In the layered cuprate perovskites, the occurrence of high-temperature superconductivity seems deeply related to the unusual nature of the hole excitations. The limiting case of a very small number of holes diffusing in the antiferromagnetic (AF) background may provide important insights into this problem. We have investigated the transport properties in a series of crystals of YBa$_2$Cu$_3$O$_y$ and found that the temperature dependencies of the Hall coefficient $R_H$ and thermopower $S$ change abruptly as soon as the AF phase boundary is crossed. In the AF state at low temperatures $T$, both $R_H$ and $S$ are unexpectedly suppressed to nearly zero over a broad interval of $T$. We argue that this suppression arises from near-exact symmetry in the particle-hole currents. From the trends in $R_H$ and $S$, we infer that the symmetry is increasingly robust as the hole density $x$ becomes very small ($x \approx 0.01$). We discuss implications for electronic properties both within the AF state and outside.

In the layered cuprate perovskites, high-temperature superconductivity appears when a moderate density of holes are introduced into the CuO$_2$ planes by chemical doping. The parent (undoped) compound is a Mott insulator with a spin-$\frac{1}{2}$ moment residing on each Cu ion in the plane. Below the Néel temperature, long-range, three-dimensional (3D) antiferromagnetism is stable. The introduction of holes destroys the static 3D antiferromagnetic (AF) order, and superconductivity appears when the relative hole population $x$ exceeds $\approx 0.05$ per Cu ion. In the quest to understand cuprate superconductivity, it has long been recognized that determining the nature of the itinerant hole moving a charge trailing a phase string (4).

Experimental Results

YBCO crystals were grown with near-optimal oxygen content in a Ba$_2$CuO$_3$ flux by using yttria-stabilized zirconia (YSZ) crucibles. To tune the oxygen content to a particular value $y$, we first attached three pairs of electrical contacts on each crystal, using EPO-TEK H20E silver epoxy, and then sealed the crystals in a quartz tube with crushed polycrystalline YBCO previously prepared with the desired $y$. The crystals (of thicknesses 10–30 μm) were initially annealed at 550°C for a week, and then slowly cooled (at 30°C/h) to 150°C, and held there for 3 weeks to optimize chain ordering (11, 15). The final cooling to 25°C takes ~12 h (we did not de-twin any of the crystals). [The low-$T_c$ annealing at 150°C seems crucial to our observations. A common practice in the past is to work with crystals that have been quenched directly from ~520°C to room temperature (quenching has the merit of providing a sharp resistive transition in crystals with $y < 6.50$). In the range $6.30 < y < 6.40$, however, the quenched disorder produces a $T_c$ that is strongly suppressed, often to below 4 K, and a nonmetallic $\rho$ profile. Subsequent “shell” annealing at room temperature produces an upward creep of $T_c$ as equilibrium is slowly restored (for $y = 6.41$, for instance, $T_c$ creeps from 13 to 27 K over a 6-day period; ref. 15). It appears that quenching leads to a large degree of in-plane disorder, which causes localization and strong $T_c$ suppression.](https://www.pnas.org/cgi/doi/10.1073/pnas.201228498)
However, the disorder can be removed by low-T annealing. The upward creep in $T_c$ is not observed in our annealed crystals.

A recent detailed calibration (Y.W., T. Kakeshita, S. Uchida, and N.P.O., unpublished data) finds that the in-plane hole density $x$ in YBCO varies linearly with $y$ in the underdoped regime as

$$x = 0.4(y - 6.20), \quad (y < 6.70).$$

In the range $6.23 < y < 6.63$, we measured in the same crystal the three in-plane quantities, resistivity $\rho$, Hall coefficient $R_H$, and thermopower $S$ (samples 1–6). $S$ and $\rho$, but not $R_H$, were measured in samples with larger $y$ (samples 7–9). $R_H$ was typically measured with an AC current by slowly sweeping the applied field $H$ from $-8$ T to $+8$ T and back with $T$ fixed (for all samples here, $R_H$ is $H$-independent). Above $250$ K, the swept-field Hall data were supplemented by high-density data obtained by a generalized van der Pauw method (fixing $H$ at $14$ T and alternating the current and Hall voltage contacts; where they overlap, the two data sets agree within our uncertainty). $S$ was measured with a thermal gradient of $0.5$ K/mm. We corrected for the small contribution from the Au leads.

Fig. 1 displays the in-plane resistivity for several of the crystals measured. In sample 1, which has the smallest $y$ (6.23), $\rho$ increases in a nearly activated way as $T$ decreases below $70$ K, implying strong localization of the carriers. At high temperature (above $320$ K), $\rho$ saturates at $38$ m$\Omega$cm (corresponding to a mobility $\mu \approx 2$ cm$^2$/Vs). Between these two limits, $\rho$ varies almost linearly with $T$. All three features qualitatively resemble those observed in very underdoped LSCO ($x = 0.04$; ref. 7, the magnitudes are also comparable if we scale by the hole density given by Eq. 1). Samples 2 and 3 also show the $T$-linear variation, but strong localization is not observed at low $T$ (see below).

Interestingly, as we cross the AF phase boundary, the $T$-linear dependence abruptly changes to the familiar “S-shaped” profile that characterizes $\rho$ in the doping range $6.40 < y < 6.70$. In this range, $\rho$ are in quantitative agreement with the data of Ito et al. (14).

In the narrow range $6.30 < y < 6.40$ just inside the AF regime, $T_c$ (determined by flux expulsion) is highly sensitive to $y$. As shown in the Inset of Fig. 1, $T_c$ in sample 3 ($y = 6.36$) equals $30$ K, compared with $27$ K measured by Veal et al. (15) in a crystal with $y = 6.38$, after long-time relaxation. However, a lower $T_c (12$ K) has been observed by Bonn et al. in a high-purity, de-twinned crystal ($y = 6.35$). Moreover, in samples 2 and 3, $\rho$ and $S$ show evidence for paraconducting fluctuations starting at $70$ K. Because of this variability, we will not base any of our conclusions on the behavior of $\rho$ in samples 2 and 3 in zero $H$ below $70$ K. [We note, however, that in a $14$-Tesla field $H \parallel \hat{c}$, $\rho$ in samples 2 and 3 saturates to a nearly $T$-independent value as bulk superconductivity is suppressed (broken lines in Fig. 1). We do not observe the upturn in $\rho$ associated with localization, in sharp contrast with sample 1. In sample 1, $\rho$ measured in a $14$-T field $\parallel \hat{c}$ (broken line) very nearly coincides with the zero-$H$ data. Provided that the $14$-Tesla field is high enough to yield the “normal-state” $\rho$ profile in samples 2 ($x = 0.04$) and $3 (x = 0.064)$, their metallic behavior implies that the critical value $x_c$ for localization in YBCO is much smaller than in LSCO ($x_c = 0.14$). This conclusion is preliminary until we repeat the $\rho$ measurements at much higher fields.]

We describe next the (weak-field) Hall coefficient $R_H$ in these samples. Fig. 2 displays the $T$ dependence of $R_H$ in samples 1–3 (with $y = 6.23, 6.30$, and 3.63, respectively), which lie within the AF region of the phase diagram. Because of the very low carrier densities in these samples, the high-temperature values of $R_H$ are dramatically enhanced (10–20 times larger than in optimally doped YBCO). At a temperature close to the Néel temperature $T_N \approx 410$ K, $R_H$ attains a peak value of $42 \times 10^{-9}$ m$^2$/C, corresponding to a Hall density $n_H$ of $1.50 \times 10^{20}$ cm$^{-3}$. Below $T_N$, $R_H$ decreases steeply, saturating below $70$ K to a very small positive value nearly $\sim 50$ times weaker than the peak value. The
Hall profiles in samples 2 and 3 also show similar decreases below $T_N$. However, the peak $R_H$ values are smaller (consistent with a higher hole concentration) and the low-$T$ values are not as strongly suppressed. These profiles differ strikingly from the standard $R_H$ profile observed in underdoped cuprates at moderately high doping. In crystals with $y = 6.60$, $R_H$ increases nominally as $1/T$ as the temperature decreases, and attains a maximum at a temperature $T_{\text{max},H} \approx 110-120$ K (this may be the most familiar profile of $R_H$ in underdoped cuprates).

The evolution of the Hall profiles in samples 1–3 to the pattern seen at higher doping is shown in Fig. 2 for samples 4–6 with $y = 6.40, 6.50, 6.60$, respectively. In examining the Hall curves in Fig. 3, we find that they separate naturally into two classes of behavior. Inside the AF phase (samples 2 and 3, which are reproduced here), $R_H$ reaches a maximum at $T_{\text{max},H}$ close to $T_N$, and then decreases below. The region over which $R_H$ is suppressed extends up to 300 K. Samples outside the AF phase (4–7), however, follow a common pattern that is distinct from that in the AF phase. Above the “notch” temperature $T_s \approx 350$ K (where a slight minimum may be observed), $R_H$ is nearly $T$-independent. (We will discuss the notch elsewhere.) Below $T_s$, $R_H$ increases to a broad peak at $T_{\text{max},H}$ that is much lower than in the AF phase.

From Figs. 2 and 3, we infer the following trend. Deep in the AF state, strong suppression of $R_H$ is apparent over a wide range of $T$ (samples 1–3). However, when the AF boundary is crossed ($y = 6.40$), this region of suppressed $R_H$ and $S$ fills in abruptly. Concurrently, the peak temperatures in $R_H$ abruptly falls from 350 K to 150 K in sample 4, and to 120 K in sample 6 ($y = 6.63$).

Remarkably, the pattern of behavior in $R_H$ is matched by the behavior of the in-plane thermopower $S$. Fig. 4 displays $S$ measured in samples 1–3. As in $R_H$, the thermopower attains a broad peak near $T_N$ and then decreases monotonically below $T_N$. The suppression of $S$ below $T_N$ extends over a broad range of $T$ in these samples. A striking feature of $S$ in these samples is that it has virtually the same $T$ dependence as $R_H$. To facilitate comparison, the curves of $R_H$ in samples 1 and 2 are replotted as broken lines in Fig. 4 and scaled to match the corresponding curves for $S$. The comparisons show that both the Hall effect and thermopower are suppressed in nearly the same way over a wide range of $T$ inside the AF phase.

We display the thermopower at higher doping in Fig. 5. The gradual evolution of the profile of $S$ vs. $T$ in the AF phase to the more familiar profile at moderate doping also agrees with the corresponding changes in $R_H$. For $y < 6.40$ the peak in $S$ is close to $T_N$, whereas for $y > 6.40$ it decreases systematically, eventually reaching 120 K at $y = 6.67$.

Fig. 3. The in-plane Hall coefficient $R_H$ of YBCO in samples 2–6 with $y = 6.30, 6.36, 6.40, 6.45,$ and 6.63, respectively. The profiles of $R_H$ in samples 2 and 3 are qualitatively different from those in samples 4–6. In samples 2 and 3, $R_H$ peaks near $T_N$ and decreases monotonically as in Fig. 2. However, in samples 4–6 ($T_N = 0$), the peak temperature in $R_H$ is systematically lower as $y$ increases beyond 6.40. All samples display a “notch” near 350 K where $\rho$ displays a slope change.

Fig. 4. The in-plane thermopower $S$ in samples 1–3 of YBCO (data points). In samples 1 and 2, $S$ decreases monotonically nearly to zero as $T$ decreases below $T_N$ with nearly the same $T$ dependence as $R_H$. The broken lines represent $R_H$ in samples 1 and 2 (values of $R_H$ are on the right scale). In sample 2 ($y = 6.30$), $R_H$ is shown multiplied by 2.25.

Fig. 5. The in-plane thermopower $S$ in samples 4–8 of YBCO (with $y = 6.40, 6.45, 6.50, 6.60,$ and 6.67), which lie outside the AF region. In each sample, $S$ attains a peak at a temperature close to the peak temperature of $R_H$. 

Wang and Ong
The simultaneous suppression of the Hall and thermopower signals is anomalous. We review below the general conditions under which cancellation of these signals is observed in conventional metals and semiconductors.

**Discussion**

In the simplest case of a single-band isotropic Fermi Surface (FS) with carrier density \( n \), the weak-field \( R_H \) is expected to increase as \( 1/n \), and \( S \approx 1/e_H \) as \( n \) decreases (\( e_H \) is the Fermi energy).

For our experiment, the important aspect of \( R_H \) and \( S \) is their sensitivity to the sign of the carriers. When both electrons and holes are present (mixed conduction), the observed Hall and thermopower signals are the algebraic sums of contributions from the two carrier types.

In terms of \( \sigma \) (conductivity) and \( \sigma_H \) (Hall conductivity), \( R_H \) equals \( \sigma_H/\sigma^2 \). As \( \sigma_H \) measures the transverse current produced by the Lorentz force in an applied \( H \), it is sensitive to the sign of the carrier charge. For mixed conduction, \( \sigma_H \) is the sum

\[
\sigma_H = \sigma_{H}^e + \sigma_{H}^h, \tag{2}
\]

where the electron and hole Hall conductivities (\( \sigma_{H}^e \) and \( \sigma_{H}^h \), respectively) are of opposite signs. In a two-dimensional (2D) metal with arbitrary FS shape and lifetime anisotropy, \( \sigma_{H} \) scales as \( \langle \ell \rangle \) (where \( \ell \) is the mean-free-path and \( \langle \cdot \rangle \) denotes averaging over the FS; ref. 16). Exact cancellation between the two terms, if any, occurs at discrete accidental temperatures where their \( \langle \ell \rangle \) happen to be equal. Away from these accidental values of \( T \), the scale of \( R_H \) is of the order \( 1/ne \) with \( n \) set by the dominant FS pocket.

Whereas the sign of \( \sigma_H \) depends on the FS curvature of the dominant pocket, the thermopower probes particle-hole asymmetry in a rather different sense. In a thermopower measurement, the current generated by an applied temperature gradient \( -\nabla T \) is cancelled by an opposite current produced by an electric field \( E \) (produced by charge accumulation at the ends of the sample). We have

\[
\alpha(-\nabla T) = -\sigma E, \tag{3}
\]

where \( \alpha \) is the Peltier conductivity and \( \sigma \) the electrical conductivity. In conventional metals with a single FS, the thermopower \( S = -E/\nabla T \) is \( \alpha/\sigma \) and is given by

\[
S = \frac{1}{\sigma} \frac{\pi k_B T}{\hbar} \frac{\partial G}{\partial e} \frac{\mu}{e}, \tag{4}
\]

where the “conductivity function” \( G(e) = e^2 D(e) \langle v_e f_e \rangle \) is essentially the integral of \( D(e) \) weighted by the product \( v_e f_e \). Hence, \( S \) may vanish if \( G \) displays exact particle-hole symmetry about \( \mu \).

(If we have separate particle and hole FS pockets, \( S \) may also vanish by accidental cancellation as discussed for \( \sigma_H \).)

Unlike the case for \( \sigma_H \), full cancellation leading to a zero \( S \) is rarely observed, especially in the low-density limit. When \( S \) is observed to cross zero, as at low \( T \) in noble metals (e.g., Au), the cause is an extraneous effect (phonon drag) rather than a matching of the derivatives of \( G(e) \). The present results are all of the more unusual because \( R_H \) and \( S \) share nearly the same \( T \) dependence over such a broad range of \( T \). Some of the issues are aired by analyzing the following situations.

It is fair to ask whether, in the most underdoped samples, the holes undergo charge segregation and are confined to macroscopic high-conductivity regions that are connected along the sample length. We argue that the local nature of current cancellation (Eq. 3) in a thermopower measurement rules out this scenario. The high-conductivity channel may be modeled as a self-avoiding chain of \( N \) segments (of conductivities \( \sigma_M \) and \( \sigma_A \) described by \( L_i = \delta_{ik} \)). Applying Eq. 3 to each segment, we have

\[
E_j \cdot d_j = -\frac{\sigma_A}{\sigma_M} (-\nabla T) \cdot d_j. \tag{5}
\]

As the sum of \( E_j \cdot d_j \) over \( j \) is the observed Seebeck voltage, Eq. 5 implies that the observed thermopower takes on the value of the high-conductivity material—i.e., the conducting channel largely determines the Seebeck voltage. We believe that the sharp differences between the profiles of \( S \) in samples 1 and 5, say, precludes this scenario.

A related issue is whether localization plays a role in the suppression of \( R_H \). In conventional systems, the onset of Anderson localization either leaves \( R_H \) unchanged through the metal-insulator transition (Si:P and InO3) or to an increase in \( R_H \) (Si:B and Ge:Sb; references to the Hall results and in Anderson localization can be found in ref. 17)—i.e., the opposite of what is needed to account for the present data. In underdoped LSCO (where the onset of strong localization is most heavily studied), localization also leads to an increasing \( R_H \) at low \( T \) (18, 19). We are not aware of a material in which an increase in \( \rho \) (driven by localization) is accompanied by a plummeting \( R_H \). Quite apart from these general remarks on the behavior of \( R_H \) in the presence of localization, we note that the regime of localization in YBCO is quite restricted. In sample 1 it onset at 70 K, whereas \( R_H \) and \( S \) start declining at 300 K (Fig. 2), so these can hardly be related phenomena. Similarly, in samples 2 and 3, the behavior of \( \rho \) in an intense field (Fig. 1) sets a conservative upper bound of \( \approx 20 \) K for the onset of localization, whereas \( R_H \) and \( S \) start falling at 300 K. We conclude that the suppression of \( R_H \) is unrelated to localization.

Finally, we note that a quasi-one-dimensional (1D) electronic state in itself does not ensure a zero \( R_H \) (see also ref. 20). Quasi-1D conductors whose Hall effect have been measured typically display a finite and strongly \( T \) dependent \( R_H \) (21) because a very small but measurable hopping amplitude transverse to the chains is sufficient to produce a finite Hall resistivity (the low carrier densities in these systems also strongly amplify \( R_H \)).

**Particle-Hole Symmetry**

With the above remarks, let us discuss \( R_H \) and \( S \) in samples 1–3 (Figs. 2 and 4). The overall scale of \( R_H \) in Figs. 2 and 3 (as indicated by its peak value) increases rapidly as \( y \) decreases to 6.20. Interestingly, we find that the Hall density \( n_H = (eR_H)^{-1} \) evaluated at the peak is in very good agreement with the calibration Eq. 1. Hence, \( R_H \) starts falling from a value fixed by the hole density near \( T_N \), and then decreases virtually to zero. The decrease is matched by that in \( S \). In this state, particle-hole symmetry of a rather unusual type appears, and becomes increasingly robust with decreasing \( T \).

As discussed above, we believe that the decrease in both quantities arises from cancellation between particle and hole-like currents. To achieve the cancellation in \( R_H \), both the carrier densities and the average mean-free-paths must be closely matched in the two pockets over an extended range of \( T \). Moreover, to maintain the same cancellation in \( S \), the derivatives of \( G(e) \) in the two pockets must be equal in magnitude. Hence, the observed behaviors of \( R_H \) and \( S \) place very stringent conditions on the electronic state. There appears to be a self-adjusting mechanism that automatically maintains the symmetry of the particle and hole currents over a broad range of \( T \) below \( T_N \).
In the striped phase that appears in Nd-doped LSCO below the LTT-LTO (low-temperature tetragonal to orthorhombic) transition at \( T_a \sim 74 \) K (22, 23), \( R_H \) (as well as \( \sigma_H \)) are observed to decrease toward zero nearly linearly with \( T \) below \( T_a \). \( S \) also decreases below \( T_a \), but at a faster rate. Whereas the earlier discussion of a vanishing \( R_H \) was in terms of quasi-1D behavior, a recent analysis proposes particle-hole symmetry as the cause (20). At the hole concentration \( x = \frac{1}{8} \) in Nd-doped LSCO, the conducting chains are \( \frac{1}{4} \)-filled, so that the charge carriers have exact particle-hole symmetry (equivalent to a \( \frac{1}{2} \)-filled 1D band for spinless fermions). This readily accounts for the rapid suppression of \( S \) as well. There is possibly a close relationship between the LTT phase in Nd-doped LSCO and the particle-hole symmetric state in YBCO. However, we note that there is a wide disparity in the hole densities. In sample 1, the estimated \( x \approx 0.01 \) is an order of magnitude smaller than that in the Nd-LSCO material. To account for the vanishing of both \( R_H \) and \( S \), it seems that the \( \frac{1}{4} \)-filled chain scenario must extend to this low concentration in YBCO. At such low doping levels, however, the chains have to be \( \sim 50 \) lattice spacings apart. This would appear to be a serious problem unless it can be shown that such widely separated \( \frac{1}{4} \)-filled chains are energetically favorable.

A competing model in the AF regime is the nodal excitation at the nodes of the pseudogap state (25). Where in \( k \)-space do the doped holes end up in the limit \( x \to 0 \)? In the \textit{insulating} oxychloride cuprate \( \text{Ca}_x\text{Cu}_2\text{O}_3\text{Cl}_2 \) angle-resolved photoemission spectroscopy (ARPES) experiments (26) reveal that the occupied states (in the lower Hubbard band) nearest the Fermi level have exact particle-hole symmetry (equivalent to a \( \frac{1}{2} \)-filled 1D band for spinless fermions). This readily accounts for the rapid suppression of \( S \) as well. There is possibly a close relationship between the LTT phase in Nd-doped LSCO and the particle-hole symmetric state in YBCO. However, we note that there is a wide disparity in the hole densities. In sample 1, the estimated \( x \approx 0.01 \) is an order of magnitude smaller than that in the Nd-LSCO material. To account for the vanishing of both \( R_H \) and \( S \), it seems that the \( \frac{1}{4} \)-filled chain scenario must extend to this low concentration in YBCO. At such low doping levels, however, the chains have to be \( \sim 50 \) lattice spacings apart. This would appear to be a serious problem unless it can be shown that such widely separated \( \frac{1}{4} \)-filled chains are energetically favorable.

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