PHYSICAL REVIEW B VOLUME 43, NUMBER 4 1 FEBRUARY 1991

Positive Hall coefficient observed in single-crystal $Nd_{2-x}Ce_xCuO_{4-\delta}$ at low temperatures

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(Received 3 October 1990)

The Hall coefficient R_H has been measured in superconducting single crystals of $Nd_{2-x}Ce_xCuO_{4-\delta}(x \sim 0.15)$. Although R_H is sample dependent in sign above ~ 100 K, it increases steeply to *positive* values in all crystals studied below ~ 80 K. R_H remains T (temperature) dependent at 2 K, in contrast to the resistivity ρ_a which saturates to a constant below 30 K. Using a two-band model, we account for the observed profiles of R_H vs T and ρ_a vs T. The analysis reveals that the scattering processes for the electronlike and holelike bands have vastly different temperature scales. The hole-scattering rate remains T dependent to 2 K. The question whether the hole or the electron quasiparticles are responsible for the superconductivity in $Nd_{2-x}Ce_xCuO_{4-\delta}$ is discussed.

I. INTRODUCTION

High-temperature superconductivity[1] was discovered by Bednorz and Muller[2] in the T-phase system, $La_{2-x}Ba_xCuO_4$, which is comprised of parallel CuO_2 layers. Each Cu ion sits in an octahedral cage formed by the oxygen ions. The related T' phase, $Nd_{2-x}Ce_xCuO_{4-\delta}$, is characterized by the absence of the apical oxygen between adjacent CuO_2 layers. Takagi, Uchida, and Tokura (TUT)[3] reported that superconductivity appears in this compound, in a narrow range of Ce concentration $(0.15 < x < 0.17)$, when the samples are annealed in a reducing atmosphere. The reduction process, together with the valency of the dopant Ce, suggest that the compound may be the first example of an oxide superconductor in which the superconducting carriers are electronlike, rather than holelike. Clearly, the existence of an n-type counterpart to the hole superconductors has significant implications for many theoretical models of high-temperature superconductivity. Hall measurements[3] on ceramic samples showed that the Hall coefficient R_H is *negative,* in apparent agreement with the inferred sign of the carriers, and only weakly temperature dependent. As x is varied, R_H (measured at 80 and 300 K) is observed to become positive when x exceeds 0.17. The variation of R_H versus x is roughly analogous to the behavior found in the T-phase system (except for the opposite sign).[4,5]

Subsequent transport measurements on a single-crystal specimen of $Nd_{2-x}Ce_xCuO_{4-\delta}$ by Hidaka and Suzuki (HS)[6] showed that the in-plane resistivity ρ_a is metallic. HS also reported R_H to be negative above 50 K. At low T, ρ_a is found to saturate to a T-independent value (when an 8-T field is applied normal to the basal plane to suppress the superconductivity). Such a low-temperature impurity-dominated regime is not observed in the hole superconductors, $La_{2-x}Sr_xCuO_4$ $(0.12 < x < 0.25)$, $YBa_2Cu_3O_7$ (Y-Ba-Cu-O), and $Bi_2Sr_2CuO_6$ (Bi 2:2:0:1) and $Bi_2Sr_2CaCu_2O_8$ (Bi 2:2:1:2).[7] $Nd_{2-x}Ce_xCuO_{4-\delta}$ appears to differ significantly from the hole superconductors in its normal state R_H and ρ_a.[7]

We have performed Hall measurements on single crystals of $Nd_{2-x}Ce_xCuO_{4-\delta}$ that display a sharp resistive transition into the superconducting state in zero field. Although the sign of R_H is sample dependent at room temperature we find that R_H increases to large, positive values below ~ 80 K in all samples. Analyzing the transport results in terms of a general two-band model we infer that a pocket of hole-type quasiparticles coexists with a larger band of electrons. Whereas the scattering rate of the electrons saturates below 30 K, that of the holes remain T dependent down to 2 K. The findings clarify the observed T dependence of both R_H and ρ_a. They also cast doubt on the claim that negatively charged carriers are responsible for the superconductivity observed in $Nd_{2-x}Ce_xCuO_{4-\delta}$.

II. EXPERIMENTAL DETAILS AND RESULTS

Single crystals were prepared in a platinum crucible using a 20-g charge of Nd_2O_3 and CeO_2.[8] The crucible was heated to 1500 °C and then slow-cooled to 1100 °C. Shiny crystals as large as $2 \times 2 \times 0.05$ mm^3 were found within cavities in the solidified flux. (To obtain crystals with Ce content between 0.12 and 0.18, excess CuO is used as a flux to lower the melting temperature to under 1300 °C.) By varying the proportion of CeO_2 in the charge we obtained a series of crystals which were studied to determine their superconducting behavior versus Ce content. The microstructure and Ce content were analyzed using scanning electron microscopy (SEM) and energy-dispersive x-ray analysis (EDX). To improve the precision of the EDX analysis, we calibrated the Ce peak against standards based on $Nd_{2-x}Ce_xCuO_{4-\delta}$ ceramic samples. By x-ray diffraction, the c-axis parameter and cell volume V_{cell} were determined to be 12.083 Å and 188.03 Å^3, respectively. The EDX scans show that the

Ce dopants are uniformly distributed over the face of the crystals, to a resolution of ~1 μm. Extensive thermogravimetry (TGA) measurements were used to determine the oxygen loss and gain profiles versus temperature. In crystals with $x=0.16$, the maximum oxygen content y is 4.01 per formula unit. When the crystals are reduced in nitrogen at 900 °C for 24 hr, y decreases to 3.97 ($\delta=0.04$). As found by TUT, superconductivity appears in a narrow range of x only after the reducing treatment. Using a 5-G field in a superconducting quantum interference device (SQUID) magnetometer, we determined the Meissner fraction to be 60% for $x=0.14$ and 40% for $x=0.16$ (with $y\sim3.97$ in both cases). Further details appear in Tarascon *et al.* [8]

Although crystals used in the present study were not analyzed individually, the Ce and oxygen contents determined from measurements on crystals from the same crucible have the values $x=0.15\pm0.005$ and $y=3.97$. The Meissner fraction was 50–60% in this batch of crystals. We further culled this batch for crystals with the sharpest transitions by performing four-probe van der Pauw resistivity measurements (over 20 crystals were studied). The four crystals with the sharpest resistive transitions were selected for the Hall measurements (the transition temperature T_c in zero field lies between 19 and 22 K in the four crystals). Contacts with resistance under 2Ω were made to the crystals using solder with the composition 90% In, 10% Ag. The Hall and magnetoresistance studies were performed by sweeping the field **H** (parallel to **c**) in a 20-Tesla Bitter magnet or in an 8-T superconducting magnet. The dc current density, applied within the basal plane *a-b*, was kept under 30 A/cm^2.

In superconducting crystals, the in-plane normal-state resistivity ρ_a has a metallic profile, as reported by HS,[6] with a room-temperature value lying between 0.5 and 1 mΩ cm (Fig. 1). By using an intense field (18 T) to suppress the superconductivity we find that, within our resolution, ρ_a is approximately T independent below ~30 K (open circles in Fig. 1). The residual resistivity ratio (RRR) varies between 2.5 and 3 in our samples (compared with ~6 in HS[6]), which implies a higher degree of disorder in our samples. Measurements of the out-of-plane resistivity ρ_c have been performed on our crystals using the Montgomery method. We obtain values of the anisotropy ρ_c/ρ_a (6000 to 10 000) quite comparable to that reported in Bi 2:2:0:1 and Bi 2:2:1:2.[9]

In Fig. 2 we show the suppression of the superconductivity by plotting ρ_a (sample 2) against H. Finite resistivity caused by vortex motion appears at a reasonably sharp threshold field. The suppression of superconductivity also occurs at a well-defined upper critical field H_{c2}. Above H_{c2} at 1.2 K (~6.5 T sample 2), there is a slight increase of ρ_a with H (approx 2% at 20 T). The *positive* magnetoresistance is apparent as well in the ρ_a versus T plot of HS taken at 8 T.[6]

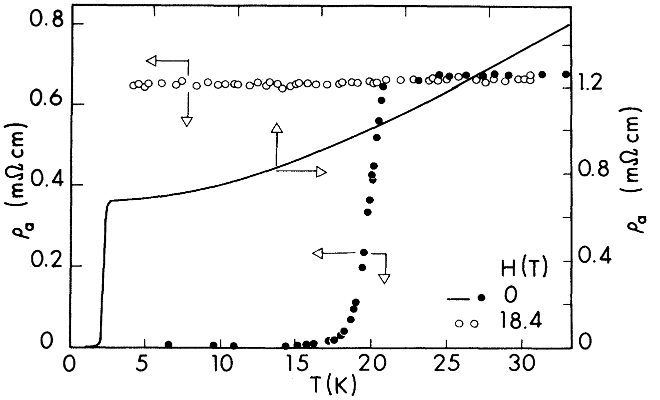

FIG. 1. Variation with temperature of the in-plane resistivity ρ_a of $Nd_{2-x}Ce_xCuO_{4-\delta}$ single crystal (sample 1), in zero field (solid circles), and in a field of 18.4 T parallel to **c** (open circles). The normal state ρ_a below 20 K is independent of T within our resolution. The solid line represents ρ_a vs T in zero field up to 300 K.

The Hall coefficient was measured in four crystals. The variation of the Hall voltage V_H with H in sample 1 is shown in Fig. 3 at several temperatures. At 2.1 K, V_H in the flux-flow regime becomes finite at ~2 T, and increases monotonically between 1 and 6 T. At the upper critical field $H_{c2}\approx6.2$ T, V_H shows a distinct break in slope when the sample becomes normal. We note that at low T, $R_H(H)=V_H/wJB$ is positive (holelike) in both the flux-flow and normal states (w is the sample width). [In the rest of the paper we take R_H to mean $R_H(0)$.] A slight deviation from linearity in V_H is apparent in the curves at low T. This is brought out more clearly by plotting $R_H(H)$ against H (Fig. 4). Whereas above 90 K, $R_H(H)$ is relatively insensitive to H up to 20 T, at 2.1 K it decreases by ~12% when H reaches 20 T. However, the most interesting feature of $R_H(H)$ is its strong temperature dependence below 90 K. The T dependence of $R_H(0)$ is displayed in Fig. 5 for the four samples studied. Above 20 K $R_H(0)$ is measured directly. Below 20 K we

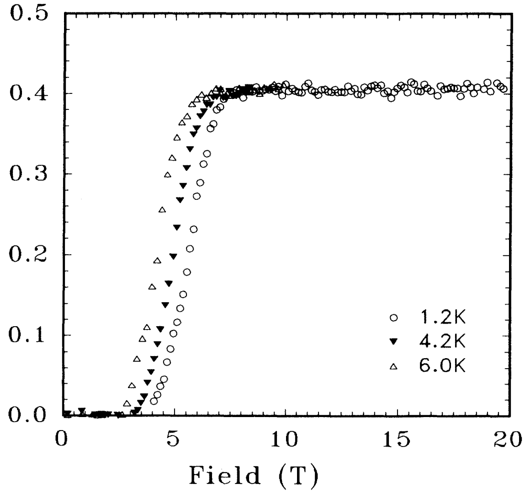

FIG. 2. Variation of ρ_a with field (**H**‖**c**) in $Nd_{2-x}Ce_xCuO_{4-\delta}$ (sample 2). In the normal state ($H>6.5$ T), a small *positive* magnetoresistance is observed.

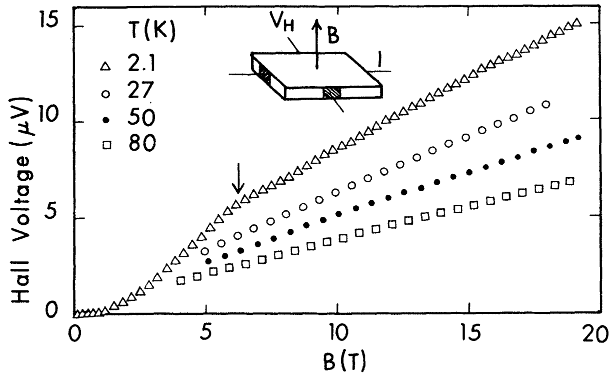

FIG. 3. The field dependence of the Hall signal V_H in single crystal $Nd_{2-x}Ce_xCuO_{4-\delta}$ (sample 1) at four temperatures ($I = 3$ mA). At 2.1 K (open triangles) the onset of vortex motion occurs at 1.5 T. (The flux-flow Hall effect is positive.) Suppression of the superconducting state when B exceeds $H_{c2} \sim 6.2$ T is indicated by the arrow. At higher temperatures data below ~ 5 T are deleted for clarity. At all T, saturation of V_H is not observed for fields up to 20 T.

estimate $R_H(0)$ by fitting the curves in Fig. 4 to a fourth-order polynomial in H and extrapolating to $H = 0$. In samples 1–3, $R_H(0)$ is positive at all T below 300 K, and increases monotonically with decreasing T.[10] For T above 80 K, the behavior of $R_H(0)$ in sample 4 (negative, with a broad minimum near 140 K) is quite similar in sign and magnitude to the polycrystalline sample of TUT.[3] However, at temperatures below 80 K, R_H in sample 4 changes sign and rises steeply to positive values comparable to those in samples 1–3. The T_c (in zero H) of all these samples exceeds 19 K.

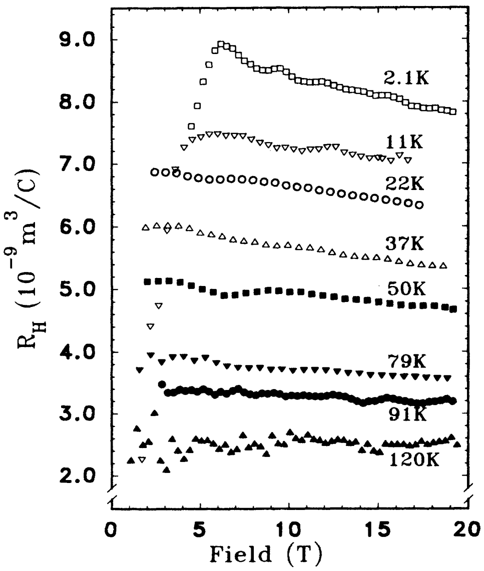

FIG. 4. The field dependence of the Hall coefficient $R_H(H)$ at temperatures 2.1 to 120 K (sample 1). At 2.1 K, $R_H(H)$ decreases by $\sim 11\%$ at 20 T.

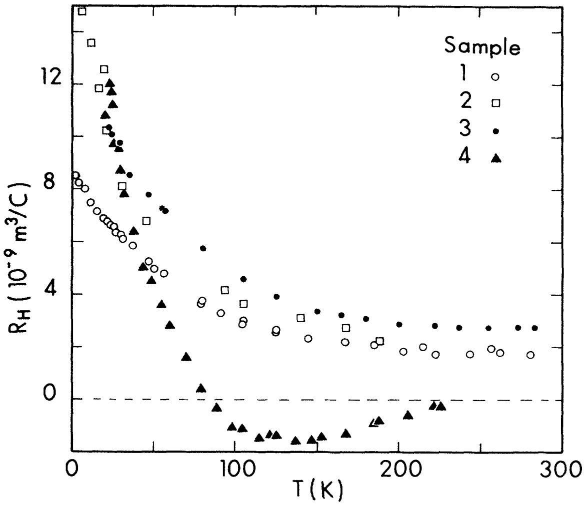

FIG. 5. Temperature dependence of the Hall coefficient $R_H(\mathbf{H} \| c)$ in four superconducting crystals of $Nd_{2-x}Ce_xCuO_{4-\delta}$. In sample 4, R_H is negative above 80 K. However, in all samples, R_H becomes positive (holelike) and strongly T dependent below ~ 80 K. [The sign of R_H in sample 1 (open circles) was erroneously reported to be negative in Ref. 10. That data and newer results from the same crystal are combined here.]

III. ELECTRON AND HOLE QUASIPARTICLES

Unlike the situation in the hole-type superconductors, Y-Ba-Cu-O, Bi 2:2:0:1, and Bi 2:2:1:2, the transport properties of $Nd_{2-x}Ce_xCuO_{4-\delta}$ present strong evidence for the coexistence of two bands with opposite charges (for x within the narrow superconductivity range). First, in several samples, R_H is observed to change sign as a function of temperature (see our sample 4). A change in sign also has been observed in some superconducting ceramic and thin-film samples of $Nd_{2-x}Ce_xCuO_{4-\delta}$ cooled to sufficiently low T.[11] Second, R_H measured at room temperature (R_H^{RT}) is very sensitive to changes in x in the vicinity of the superconductivity window ($0.15 < x < 0.17$).[3] Thus, given the steepness of R_H^{RT} versus x as x crosses this window, it is not surprising that three of our crystals show a positive R_H^{RT} while one shows a negative R_H^{RT}. Indeed, in the phase-diagram reported by TUT,[3] R_H^{RT} changes sign very close to the large-x edge of the window (0.17). While the general features of phase-diagram are qualitatively correct, we argue that the sign of R_H alone (at any reference T) provides a poor criterion for justifying n-type as opposed to hole-type superconductivity. The steep variation of R_H with x (and T) and the narrow width of the superconductivity window make this a precarious business at best. (For instance, if the reference T is chosen closer to T_c, say 25 K, the window would fall unambiguously on the "hole" side.)

A sharper criterion for the sign of the relevant superconducting carriers requires a qualitative understanding of the trend of R_H. The sensitivity of its sign to both x and T indicates that R_H^{RT} is largely determined by two competing bands, i.e., a hole band coexists with an electron band in the vicinity of the window. Yet another indication of two bands is the great disparity between the temperature scales of R_H and ρ_a. The resistivity data seem to show only T-independent disorder-potential scattering present below 30 K. However, the persistent increase in R_H even for $T \ll 30$ K indicates the existence of a different T-dependent scattering mechanism that operates down to 2 K. The second mechanism presumably affects a second band of carriers.

Quite generally, R_H of a two-band system is given by

$$R_H=(\sigma_{H1}+\sigma_{H2})/B(\sigma_1+\sigma_2)^2 , \tag{1}$$

where σ_i and σ_{Hi} are, respectively, the diagonal and Hall conductivity elements of band i. For definiteness we take band 1 (2) to be electronlike (holelike), i.e., $\sigma_{H1}<0$, and $\sigma_{H2}>0$. It is possible to account for the major features of R_H and ρ_a without making specific assumptions about either band.[12] [In a two-dimensional (2D) system, a geometrical representation[13] for σ_{Hi} may be used to evaluate R_H. There are also topological constraints on the Fermi surface that may be exploited. For example, there are no open orbits in a 2D crystal with fourfold symmetry.] σ_{Hi} is the product $\sigma_i \tan\theta_{Hi}$ (θ_{Hi} is the Hall angle), so its T dependence follows that of the relaxation time τ_i raised to some positive integer p ($p=2$ in Drude theory). Matthiessen's rule for both bands implies

$$1/\tau_i=1/\tau_i^0+1/\tau_i^{\text{in}} \quad (i=1,2) , \tag{2}$$

where $1/\tau_i^0$ and $1/\tau_i^{\text{in}}$ are the scattering rates due to (disorder) potential scattering and inelastic processes, respectively. The corresponding T independent and T-dependent parts of the resistivity will be called ρ_i^0 and ρ_i^{in}, respectively, i.e., Eq. (2) is equivalent to $1/\sigma_i=\rho_i^0+\rho_i^{\text{in}}$.

In the low-temperature regime ($T<30$ K), R_H is observed to be positive and to increase steeply with decreasing T, but the total resistivity $\rho_a=1/(\sigma_1+\sigma_2)$ remains nominally T independent. Rewriting Eq. (1) as $R_H B/\rho_a^2=\sigma_{H2}-|\sigma_{H1}|$, we see that σ_{H2} must also increase steeply, but $|\sigma_{H1}|$ may be either T independent, or increase (less steeply than σ_{H2}). The second option would require both τ_1 and τ_2 to increase with decreasing T, in contradiction with the observed constancy of ρ_a. Therefore, the only possibility is that τ_1 is T independent in this regime, *and* that σ_1 dominates σ_2 (so that ρ_a is hardly affected by the increase in τ_2^{in}). Thus, the two-band model is consistent with the measurements provided:

$$\sigma_{H2} \gg |\sigma_{H1}| , \tag{3a}$$

$$\sigma_2 \ll \sigma_1 \quad (T<30 \text{ K}) . \tag{3b}$$

[Actually, a less restrictive inequality suffices in place of Eq. (3b). We only need the temperature-dependent part of $1/\sigma_2$ to be small compared with the sum of σ_1 and σ_2, i.e., $\rho_2^{\text{in}} \ll (\rho_2^0)^2(\sigma_1+\sigma_2)$.]

Adopting Eqs. (3), we may understand the data on R_H and ρ_a as follows. A pocket of holes coexists with a larger band of electrons. At all T, the conductivity of the electron band dominates that of the hole pocket. However, the Hall conductivities (determined by the respective mean free paths l_i) are comparable at high T. Thus, the room temperature R_H is small and sensitive to the relative magnitudes of l_1 and l_2, i.e., R_H may be of either sign, as we observe. When T decreases below 30 K, the scattering rate of the electron band saturates because disorder scattering dominates ($1/\tau_1 \sim 1/\tau_1^0$), whereas that of the holes continues to decrease down to 2 K ($1/\tau_2<1/\tau_2^0$). As a result, the Hall current of the holes dominates that of the electrons, and R_H increases to large positive values [see Eq. (1)]. [These inferences have been confirmed by fitting the data to a two-band model. The calculation, reported elsewhere,[12] shows that $1/\tau_2$ is actually *linear* in T (plus a constant term $1/\tau_2^0$) down to 2 K.]

IV. DISCUSSION OF TWO-BAND ANALYSIS

Our central conclusion is that the anomalous inelastic scattering ($1/\tau_2^{\text{in}}$) that persists to 2 K should be identified with the holes, while the scattering rate $1/\tau_1$, which saturates near 30 K, is that of the electrons. We discuss the electronlike band (band 1) first. Since σ_1 dominates the total conductivity, the behavior of $1/\tau_1$ may be inferred from ρ_a. The RRR in $\text{Nd}_{2-x}\text{Ce}_x\text{CuO}_{4-\delta}$ is highly variable among single crystals (2 to 6), so the scattering rate $1/\tau_1^0$ is sensitive to the degree of disorder present. Although ρ_a becomes T independent only below ~ 30 K, deviation from the high-temperature linear behavior is observable as high as 150 K. The ρ_a versus T profile is qualitatively different from that in the hole superconductors, but similar to what is seen in conventional highly disordered metals. The infrared reflectivity $R(\omega)$ is also dominated by the electron band. Although $R(\omega)$ measured on these crystals is comparable in size with that of Y-Ba-Cu-O in the range 1000–10 000 cm^{-1}, the $R(\omega)$ versus ω profile fits much better to a Drude model than in the case of Y-Ba-Cu-O (ω is frequency).[14] The fit yields a plasma frequency of 19 700 cm^{-1} or a density of $(4.4\times10^{21})(m_1^*/m_0)\text{cm}^{-3}$ (m_1^*/m_0 is the effective mass ratio in the electron band). Hence, both ρ_a and $R(\omega)$ suggest that the electron band is comprised of weakly correlated quasiparticles that behave much like carriers in a conventional disordered metal. The observation of a positive, classical magnetoresistance below 4 K (for $H>H_{c2}$) also suggests that the electron band is quite conventional: The hole superconductors have a small *negative* magnetoresistance.

The hole band is much more interesting. By the analysis, its inelastic scattering rate is observable to 2 K. While a T-dependent resistivity and R_H may be observed down to rather low T in conventional metals [e.g., 6 K in pure bismuth metal with mobilities $\sim 10^7$ cm^2/Vs (Ref. 15)], exceptionally high purity samples are required (for comparison, the *effective* Hall mobility here $\mu_H=R_H/\rho_a$ varies from 2.5 at 300 K to 25 cm^2/Vs at 4.2 K). What is remarkable here is that the electron-band resistivity shows that a high degree of disorder exists, yet the hole band displays a scattering rate $1/\tau_2$ that remains T dependent at 2 K. The disparity between $1/\tau_1$ and $1/\tau_2$ presents, within the same sample, clear evidence that the hole quasiparticles experience an inelastic scattering mechanism qualitatively different from those of the electrons. The origin of the hole inelastic scattering is unlikely to be due to phonons.[16] (Otherwise, $1/\tau_2$ should also saturate to a constant below 30 K, like $1/\tau_1$). The absence of saturation at low T is similar to the resistivity profile observed in Bi 2:2:0:1, and suggests that the holes may, in fact, be the carriers responsible for superconductivity in $Nd_{2-x}Ce_xCuO_{4-\delta}$. The electron band may become superconducting by proximity with the holes, but is of secondary importance. These results present evidence against the case for n-type superconductivity. We are currently investigating how the hole density (as revealed by the positive R_H tail at low T) correlates with the disappearance of superconductivity.

V. MAGNETIC SKEW SCATTERING?

In magnetic systems, skew scattering[17] between conduction electrons and local moments leads to an extra contribution R_S to the Hall coefficient. Theoretical models are most well developed for systems in which interaction between local moments can be neglected (e.g., in the paramagnetic phase of the ferromagnets, in the high-T phase of the mixed-valence systems,[18] and in systems with low concentrations of rare-earth impurities.[19]) In this regime, the susceptibility χ increases monotonically with decreasing T. The scattering events are all independent, and R_S is observed to be proportional to χ.[17,20] In both the hole superconductors and the data reported here, R_H increases strongly as T decreases. Presumably, if magnetic skew scattering is to provide an explanation of R_H versus T in the high-T_c oxides, this is the regime (negligible interaction between the moments) that would be the most relevant. However, when the Zeeman energy of the spins dominates the thermal energy ($g\mu B \gg k_B T$, where g is the g factor, μ the Bohr magneton, and k_B the Boltzmann constant), both χ and R_S saturate. Thus, a key signature of magnetic scattering in paramagnetic systems is the saturation of the anomalous part of V_H when H exceeds the field $H_s = k_B T / g\mu$. For a g factor equal to 2, H_s equals 1.43 T at a temperature of 2 K.

However, it is now known that this signature is not observed in the high-T_c cuprates. Suzuki[21] finds that, in $La_{2-x}Sr_xCuO_4$, the Hall voltage V_H at 52 K remains linear in H up to 12 T (see also Takagi *et al.*[5]). Our work on $Nd_{2-x}Ce_xCuO_{4-\delta}$ pushes this limit to higher fields and lower T. Although $R_H(H)$ is slightly H dependent, no saturation of V_H is observed at 2 K for fields up to 20 T. This implies that either the g factor is anomalously small (<0.14), or that magnetic skew scattering from independent local moments is not playing a role in the Hall effect.

VI. SUMMARY

In all four crystals of $Nd_{2-x}Ce_xCuO_{4-\delta}$ studied ($x \sim 0.15$), R_H is found to be positive below ~ 80 K, although it may assume either sign at room temperature. The most interesting feature of R_H is its persistent T dependence down to 2 K, despite saturation of the ρ_a versus T curve below 30 K. We argue that the *sign* of R_H at any reference T is a poor criterion for deciding whether $Nd_{2-x}Ce_xCuO_{4-\delta}$ is a hole-type or an n-type superconductor. Using a two-band model that has broad validity we account for the general features of R_H and ρ_a. There exists a large band of negatively charged carriers which dominate the normal state resistivity. The resistivity profile, the infrared reflectivity, and the low-temperature magnetoresistance, all dominated by this band, show that the negatively charged carriers in the band are quite conventional in behavior. The saturation of ρ_a below 30 K, especially, reflects the behavior of the total scattering rate in the electron band. However, the low-temperature Hall effect reveals the existence of a smaller pocket of holes. We show that the temperature dependence of R_H, which persists to 2 K, reflects the anomalous inelastic-scattering mechanism of the holes. Thus, the fundamentally distinct scattering mechanisms in the two bands explains the different temperature scales observed in the two transport quantities. The Hall results provide direct evidence that an inelastic-scattering mechanism involving the holes remains observable to 2 K in this compound. The similarity between the behavior of the hole-scattering rate and that in earlier "hole" superconductors suggests to us that the holes, in fact, may be driving the superconducting transition in $Nd_{2-x}Ce_xCuO_{4-\delta}$.

Note added. A recent Hall study (Y. Iye *et al.*) on thin-film $Nd_{2-x}Ce_xCuO_{4-\delta}$ also shows an R_H that increases to large positive values at low T, in qualitative agreement with sample 4 of Fig. 5. Positive magnetoresistance was also measured by Iye. Tokura has also observed a positive R_H at low T in a $Nd_{2-x}Ce_xCuO_{4-\delta}$ single crystal with a $T_c \sim 20$ K.

ACKNOWLEDGMENTS

We acknowledge useful discussions with P. W. Anderson, H. Fukuyama, H. Takagi, Y. Tokura, and Y. Watanabe. This research is supported by the Office of Naval Research (Contract No. N00014-90-J-1013). The high-field experiments were carried out at the Francis Bitter National Magnet Laboratory, Cambridge, which is supported by the National Science Foundation.

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