Mechanisms of photodoping in oxygen-deficient YBa$_2$Cu$_3$O$_x$ films studied by *in situ* transport measurements

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In situ studies of the superconducting and normal-state transport properties in partially oxygen-depleted, metallic YBa$_2$Cu$_3$O$_x$ ($T_{c,\text{mid}} \approx 52$ K) thin films exposed to long-term white-light illumination (photodoping) are reported. We observed that the effects of photoexcitation strongly depended on the temperature at which the photodoping was performed. The Hall number increased during the illumination in the entire tested temperature range from 70 to 290 K, with the strongest increase near room temperature, whereas, the Hall mobility increased steadily only upon low-temperature illumination. At temperatures above 250 K, it showed an abrupt initial increase followed by a long-term decrease. At high temperatures, the Hall quantities react on the impact of light excitation independently from each other, which strongly suggests that both the photoassisted oxygen ordering and charge-transfer mechanisms contribute to photodoping, the former acting mainly on the mobility, while the latter on the density of charge carriers. The photoinduced enhancement of the superconducting transition temperature T_c exhibited essentially the same temperature dependence as the enhancement of the Hall number, being largest ($\Delta T_c \approx 2.6$ K) for the illumination performed at high temperatures. Thus, the T_c enhancement results from the change of the density more likely than of the mobility of the charge carriers. [S0163-1829(98)05714-2]

I. INTRODUCTION

It has been shown in various studies[1–14] that physical properties of YBa$_2$Cu$_3$O$_x$ (YBCO) with $6 < x < 7$ are changed persistently by long-term illumination with white light or visible laser radiation. This remarkable phenomenon, known as persistent photoconductivity (PPC), is a common feature of all oxygen-deficient YBCO samples. In semiconducting samples ($x \approx 6.4$), prolonged illumination progressively transforms YBCO from a semiconducting (nonsuperconducting) state to a metallic (superconducting) state.[1–3] In the superconducting samples ($6.4 < x < 7$), the illumination causes, in addition to the decrease of the electrical resistivity, a simultaneous increase of the superconducting transition temperature T_c.[4–8] The observed light-induced changes of the Hall coefficient and the Hall mobility,[5–7,9] the contraction of the c-axis lattice parameter,[10,11] and the increase of the superconducting coherence lengths ξ_{ab} and ξ_c,[12] give evidence that prolonged light illumination modifies both the electrical and the structural properties of YBCO. The photoinduced effects decay only at temperatures above 250 K in metallic YBCO (Ref. 13) and above 270 K in insulating YBCO,[3,14] below these temperatures the PPC was found to be indeed persistent.

The aforementioned results, as well as the apparent difficulties to clearly observe PPC in other high-temperature superconducting (HTSC) materials,[7,15,16] suggest that PPC is strongly correlated to the oxygen content and stoichiometry of YBCO. The unit cell of the $x = 7$ compound contains CuO$_2$ double planes and CuO chains, the latter playing the role of charge carrier reservoirs for the planes.[17] The chain-oxygen ions are weakly bounded and can be easily removed by temperature treatments in the oxygen-poor atmosphere.[18,19] Near $x = 6.5$ the CuO-chain layer seems to be partially ordered in the so-called ortho-II phase,[20] but, in general, the CuO chains in partially oxygen-deficient samples consist of more or less disordered segments.

All models proposed for PPC in YBCO are based on that disordered structure of the CuO chains. In the so-called *charge-transfer* model,[3] the photoexcited electrons are transferred to the CuO chains and trapped there in unoccupied p levels of oxygen ions, while holes remain mobile in the planes. The trapped electrons induce a local potential distortion resulting in an energy barrier of about 1 eV[3,14] which, at low temperatures, prevents recombination of electrons with excess holes, resulting in PPC. The *photoassisted oxygen-ordering* model[5] assumes that the visible light induces an oxygen reordering process in a way that the average length of the CuO-chain segment increases. It is well known that the increased length of the CuO chains results in the increase of free-carrier (hole) concentration within the CuO$_2$ planes. In the *oxygen vacancy capture* model,[21,22] the photoexcited electrons are trapped by oxygen vacancies in the CuO chains. This model seems most appropriate to explain PPC in oxygen-poor (insulating) samples.

Each of the above models has its characteristic strengths and problems. The *charge-transfer* model, as well as the *oxygen vacancy capture* model, give reasonable explanations for

the observed wavelength dependence of PPC, predicting the onset of the photodoping efficiency[3,23,24] at the charge-transfer gap energy (approximately 1.6 eV) of the semiconducting YBCO.[25] These models, however, have difficulties describing the c-axis shrinking and the increase of the Hall mobility during the photoexcitation. These findings are more easily described by the *photoassisted oxygen-ordering model*, which, on the other hand, has problems with the PPC in the oxygen-poor YBCO samples and also gives no straightforward picture of the wavelength dependence of PPC. We want to stress, however, that the disordered structure of the oxygen-deficient YBCO, where one can expect the presence of unoccupied p levels, oxygen vacancies, and a more-or-less random arrangement of chain segments, suggests the possibility that all of the above processes may simultaneously contribute to PPC, with their relative importance depending on the oxygen content x and on the temperature at which the photodoping is performed.

In this paper, we present *in situ* studies of the time evolution of the transport properties of partially oxygen-depleted epitaxial YBCO thin-film samples, light illuminated at different temperatures. Our aim is to determine the role and contribution of each PPC mechanism in the photodoping process. Thereby, we extend our preliminary studies[26] by including different doping temperatures and giving a comprehensive description of the impact of prolonged light illumination on the transport properties of YBCO, including the resistance, Hall resistivity, and T_c. Our results indicate that not only the effectiveness of photodoping, but also the electronic properties of the photodoped state are strongly dependent on the temperature at which the photodoping process is conducted. Our conclusion is that several physical mechanisms simultaneously contribute to the photodoping process. In addition, we find evidence that near room temperature, there is a direct competition between the oxygen *ordering* and *disordering* processes. These structural instabilities directly influence the carrier mobility but at the same time seem to have only a minor, indirect influence on the free-carrier concentration in the material.

II. SAMPLE PREPARATIONS AND MEASUREMENT METHODS

In our experiments, we used about 140-nm-thick YBCO films deposited *in situ* by rf magnetron sputtering on LaAlO$_3$ substrates. As-deposited films were fully oxygenated and exhibited a 0.5-K-wide superconducting transition at approximately 90 K. The oxygen content of the films was subsequently reduced by annealing for 1 to 2 h at 330–450 °C in 20 mTorr of oxygen and cooling down to 270 °C within 20 min, followed by a quench to room temperature. T_c values from 10 to 80 K were obtained with this method.[27] By reoxidizing some of the previously oxygen-depleted films, the initial T_c value was reproduced within 1–2 K, showing that the annealing procedure did not degrade our films or change their structural integrity. For the presented studies, samples with $T_{c,\mathrm{mid}}$ of about 52 K were used, applying the midpoint-of-transition criterion. According to Ref. 7, this corresponds to an oxygen content of $x \approx 6.5$ to 6.6. The films were patterned by a laser inhibition technique[28] into six-contact test structures, suitable for measurements of the resistivity and the Hall voltage.

In situ measurements of the resistivity and the Hall effect during light illumination were performed in a temperature-controlled closed-cycle refrigerator, with the sample mounted on a cold finger. The sample was illuminated through a Suprasil glass window with a 100-W quartz tungsten halogen lamp. The light intensity on the surface of the sample was estimated to about 1 W/cm^2. A water filter was used to eliminate the infrared part of the radiation. Long-term temperature stability was substantially improved, compared with our previous studies[26] by (a) applying an additional inner heat shield around the sample with an entrance slit of about 1 cm^2, and (b) mounting the temperature sensor close to the sample and illuminating it together with the sample. These measures limited the initial temperature rise after turning on the lamp to $\leqslant 0.5$ K, which, within a few minutes, was completely nullified by the temperature control system.

For the Hall-effect measurements, an electromagnet provided a magnetic field of $B = 0.52$ T perpendicular to the film's surface. The electrical measurements were performed with a lock-in technique and a very stable 17-Hz current source. The experimental setup was fully computer controlled. The longitudinal and Hall voltages in both polarities of the magnetic field and at the zero field were simultaneously recorded during the experiment at every 1.4 min time interval.

To ensure comparability of the measurements taken at different temperatures, the following experimental procedure was implemented. After an initial cooldown to 40 K to determine its intrinsic T_c, the sample was heated to a desired experimental temperature T and was photodoped for time t of 48 h. Next, it was immediately cooled down again to determine the T_c enhancement. Finally, the sample was warmed up to 310 K and kept there for 24 h to allow a complete decay of PPC. Later, the cycle was repeated, including the initial cool-down to check reproducibility of our measurements, with the photodoping carried out at a different temperature. During the entire sequence of measurements, the experimental setup was kept absolutely unchanged.

III. EXPERIMENTAL RESULTS

Figure 1 shows the normalized longitudinal resistivity $\rho_{xx}(t)/\rho_{xx}(0)$, measured during white-light illumination for 48 h at 70, 100, 200, 260, and 290 K, respectively, using the same light intensity in each of the measurements. At the beginning, $\rho_{xx}(t)/\rho_{xx}(0)$ dropped rapidly, followed by a slower decrease at long illumination times. It is evident from Fig. 1 that the total reduction of the resistivity achieved after 48 h (we will call this value the photodoping effectiveness) strongly depended on the temperature. The effectiveness was largest at 260 K (about 13%), and smallest at 200 K (about 9%). At the other studied temperatures, the values of effectiveness of about 10–11 % were observed.

In Fig. 2, the normalized Hall resistivity $\rho_{yx}(t)/\rho_{yx}(0)$ is shown as a function of the illumination time at the studied temperatures. Compared with $\rho_{xx}(t)/\rho_{xx}(0)$, $\rho_{yx}(t)/\rho_{yx}(0)$ displayed a quite different behavior of the effectiveness of
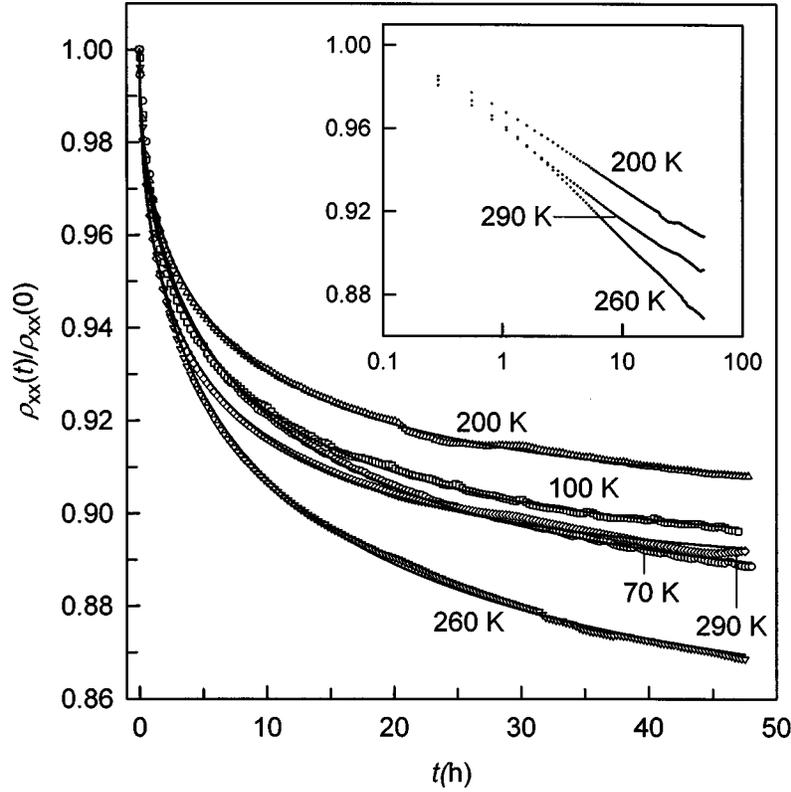
FIG. 1. Time dependence of the normalized resistivity, $\rho_{xx}(t)/\rho_{xx}(0)$, of $YBa_2Cu_3O_x$ ($x \approx 6.5$, $T_{c,\text{mid}} \approx 52$ K) during white-light illumination, measured at the indicated temperatures. Solid lines represent the fits obtained with the help of Eq. (1). The inset shows three of the data curves plotted on a logarithmic time scale.

photodoping. At temperatures up to 200 K, the effectiveness of only 5–6 % was achieved, whereas at both 260 and 290 K, it sharply increased to about 11%. Similarly to $\rho_{xx}(t)/\rho_{xx}(0)$, no saturation was observed in the $\rho_{yx}(t)/\rho_{yx}(0)$ dependence (see insets in Figs. 1 and 2).

$\rho_{xx}(t)$ and $\rho_{yx}(t)$ dependences are clearly nonexponential. In agreement with previous results,[13,26] our experimental data can be well described by the empirical stretched-exponential Kohlrausch expression:[3]

$$\rho_{ik}(t) = \rho_{ik}(t \to \infty) + [\rho_{ik}(0) - \rho_{ik}(t \to \infty)]$$
$$\times \exp\{-(t/\tau)_{ik})^{\beta_{ik}}\}, \qquad (1)$$

where "ik" is replaced by either "xx" or "yx" depending on the quantity under consideration. $\rho_{ik}(t \to \infty)$ is the saturation value of $\rho_{ik}(t)$, τ_{ik} is a time constant, and β_{ik} is a dispersion parameter ($0 < \beta_{ik} < 1$). As reported previously,[13] the measured values show a general tendency to fall somewhat below the theoretical curves at large illumination times.

To separate the different contributions to the photodoping effect, the Hall number $p_H = 1/(R_H e)$ ($R_H = \rho_{yx}/B$ is the Hall coefficient) and the Hall mobility $\mu_H = R_H/\rho_{xx}$ were calculated from $\rho_{xx}(t)$ and $\rho_{yx}(t)$. Within a single-band model, p_H and μ_H correspond to the free-hole concentration and the hole mobility, respectively. Since $p_H \propto 1/\rho_{yx}$, the temperature dependence of p_H (not shown) is just the inverse of ρ_{yx}, shown in Fig. 2. Hence, p_H increases strictly monotonously as a function of the illumination time at all studied temperatures, and even after 48 h of illumination there is no sign of a saturation in any of the curves. We note that, for

times below 30 h, the increase of p_H is largest at 290 K but shows a flattening dependence at larger illumination times. This latter effect is a sign of PPC relaxation, which becomes important in this temperature and time range.

As compared to p_H, $\mu_H(t)$ displays a remarkably different behavior (Fig. 3). At 70 and 100 K, $\mu_H(t)$ shows, similarly to $p_H(t)$, a monotonical increase without a saturation for up to 48 h of illumination. However, the $\mu_H(t)$ dependence changes dramatically when the photodoping temperature is increased. At 200 K, $\mu_H(t)$ saturates after approximately 18 h of illumination. At both 260 and 290 K, $\mu_H(t)$ increases rapidly at the very beginning of the photodoping process, reaches a maximum value after a few hours of illumination, and subsequently *decreases*. Based on the above data, we believe that the process responsible for the mobility enhancement at low temperatures is still present at higher temperatures, but it is counteracted by another process showing a falling tendency. It should be noted that, in contrast to p_H (48 h), μ_H (48 h) shows a monotonical temperature dependence and that at 290 K, μ_H (48 h) is somewhat *smaller* than the original, undoped value. This latter finding confirms our previous results[26] but is considerably different from the results presented by other groups. The data published by others show only a monotonical increase of the Hall mobility during the illumination[6,9] with the exception of a remark made by Osquiguil *et al.*,[7] who stated (without showing an appropriate figure) that in a YBCO sample with the oxygen content $x = 6.7$, μ_H decreased during the photodoping at room temperature. We also observe in Fig. 3 that the rising as well as the falling components of $\mu_H(t)$ become clearly
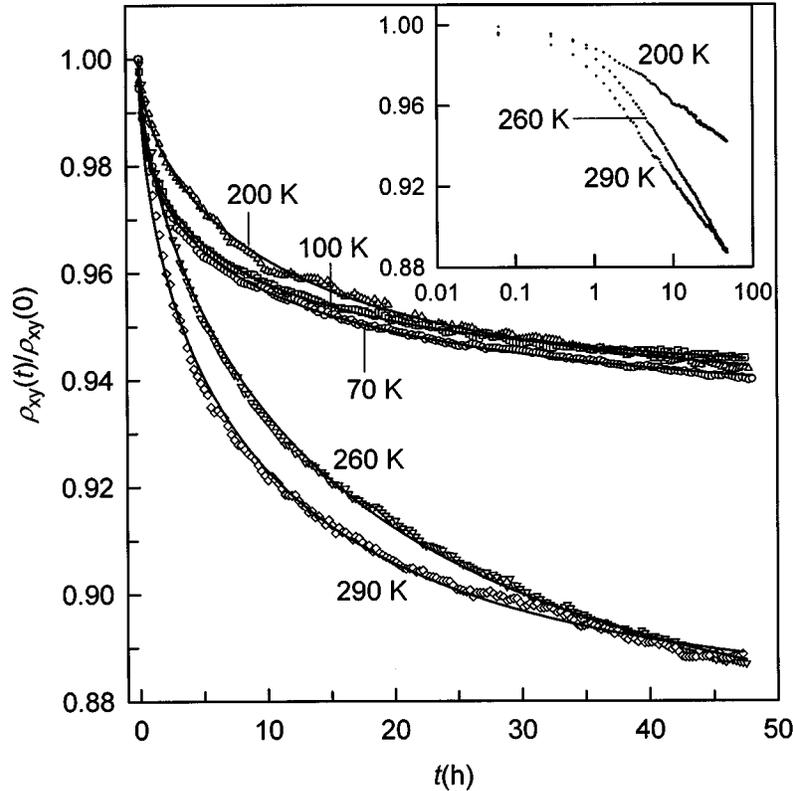
FIG. 2. Time dependence of the normalized Hall resistivity, $\rho_{yx}(t)/\rho_{yx}(0)$, of $YBa_2Cu_3O_x$ ($x \approx 6.5$, $T_{c,\mathrm{mid}} \approx 52$ K) in a magnetic field of $B = 0.52$ T, measured under the same conditions as $\rho_{xx}(t)/\rho_{xx}(0)$ in Fig. 1. Solid lines represent the fits obtained with the help of Eq. (1). The inset shows three of the data curves plotted on a logarithmic time scale.

steeper with the increasing temperature. Obviously, the kinetics of both components is substantially accelerated by rising the temperature.

The T_c of our samples was measured before and after each of the photodoping experiments. The $\rho_{xx}(T)$ curves measured for the ''erased'' samples always overlaid, within the accuracy of our measurement, the characteristics obtained for an undoped, nonilluminated sample. In contrast, the $\rho_{xx}(T)$ characteristics of the photodoped samples were different and depended on the temperature of the preceeding photodoping experiment. Up to 200 K, the curves overlayed each other and were more or less shifted parallel with respect to the undoped curve. The curves collected at the 260 and 290 K doping temperatures exhibited substantially lower values of resistivity and intersected at 290 K. These data will be presented elsewhere. Here, we only state that photodoping carried out at different temperatures, but with identical photon doses, lead to different $\rho_{xx}(T)$ characteristics and, hence, to the photoexcited states with different electronic structures. If we define the T_c enhancement ΔT_c as the shift of the offset temperature [i.e., where $\rho_{xx}(T) = 0$], we find that ΔT_c depended strongly on temperature at which the photodoping was performed. Photodoping at temperatures up to 200 K resulted in $\Delta T_c \approx 1.3$ K, but at 260 and 290 K, ΔT_c was approximately twice as large. This final result is of particular importance since the value of T_c is correlated to the carrier concentration in the material.[29]

In Fig. 4, we summarize the effects of photodoping in our YBCO samples. The relative changes $\delta\sigma_{xx}$, δp_H, and $\delta\mu_H$ of $\sigma_{xx} = 1/\rho_{xx}$, p_H, and μ_H are expressed as the percentage

of the corresponding initial values, respectively, and are plotted together with ΔT_c. As can be clearly seen in Fig. 4, ΔT_c depends on the photodoping temperature *essentially in the same way* as δp_H, which confirms that the enhancement of both these quantities is governed by the enhancement of the same electronic parameter, i.e., the free-carrier concentration. In obvious contrast, $\delta\sigma_{xx}$ and $\delta\mu_H$ show completely different temperature dependences, due to complicated behavior of μ_H during the illumination process (see Fig. 3).

IV. DISCUSSION

In HTSC materials, p_H shows an unexpected temperature dependence. In metallic YBCO,[30] as well as in Bi-based[31] and Tl-based[32] compounds, p_H depends almost linearly on T above T_c. Thus, p_H calculated from the simple one-band formula $p_H = 1/(R_H e)$ cannot be directly identified as the density of charge carriers, which should be more-or-less constant in a metal. However, starting with the Anderson model,[33] Jones *et al.*[34] have argued that the *changes* of p_H and μ_H, observed at any arbitrarily *fixed* temperature, measure the *changes* of the *true* carrier concentration and the mobility. This consideration is strongly supported by the fact that δp_H and ΔT_c in our PPC experiments show essentially the same dependence on the photodoping temperature (Fig. 4). Therefore, in the following discussion, we will use the terms carrier concentration n and mobility μ interchangeably with p_H and μ_H.

It is helpful for the further discussion of our results to briefly summarize predictions that the different PPC models
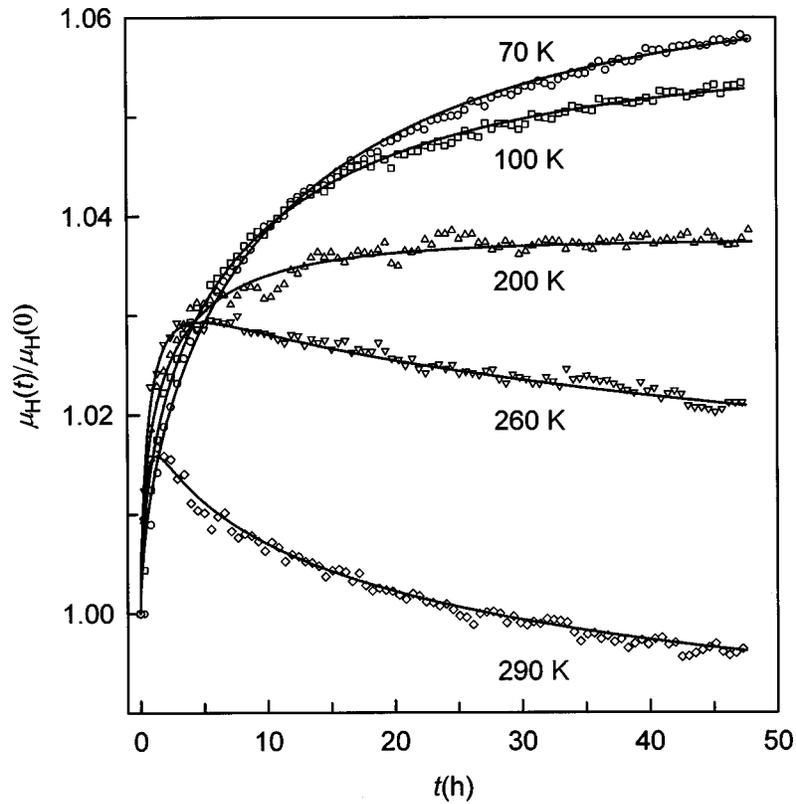
FIG. 3. Normalized Hall mobility $\mu_H = R_H / \rho_{xx}$ as a function of the illumination time, taken at the indicated temperatures. Solid lines are the sum of two Kohlrausch expressions [Eq. (1)], one of them rising and the other falling with the increasing illumination time. They are meant to serve as guides to the eye.
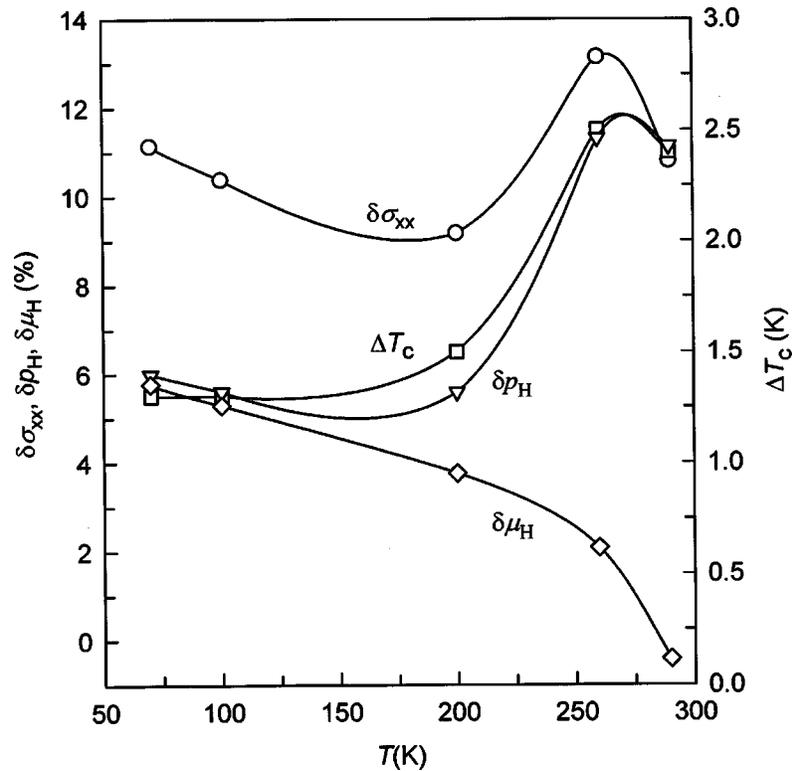
FIG. 4. Relative changes of the longitudinal conductivity $\delta\sigma_{xx}$, the Hall number δp_H, the Hall mobility $\delta\mu_H$, and the absolute T_c enhancement ΔT_c, after light excitation for 48 h, plotted as a function of the photodoping temperature. Solid lines are just guides to the eye.

(outlined in Sec. I) give for photodoping's impact on carrier properties. In the *charge-transfer* as well as in the *oxygen vacancy capture* model, photodoping should increase mainly the carrier concentration. As far as the drift mobility is concerned, a decrease is more likely to occur, because of the additional disorder introduced by randomly distributed trapped electrons, and the increased fermion-fermion scattering rate due to the photoinduced charge carriers (holes).

In the *photoassisted oxygen-ordering* model, n is enhanced in two ways: first, electrons trapped at the ends of chain segments result in mobile holes in the CuO_2 planes; second, the chains are lengthened by the oxygen reordering,[7] inducing an additional charge transfer from planes to chains.[35] The growth of the chains must change the electronic and the crystal structure of the material, and, hence, the carrier mobility μ. We expect that oxygen ordering should *increase* the carrier mobility since it reduces disorder in a system, increasing the average scattering time. However, in experiments of room-temperature aging of quenched YBCO samples, a *decrease* of μ_H was observed.[6] Admittedly, the analogy between the photoassisted oxygen ordering and oxygen ordering in quenched samples is limited. In the photodoping experiments, the sample is *removed* from its initial thermodynamical state by lowering the thermally driven disorder. This is in sharp contrast to the room-temperature aging process, which moves the system *towards* the intrinsic equilibrium state. Thus, despite apparent analogies [e.g., the applicability of Eq. (1); see Ref. 36], the results of room-temperature aging experiments cannot be directly taken as a model for photoassisted oxygen ordering.

Based on the above considerations we will now assess the experimental results discussed in Sec. III. Most striking are the markedly different time dependences of p_H and μ_H, which certainly cannot be explained by implementing exclusively the *oxygen-ordering* model, since this model predicts a very close relationship between n and μ. The results give no evidence for such relationship, in particular, the maximum of $\mu_H(t)$ at high temperatures (Fig. 3) is not reflected in $p_H(t)$. On the other hand, only *oxygen ordering* seems to give a reasonable explanation for the *increasing* part of the mobility observed at all temperatures. Thus, our conclusion is that oxygen ordering enhances mainly μ and has only a very limited effect on n.

Another intriguing phenomenon established by our experiments is the decreasing component μ_{dec} of the mobility during a prolonged illumination (Fig. 3). μ_{dec} is observable only at $T > 250$ K, where the thermal relaxation of PPC occurs in metallic YBCO.[13] Most likely, both effects have a common physical background in oxygen ordering/disordering processes, which are strongly accelerated, when the temperature approaches room temperature, due to the increased diffusivity of oxygen.[37] A reasonable picture is that the thermal relaxation process gradually destroys photoinduced ordering in the chains, simultaneously, either having only a limited influence on the carrier density or being over-compensated by an enhanced photogeneration of additional carriers. This competition between the photoinduced oxygen ordering and thermally driven oxygen disordering processes could also explain why, after long exposure times, μ_H falls

below its initial, undoped value, despite the fact that one would expect PPC relaxation to cancel only the photoinduced enhancement of μ. A similar behavior of μ was observed in our other photodoping/relaxation experiments,[38] so the assignment of the falling component of μ to the thermal PPC relaxation is a consistent interpretation of our data. However, μ_{dec} keeps decreasing even after 48 h of illumination (Fig. 3), while μ_H under dark conditions saturates within about 15 h,[38] indicating differences between relaxation processes with or without light exposure and again pointing to *photoassisted oxygen ordering* as the mechanism for enhancing mobility.

The above considerations imply that the main contribution to the photoinduced enhancement of n originates from mechanisms that have little or no influence on μ. The most obvious candidates for n enhancement are the *charge transfer* and the *oxygen vacancy capture* mechanisms, but one cannot exclude trapping of photoexcited electrons by impurities or on lattice distortions/grain boundaries. Figure 2 clearly demonstrates (note that $p_H \propto 1/\rho_{yx}$) that the enhancement of n is the largest near room temperature, i.e., in the same temperature region where μ_H exhibits its unusual behavior (Fig. 3), and the thermal relaxation of PPC becomes important.[13,14] Thus, our data seem to indicate that the thermal disordering creates additional oxygen vacancies or lattice defects that must act as trapping centers for the photogenerated electrons.

V. CONCLUSIONS

The effect of white-light illumination on the transport properties of partially oxygen-depleted YBCO ($x \approx 6.5$, $T_{c,mid} \approx 52$ K) depends strongly on the temperature at which the photodoping experiment is performed. At low temperatures, photoexcitation leads to the enhanced carrier concentration *and* mobility. At temperatures above roughly 250 K, μ is *reduced* after long illumination times, whereas n is *even more* enhanced, as compared to low temperatures. Thus, prolonged photodoping at different temperatures generates in YBCO, metallic states with different electronic properties.

From the observed asynchronous behavior of the Hall quantities, we conclude that the photoassisted oxygen-ordering and charge transfer processes from CuO_2 planes to CuO chains are the main contributors to the PPC effect, but their contributions are qualitatively different and strongly depend on the temperature of the photodoping process. The *oxygen-ordering* mechanism is mainly responsible for the changes of μ, while the *charge transfer* acts mainly on n. We have also shown that the T_c enhancement due to photodoping is a function of the carrier concentration rather than of the mobility.

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