating bulk superconductivity. The much higher values of $M(T)$ for $\mathbf{H}\parallel\mathbf{c}$ than $\mathbf{H}\perp\mathbf{c}$ are due to the demagnetization field inside the sample, which is not corrected for actual real field for the measurements. The superconducting transition temperature T_c is found to be 16.5 K, the temperature at which more than 1% of superconducting volume fraction appears. It is noted that the superconductivity in $Nd_{2-x}Ce_xCuO_{4-\delta}$ appears only both in the very limited Ce concentration range of $x\approx0.15$ and in the reduced oxygen content of $\delta\approx0.07$.[4] The superconducting properties of $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ single crystal are quite similar to those of $Nd_{2-x}Ce_xCuO_{4-\delta}$ samples, indicating the apparent oxygen deficiency in our sample. In addition, the observed $T_c\approx16.5$ K and the broad superconducting transition in the magnetization under low magnetic fields are often found in the $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ samples, due to the partial occupancy of apical oxygen in T'-phase structure.[11] It should be noted that, recently, the microwave surface resistance measurement, which depends *neither* on electric percolation nature *nor* on magnetic shielding current, shows that the real T_c, clearly higher than the T_c determined above, exists without a measurable bulk Meissner effect in $Sm_{2-x}Ce_xCuO_{4-\delta}$ compounds.[12] So the T_c, which is determined in this study, is believed to be a lower bound of real T_c.

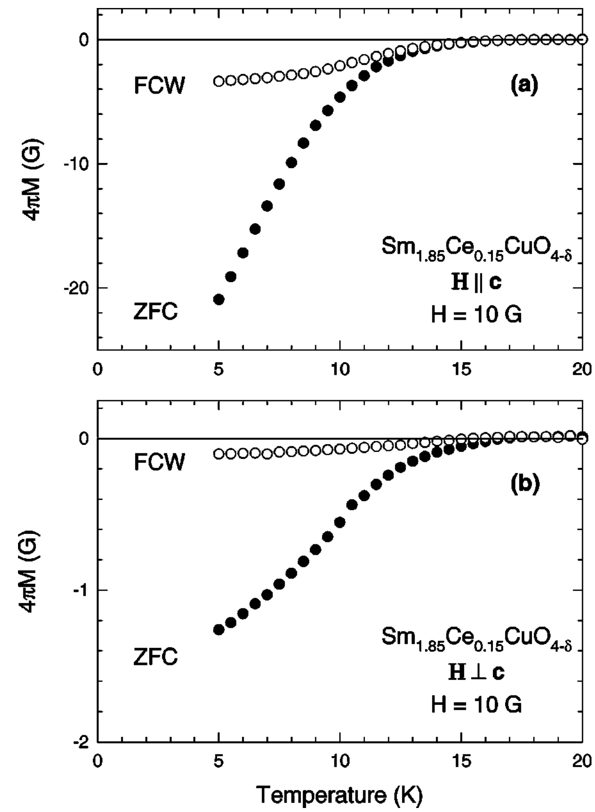

FIG. 1. Superconducting state volume magnetization M in an applied field $H=10$ G versus temperature of single crystal $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ for (a) $\mathbf{H}\parallel\mathbf{c}$ and (b) $\mathbf{H}\perp\mathbf{c}$: zero-field-cooled (ZFC) (dark circles) and field-cooled (FCW) (open circles) data taken on warming as shown.

Typical $M(H)$ isotherm data for $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ are shown in Figs. 2(a) for $\mathbf{H}\perp\mathbf{c}$ and 2(b) for $\mathbf{H}\parallel\mathbf{c}$ at several different temperatures for 0 G$\leqslant H\leqslant$70 kG. For both field orientations, the magnetization is linear in the whole applied field range for temperature above 50 K, except in the H <10 kG range at 5 K, where superconducting signals ap-

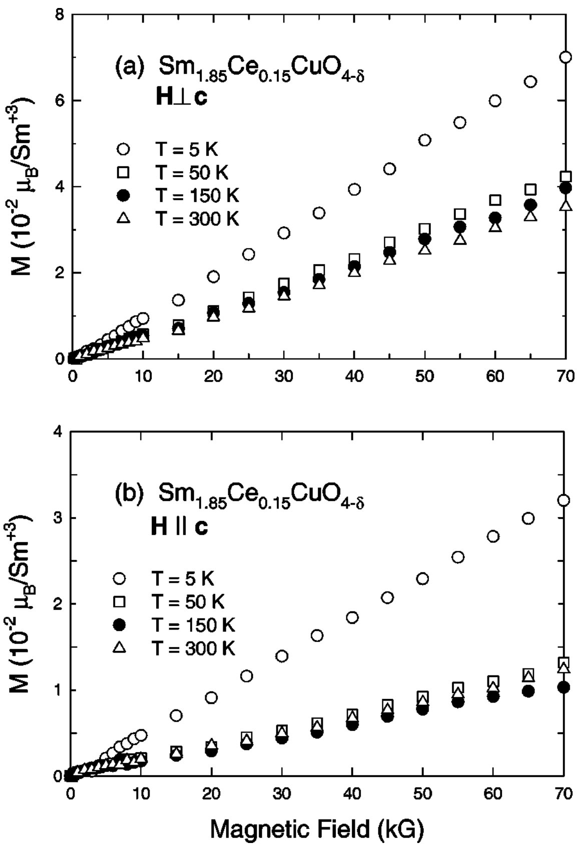

FIG. 2. Magnetization M versus applied magnetic field H of single crystal $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ at the indicated temperature: (a) $\mathbf{H}\perp\mathbf{c}$ and (b) $\mathbf{H}\|\mathbf{c}$.

pear. It is noted that the nonlinear behavior of magnetization, leading to a saturation of the Sm^{+3} magnetic moments, is not observed even at $T=5$ K and $H=70$ kG. The magnetic moment at this temperature and field is found to be 0.071 μ_B/Sm^{+3} and 0.032 μ_B/Sm^{+3} for $\mathbf{H}\perp\mathbf{c}$ and $\mathbf{H}\|\mathbf{c}$, respectively. Those values are much smaller than the theoretically expected value of $0.845\mu_B/Sm^{+3}$ for Hund's isolated Sm^{+3} ion $^6H_{5/2}$. It should be noted that the magnetizations for $\mathbf{H}\|\mathbf{c}$ manifest nonmonotonic behavior, i.e., $M(300\text{ K}) > M(150\text{ K})$ while those for $\mathbf{H}\perp\mathbf{c}$ decrease as temperature increases.

Figure 3 shows the temperature-dependent magnetic susceptibility $\chi(T)$ for $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ with $H=5$ kG perpendicular and parallel to the c axis and their powder average for 5 K$\leqslant T \leqslant$350 K. The large anisotropy in $\chi(T)$ between $\mathbf{H}\perp\mathbf{c}$ and $\mathbf{H}\|\mathbf{c}$ is quite clear and the temperature dependence for both field orientations clearly deviates from the typical Curie-Weiss behavior. In addition, the temperature dependences of $\chi(T)$ for both field orientations are also significantly different: with increasing temperature, $\chi_{\|}(T)$ for $\mathbf{H}\|\mathbf{c}$ shows a broad local minimum around 100 K and a slow increase whereas $\chi_{\perp}(T)$ for $\mathbf{H}\perp\mathbf{c}$ shows a monotonic decrease. This temperature dependence of $\chi(T)$ is consistent with the $M(H)$ isotherm data in Figs. 2(a) and 2(b). The similar $\chi(T)$ of non-Curie-Weiss behavior is found in the magnetization of Sm^{+3} ions and ascribed to the size of J multiplet comparable to k_BT in Sm^{+3} Hund's ground state of $J=5/2$.[13] Thus, the van Vleck contribution due to the higher level of $J=7/2$ should be considered to account for the observed susceptibility. The observed magnetic susceptibility is described according to the standard formula of

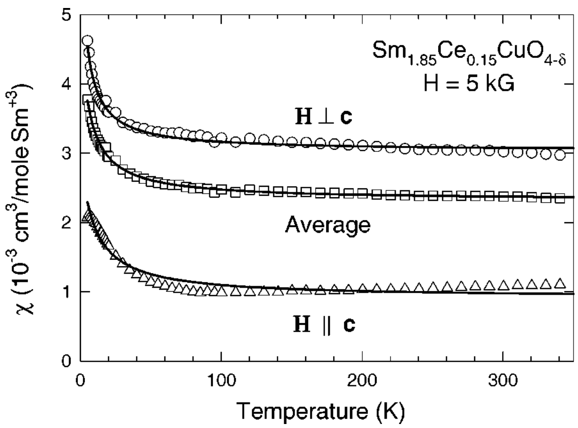

FIG. 3. Anisotropic magnetic susceptibility χ versus temperature T of single crystal $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ for 5 K$\leqslant T \leqslant$350 K for $\mathbf{H}\perp\mathbf{c}$, $\mathbf{H}\|\mathbf{c}$, and powder average. Fits to Eq. (1) in the text are shown by the solid curves for each field orientation.

$$\chi(T)=N_A\left[\frac{\mu_{\text{eff}}^2}{3k_B(T-\Theta)}+\frac{20\mu_B^2}{7\Delta E}\right], \qquad (1)$$

where the first term is a Curie-Weiss contribution from the $J=5/2$ ground state multiplet, and the second one is a temperature independent van Vleck susceptibility due to coupling of the $J=5/2$ ground state multiplet with the $J=7/2$ multiplet at an average energy ΔE above the ground state. The best fits for the data of $\mathbf{H}\|\mathbf{c}$, $\mathbf{H}\perp\mathbf{c}$, and powder average are plotted by solid lines as shown in Fig. 3. The results are unsatisfactory for both field orientations but apparently quite good for the powder-average case. From the fitting results of the powder average data, the splitting ΔE, the effective moment μ_{eff}, and the Curie-Weiss temperature Θ are extracted to be 466 K, $0.36\mu_B$, and -6.4 K, respectively. The value of ΔE is smaller than that of Sm_2CuO_4, (≈ 1150 K),[5] which is probably due to the doping of electrons by Ce^{+4} ions and the interaction between the localized and the doped electrons.

A particularly interesting feature in Fig. 3 is that the susceptibility for $\mathbf{H}\|\mathbf{c}$ reaches a minimum and then increases slowly as the temperature increases still further, which is similar to that in Sm due to the small interval between $J=5/2$ and $J=7/2$ multiplets. This minimum point is not observed for $\mathbf{H}\perp\mathbf{c}$ and the powder average, which show a monotonic decrease with increasing temperature. One of the possible scenarios for this remarkable anisotropy is that the splitting of J multiplets has angular dependence. It is conjectured that this can be caused by the non-negligible anisotropic hybridization of conduction electrons with the localized Sm^{+3} ions and its angular dependence. This unusual hybridization probably has a close relation with the relatively large γ (≈ 82 mJ/mole Sm K^2) value in Sm_2CuO_4. The

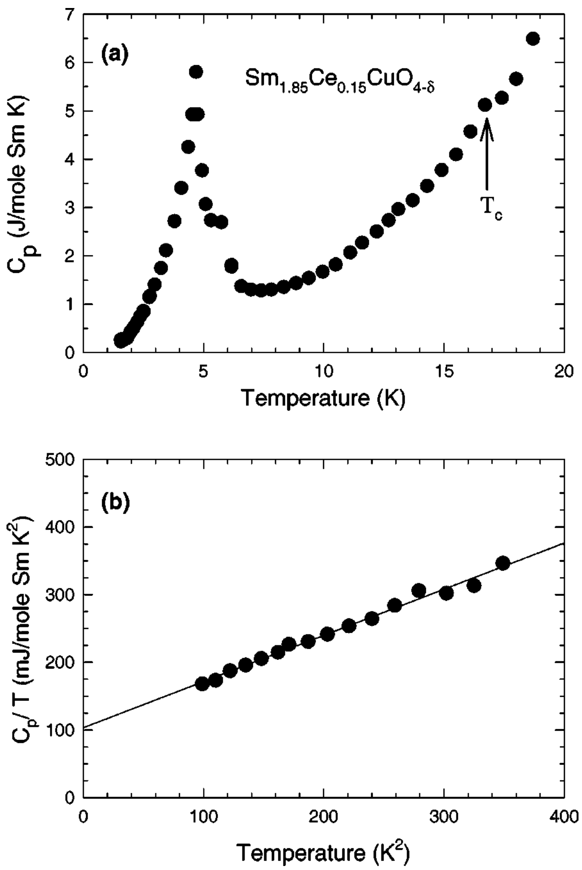

FIG. 4. (a) Specific heat C_p versus temperature T of single crystal $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ for 1.5 K$\leqslant T \leqslant$19.0 K. (b) C_p versus T^2.

large value of γ of electron doped $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ is confirmed by our study of specific heat measurements (see below).

Another possibility for the slow increase with the temperature is an opening of pseudo-spin-gap which is observed in the hole-doped $La_{2-x}M_xCuO_4$ (M=Sr) system. The anisotropic behavior may be caused by Sm spins. If the anisotropic susceptibility from Sm spins is corrected in some way, isotropic temperature dependence would be observed. It will be nice to measure the system free from large rare earth spins to check this possibility. Recently, in the electron doped system, the pseudogap state is found in the antiferromagnetic as well as in the superconducting samples.[14]

The temperature dependent specific heat, $C_p(T)$, for $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ is plotted in Fig. 4(a). Clear evidence of a phase transition in $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ is given by the sharp peak at $T=4.7$ K and the superconducting transition is seen near $T_c \approx 16.5$ K as a slight jump of C_p, which is consistent with the T_c from low field magnetization. The data at $T=5.7$ K, which is level off the measured data, is not understood yet and probably sample dependent (or a measurement error). It was shown that a phase transition in Sm_2CuO_4 at $T\approx 6$ K is attributed to an AFM transition from the $M(T)$ and $C_p(T)$ measurements.[4] Thus, the observed transition in $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ is also of AF nature and T_N is shifted to a lower temperature with doped charge carrier (electrons).

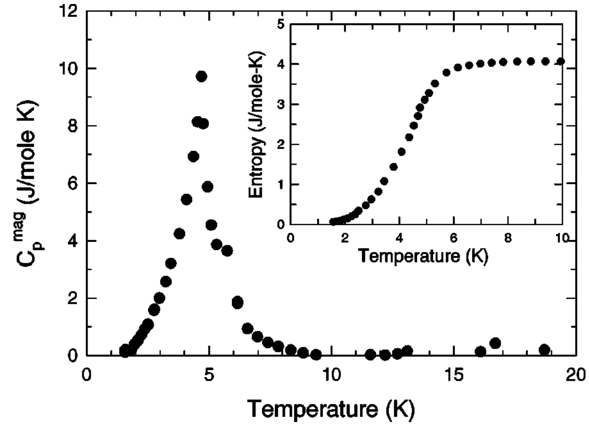

FIG. 5. Magnetic specific heat ($C_p{}^{\mathrm{mag}}$) versus temperature T of single crystal $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$, $C_p{}^{\mathrm{mag}}=C_p-C_p{}^{\mathrm{NM}}$ $(=\gamma T+\beta T^3)$ (see text). Inset: entropy associated with the magnetic transition versus temperature.

In order to separate the magnetic and nonmagnetic contributions to C_p, the data for 10 K$\leqslant T\leqslant$18 K is fitted to the equation

$$C_p^{\mathrm{NM}}(T)=\gamma T+\beta T^3, \tag{2}$$

where the linear and the cubic terms correspond to the electronic and lattice contributions to the specific heat, respectively. Here it is assumed that the contribution of superconducting transition near $T\approx 16.5$ K is so small that it will not affect significantly the C_p from nonsuperconducting components. The $C_p{}^{\mathrm{NM}}(T)$ for 10 K$\leqslant T\leqslant$18 K from Fig. 4(a) is plotted again with $C_p{}^{\mathrm{NM}}(T)/T$ versus T^2 in Fig. 4(b) together with the fitting values (solid line), which shows nice agreement between the data and Eq. (2). It is found that γ = 103.2 (7) mJ/mole Sm K^2 and $\beta=0.7$ (1) mJ/mole Sm K^4, yielding the Debye temperature $\Theta_D\approx 219$ K from the relation of $\Theta_D \propto (n/\beta)^{1/3}$, where n is the number of atoms in a formula unit. Although the above equation for the specific heat is valid for temperatures below $\Theta_D/50$ in usual metals, the equation often works quite well for temperatures below $\Theta_D/10$ within an error of a few percent, which is basically our temperature range.[15]

The observed value of $\gamma=103.2$ (7) mJ/mole Sm K^2 is significantly larger than those found in other Ln_2CuO_4 compounds, 0 ± 10 mJ/mole Nd K^2 for Ln=Nd, 1.3 ± 0.1 mJ/mole Pr K^2 for Ln=Pr.[16] For $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$ compound, which has the highest superconducting transition temperature among electron-doped superconductors, the value of γ is enhanced to $\approx$29 mJ/mole Nd K^2.[4] It is natural to judge that the enhanced γ is due to the doped electrons. Even for Sm_2CuO_4, the γ value was previously found to be exceptionally large ($\approx$82 mJ/mole Sm K^2).[16] It was speculated that the effects of magnetic correlation exist well above $T_N\approx 5.9$ K, thereby making accurate determination of γ difficult. Our estimated γ for $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ is still quite large even though T_N is now lowered to be 4.7 K. It is not clear now, however, what the origin of the large value of γ in the superconducting state is.

The contribution of magnetic correlation to the measured $C_p(T)$ is calculated as $C_p{}^{\mathrm{mag}}(T)=C_p(T)-C_p{}^{\mathrm{NM}}(T)$, where the extrapolation of $C_p{}^{\mathrm{NM}}(T)$ for low temperature with the constants determined above is used, and is plotted in Fig. 5. The entropy associated with the magnetic transition is calculated from the $C_p{}^{\mathrm{mag}}(T)$ and its temperature dependence is plotted in the inset of Fig. 5. The magnetic entropy saturates rapidly above T_N to be ≈ 4.1 J/mole K, indicating that the transition is driven by localized electrons. The accumulated entropy is clearly smaller than $1.85R\ln 2$, however, where R is the gas constant, which is the usual value of a doublet ground state of Sm^{+3}.[6] It is interesting to see what temperature would be needed to get an entropy that would make the total system entropy $1.85R\ln 2$. By setting $1.85R\ln 2 = S_{\mathrm{anomaly}}+\gamma T^* = 4.2+\gamma T^*$, the T^* is found to be ≈ 33 K. It was reported that the magnetic entropy associated with a magnetic transition is significantly reduced if the magnetic transition is due to itinerant heavy fermionic electrons, which is analogous to a BCS type of transition.[17] Thus, the reduced entropy can be explained by the fact that itinerant electrons with heavy effective mass are involved in the transition. This explanation is also consistent with the anisotropic temperature-dependent behavior of magnetization and the enhanced electronic specific heat contribution.

IV. SUMMARY

Single crystals of superconducting $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ compounds are studied in terms of magnetization and specific heat measurements. The largest difference in susceptibility, so far reported, between $\mathbf{H}\|\mathbf{c}$ and $\mathbf{H}\perp\mathbf{c}$ is found and the temperature dependences for both field orientations do not follow the Curie-Weiss behavior due to the small energy gap of the $J=7/2$ multiplet above the $J=5/2$ ground state. With increasing temperature, $\chi_{\|}(T)$ for $\mathbf{H}\|\mathbf{c}$ exhibits a broad local minimum around $T=100$ K and a slow increase while $\chi_{\perp}(T)$ for $\mathbf{H}\perp\mathbf{c}$ shows a monotonic decrease. The specific heat data show a sharp peak at $T=4.7$ K, which is due to an AF transition. The estimated γ value of electronic contribution is enhanced with electron doping and clearly larger than those reported so far. The entropy associated magnetic transition is obviously smaller than the expected one of a doublet ground state. The peculiar features found in this paper in $Sm_{1.85}Ce_{0.15}CuO_{4-\delta}$ seem to be related to the conduction electrons, which are strongly interacting with the localized electrons. It is necessary to study more in experiments and in theory to understand the magnetic and electronic properties and, further, the mechanism of superconductivity in electron doped superconductors.

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