Disorder, metal-insulator crossover and phase diagram in high-\(T_c\) cuprates

F. Rullier-Albenque\(^1\) (a), H. Alloul\(^2\), F. Balakirev\(^3\) and C. Proust\(^4\)

\(^1\) Service de Physique de l’Etat Condensé (CNRS URA 2464), CEA Saclay - 91191 Gif-sur-Yvette cedex, France
\(^2\) Laboratoire de Physique des Solides, UMR CNRS 8502, Université Paris-Sud - 91405 Orsay, France
\(^3\) National High Magnetic Field Laboratory, Los Alamos National Laboratory - Los Alamos, NM 87545, USA
\(^4\) Laboratoire National des Champs Magnétiques Pulsés, UMR CNRS-UPS-INSA 5147 - Toulouse, France

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Abstract – We have studied the influence of disorder induced by electron irradiation on the normal-state resistivities \(\rho(T)\) of optimally and underdoped \(\text{YBa}_2\text{Cu}_3\text{O}_x\) single crystals, using pulsed magnetic fields up to 60 T to completely restore the normal state. We evidence that point defect disorder induces low-\(T\) upturns of \(\rho(T)\) which saturate in some cases at low \(T\) in large applied fields as would be expected for a Kondo-like magnetic response. Moreover, the magnitude of the upturns is related to the residual resistivity, that is to the concentration of defects and/or their nanoscale morphology. These upturns are found quantitatively identical to those reported in lower-\(T_c\) cuprates, which establishes the importance of disorder in these supposedly pure compounds. We therefore propose a realistic phase diagram of the cuprates, including disorder, in which the superconducting state might reach the antiferromagnetic phase in the clean limit.

Introduction. – One of the most exotic aspects of superconductivity in correlated electron systems remains its interplay with magnetism. In the so-called “generic” phase diagram of the cuprates, a dome-shaped superconducting region is usually shown separated from an antiferromagnetic phase which is insulating in the undoped compounds. A metal-insulator crossover (MIC) has been initially assigned to the doping at which low-\(T\) upturns of \(\rho(T)\) appear. Those usually occur as logarithmic divergences of the resistivity, \(i.e.\ \rho(T) \sim -\log T\), that cannot be explained by usual electron localization theories. This has been mainly evidenced in low-\(T_c\) compounds \(T_c < 40\) K \(\text{La}_{2-x}\text{Sr}_x\text{CuO}_4\) (LSCO) \([1,2]\), \(\text{Bi}_2\text{Sr}_2-x\text{La}_x\text{CuO}_{6+\delta}\) (La-Bi2201) \([3]\) and \(\text{Bi}_2\text{Sr}_2\text{CuO}_6\) \([4]\) for which the highest available magnetic fields are sufficient to suppress superconductivity. In the underdoped regime of these compounds the \(-\log T\) behavior has been often considered as an intrinsic feature induced by the applied field and interpreted as a signature of a competing order that coexists with superconductivity \([5,6]\). As static antiferromagnetic (AF) order has been found to be also induced by high magnetic field in LSCO \([7]\), it has even been suggested that the ground state of these compounds for \(H > H_{c2}\) would be an AF insulator.

On the other hand, a zero-field superconducting-insulator transition can also be induced in the cuprates by the introduction of Zn at the Cu site of the CuO\(_2\) planes, with a similar \(-\log T\)-dependence of the resistivity \([8–10]\). This has been taken as an indication that the “insulating” behaviors induced by the magnetic field or by disorder have a common origin \([11,12]\). However, as it is now often suggested that defects are at the origin of low-\(T_c\) values \([13,14]\), it remains very difficult to distinguish the influence of the applied field from that of disorder.

In this letter we address this issue by studying a clean compound in which defects were intentionally introduced in a controlled manner. As a starting material we have chosen YBCO, which has been shown by NMR to be a very clean compound \([15]\). We have further established recently that underdoped YBCO\(_{6.6}\) displays a metallic behavior under high magnetic fields down to 1.5 K \([16]\). We have previously shown that low-\(T\) electron irradiation is a reliable means to introduce point defects, presumably oxygen and copper vacancies, in the CuO\(_2\) planes of

\(^{(a)}\)E-mail: florence.albenque-rullier@cea.fr
the cuprates [17,18]. In YBCO$_7$ superconductivity is depressed in a quantitatively similar manner to Zn substitution, without changing the hole doping. We show here that the introduction of defects by electron irradiation in optimally (OP) or underdoped (UD) YBCO always induces a MIC which is better revealed by fully restoring the normal state with high magnetic fields. For low defect content, we find that the upturn contributions to the resistivity display the main features expected for the Kondo effect in dilute alloys. We reinforce then previous suggestions based on magnetic and transport properties [19,20] and demonstrate that disorder is responsible for the apparent “insulating” behavior. By introducing nanoscale inhomogeneities, we observe that $\Delta \rho_{2D}$ still displays a $-\log T$-dependence but with a much steeper slope than for point defects. The comparison with the results reported in “pure” low-$T_c$ compounds leads us to suggest that the magnitude of the upturns is governed by the length scale of the disorder.

**Samples and techniques.** The single crystals were grown using the standard flux method. Contacts with low resistivity were achieved by evaporating gold pads on the crystals on which gold wires were attached with silver epoxy. Subsequent annealings were performed in order to obtain crystals with oxygen content around 7 and 6.6. The characteristics of the samples used in this study are described in table 1.

The irradiations were carried out with MeV (usually 2.5 MeV) electrons in the low-$T_c$ facility of the Van de Graaff accelerator at the Laboratoire des Solides Irradiés (Ecole Polytechnique, Palaiseau, France). The samples were immersed in liquid hydrogen and the electron flux was limited to $10^{14}$ e/cm$^2$/s to avoid heating of the samples during irradiation. The thicknesses of the samples were very small compared to the penetration depth of the electrons, ensuring a homogenous damage throughout. After irradiation, the samples were warmed up to room $T$ then cycled back to low $T$ and $\rho(T)$ was measured simultaneously using a standard four-probe dc technique.

As long as $\rho(T)$ after irradiation and annealing at room $T$ is identical to that observed in situ for lower irradiation fluences, we can assert that the resistivity is only sensitive to the concentration $n_d$ of randomly distributed in-plane defects. This has been the case for all samples of table 1 except O$_{6.6}$-C and -D and was thus confirmed later.

Pulsed fields up to 60 T were used in Los Alamos (LA) or in Toulouse (T) with the field applied along the c-axis in order to better suppress SC. The durations of the pulses have imposed the ac current frequencies of the measurements which vary from 10 kHz for long pulses (150 ms), to 250 kHz for short pulses (20 ms). In the latter case, we have taken particular care to control the absence of eddy current heating by comparing the up and down field sweep data or by using pulses with different peak fields.

**High-field measurements and data analysis.** Figure 1 shows the $\rho(H)$ curves for two irradiated samples.

One can see that a 60 T magnetic field is hardly sufficient to suppress SC below 15 K in the UD sample with $T_c \sim 25$ K (fig. 1a) and that a 35 T field is still needed for the OP sample with $T_c = 1.9$ K (fig. 1b).

The magnetoresistance in the highly disordered OP sample is nearly zero, so the resistivity value at 40 T can be taken as the normal-state value for $T < T_c$. This is also valid for the most irradiated O$_{6.6}$-C sample. However, in some samples such as that of fig. 1a or O$_{6.6}$-B1, for which the $\rho(H)$ curves were reported in [16], the high-field magnetoresistance is not negligible. We have thus extrapolated the high-field data down to zero field using the fact that the magnetoresistance increases as $H^2$ in the normal state [16]. In any case the $\rho(T)$-dependences

| Sample | $T_c$ | $\rho_0^{2D}$ | $n_d$ | Magnet       |
|--------|-------|--------------|------|-------------|
| O$_7$-A | 30 K  | 2.3 kΩ/□    | 3.9% | T-60T-126 ms |
| O$_7$-B | 1.9 K | 4.8 kΩ/□    | 8.2% | LA-45T-500 ms |
| O$_{6.6}$-P | 57 K  | 0.6 kΩ/□ | -    | T-60T-126 ms |
| O$_{6.6}$-A1 | 25 K  | 3.7 kΩ/□ | 1.6% | LA-60T-20 ms |
| O$_{6.6}$-A2 | 25 K  | 3.7 kΩ/□ | 1.6% | T-60T-126 ms |
| O$_{6.6}$-B1 | 6.8 K | 6.1 kΩ/□ | 2.8% | T-60T-126 ms |
| O$_{6.6}$-C | 3.5 K  | 8 kΩ/□ | ~4%  | LA-60T-20 ms |
| O$_{6.6}$-D | -   | 12.4 kΩ/□ | ~6%  | zero field |

Table 1: Characteristics of the samples. The residual resistivities $\rho_0^{2D} = 2\rho_0/c$ (with $c$ the c-axis lattice spacing) have been determined by fitting the high-$T_c$ parts of the $\rho(T)$ curves as explained in the data analysis. The defect content $n_d$ has been estimated from the increase of $\rho_0$ with electron fluence [21]. O$_{6.6}$-A1 and O$_{6.6}$-B1 correspond to the same single crystal irradiated at two different fluences.

Fig. 1: Resistivity $\rho$ vs. magnetic field at various temperatures for (a) YBCO$_{6.6}$-A2 and (b) YBCO$_7$-B.
where $\rho_0$ and $\Delta \rho(T)$ are, respectively, the residual resistivity and the upturn defect contribution and $\rho_i(T)$ is the $T$-dependent resistivity of the host, which is seen to be nearly unaffected by disorder (so $\rho_i(0) = 0$). In order to extract the defect contribution $\Delta \rho(T)$, it is necessary to extrapolate the high-$T$ part of the resistivity down to low $T$. In the case of $O_7$-$B$ a linear dependence of $\rho_i(T)$ was used to extract $\rho_0$ and $\Delta \rho(T)$. For YBCO$_{0.6}$ one can see in the inset of fig. 2 that the $T^2$-dependence of $\rho_i(T)$ which is observed in the pure crystal in the range $\sim 60-170$ K [16] is still observed above $\sim 100$ K in the irradiated samples. The $\rho_0$ values were determined by extrapolating this $T^2$-dependence down to 0 K, as indicated in fig. 2. The corresponding data for $\Delta \rho(T)$ as obtained from eq. (1) are found to become significant below temperatures $T_{dev}$ that slightly increase with increasing defect content (arrows in fig. 2).

The values of $\Delta \rho(T)$ normalized to $\rho_0$ are plotted in fig. 3a vs. $\log T$. We have analyzed the results obtained in low-$T_c$ cuprates LSCO [2] and La-Bi$_2$2201 [3] as well as in the lightly doped YBCO$_x$ with $x$ ranging from 6.5 to 6.3 [22,23] with the same method. We used a linear $T$-dependence for $\rho_i(T)$ in the case of LSCO at optimal doping and a $T^2$-dependence in the case of all the underdoped compounds. We obtain the results of figs. 3b and c which display a quantitative analogy with our data of fig. 3a. This allows us to conclude that the resistivity upturns are related to $\rho_0$, that is to the presence of disorder even in the “pure” cuprates. As will be discussed below the differences between the cuprate families can be assigned to distinct nanoscale disorder.

“Kondo” analysis for low defect content. – Let us now discuss quantitatively the $\Delta \rho/\rho_0$ curves for irradiated samples labeled A and B in fig. 3a. They display a $-\log T$-dependence at high enough $T$, but with a downward deviation below $\sim 30$ K in samples $O_{6.6}$-$A$ and $O_{7}$-$B$ (see footnote 1). This saturation cannot be related to superconductivity as we have seen in fig. 1 that the applied field fully restores the normal state in these cases. Thus, we can conclude that the resistivity upturns and saturations observed here are not signaling a metal-insulator transition but are associated with an inelastic scattering on the point defects.

It is therefore tempting to relate these effects with the Kondo-like behavior observed by NMR for the local-moment susceptibility induced by spinless impurities like Zn or Li [19]. We had indeed shown [20] that for less irradiated $O_{6.6}$ samples the $\Delta \rho(T)$ taken in the irradiation set-up scaled linearly with the defect content as long as $\rho_0^2 < 5 \, $k\, \Omega/\square$. In such zero-field experiments it was impossible to extract the actual $T$-dependence of $\Delta \rho$ because of the proximity of $T_c$. When re-analyzing these data with the procedure used here to subtract $\rho_i(T)$, we get the very good scaling of $\Delta \rho$ with $\rho_0$ displayed in fig. 4a.

1One cannot exclude that such a deviation might also exist for $O_{6.6}$-$B$ as data acquisition was done above 2 K in this experiment.
Fig. 3: Defect contribution to $\rho(T)$ normalized by the residual resistivity $\rho_0$ vs. $T/T_{dev}$: (a) for the irradiated YBCO samples that display an upturn (symbols as in fig. 2). A clear MIC is only visible on the sample $O_{6.6}$-D in which superconductivity has been totally suppressed by irradiation; (b) For LS$_x$CO and La$_x$Bi-2201. These data were taken from ref. [2] with $T_{dev} = 65$ and 70 K, respectively for $x = 0.15$ and 0.08 and from ref. [3] with $T_{dev} = 80$ and 25 K for $x = 0.84$ and 0.76; (c) For pure UD YBCO with $x$ ranging from 6.5 to 6.35 [22] and $x = 6.3$ [23]. Here $T_{dev} = 115, 140, 155$ and 160 K, respectively from 6.5 to 6.3. The dotted line, which is the high-$T$ fit of the $\log T$ behavior in panel (a), is repeated in panels (b) and (c) for comparison. (*) Here the normalization by $T_{dev}$ helps to compare quantitatively the data for different samples.

Fig. 4: Evolution of the defect contribution to the resistivity with defect content and nanostructure in $O_{6.6}$ samples. (a) For increasing point defect content: comparison of the $\Delta \rho/\rho_0$ data vs. $T/T_{dev}$ obtained in high field in $O_{6.6}$-B$_1$ -A$_2$ with those measured in situ in zero field in another $O_{6.6}$ sample (small blue squares). These latter are labelled by increasing numbers corresponding, respectively, to $\rho^{2D}_0$ values of 1.5, 2.2, 3.0, 3.8, 4.8 kΩ/□. (b) For clustered disorder: comparison of the $\Delta \rho/\rho_0$ data vs. $T/T_{dev}$ obtained in high field in $O_{6.5}$-B$_1$ and -C, with those measured in situ in zero field in the same $O_{6.6}$ sample (small blue squares) after annealing and further irradiation. These latter are labelled by increasing numbers corresponding, respectively, to $\rho^{2D}_0$ values of 3.3, 4.2, 6, 8.5 kΩ/□.

All the features above: i) proportionality to defect content, ii) $-\log T$-dependence and iii) saturation at low $T$ in some cases are the well-known benchmarks expected for Kondo inelastic spin-flip scattering effects induced by magnetic impurities in classical metals. In UD YBCO$_{6.6}$ for which the “Kondo” temperature is very low [19], the spin-flip scattering should be suppressed by magnetic fields. However, precise studies of the magnetoresistance are not possible as it is difficult to separate the suppression of superconductivity from the normal-state field variation of $\rho$. The downward deviation from the $-\log T$-dependence observed below 30 K in the $O_{6.6}$-A samples might be speculatively associated with a saturation of the magnetization, as it occurs for $\mu_B H \sim k_B T$ (effective moment of $\sim 1 \mu_B$ [24]). Let us note here that in under-doped La-Bi2201 in which 2.2% Zn totally suppressed SC, a negative magnetoresistance has been reported, and interpreted by a Kondo scattering due to the Zn-induced local moments [10]. In the case of Li-substituted optimally doped YBCO$_7$ samples, NMR studies have shown that the susceptibility of the moments induced by Li impurities, which behave as for Zn, displays a $\Theta = k_B T$ variation, $\Theta \sim 100 K$ being possibly associated with a Kondo temperature [19]. In that case one would expect a saturation of the resistivity at $T \leq \Theta$. But attempts to control this possibility could only be performed for a large concentration of defects in order to suppress SC. Indeed the $-\log T$-dependence observed in $O_7$-B displays a saturation around 2 K and would be compatible with $\Theta \approx 10 K$. Such a reduction of $\Theta$ could be related to strong interactions between defects (here $n_d \sim 8\%$).

Compared to the Kondo effect found in classical metals, the situation investigated here is somewhat unusual as the local-moment formation and its screening result from the same bare interactions [24]. Recent calculations which consider AF fluctuations in a Fermi liquid approach...
have shown that impurities might induce “Kondo-like” upturns of resistivity when the system is close to the AF quantum critical point [25]. Although this approach might be questionable in underdoped cuprates, it provides a first attempt to explain resistivity upturns induced by spinless impurities in these strongly correlated systems.

**Influence of the disorder morphology**. – It is clear from the three panels of fig. 3 that the high-$T$ logarithmic slope of $\Delta \rho(T)/\rho_0$ has the same magnitude in La-Bi2201 and in YBCO$_x$ as in our YBCO crystals with a moderate defect content. This leads us to suggest that point defects are responsible for the apparent MIC in these compounds. As previously noticed [3] the “insulating” behavior in LSCO samples is much stronger than in La-Bi2201. This could be associated with the formation of inhomogeneous states such as stripes and/or peculiar disorder dominated by tilts of the CuO octahedra [26,27].

In order to clarify the role of the defect microstructure, we have induced a clustering of the defects in sample O$_{6.6}$C. This sample was irradiated by 1.8 MeV electrons at a fluence which allowed to fully suppress its $T_c$. Annealing at high $T$ (400 K) that favors defect migration on large distances, results in clustering of defects. Meanwhile superconductivity was recovered with $T_c \sim 3.5$ K. One can see in fig. 4b that the $\Delta \rho(T)/\rho_0$ curve deviates from the dependence observed in the other samples towards a new $-\log T$ with a slope about three times larger and no sign of saturation down to 0.5 K.

To confirm the generality of this observation, we have considered an UD sample which had been aged at room $T$ for two years after irradiation, so that clustering of defects did occur. In such a sample further irradiation does not simply introduce additional random point defects but provokes an evolution of the nanostructure of the disorder. As seen in fig. 4b, $\Delta \rho(T)/\rho_0$ progressively deviates from curve B to eventually reach curve C when $\rho_0^{2D} \sim 8$ kΩ/□. A clear insulating behavior is only observed when $\rho_0^{2D}$ exceeds $\sim 10\,$kΩ/□ (curve D in fig. 3a). The data displayed in fig. 3b for the two LSCO samples exactly coincide with curve C. This is also found when analyzing the data reported for LS$_{0.15}$CO with 2% Zn [12] and for LS$_{0.15}$CO with 4% Zn [8]. We might then conclude that although different types of disorder produce qualitatively similar behavior, nanostructured disorder induces larger upturns in $\Delta \rho(T)/\rho_0$.

**Realistic phase diagram including disorder**. – The results obtained here allow us to propose in fig. 5 an extended 3D version of the $(T,n_h)$ phase diagram of the cuprates in which disorder is introduced as the third axis parameter. While disorder only affects moderately the AF phase, it depresses $T_c$ much more in the underdoped case than in the overdoped one [28–30], hence the asymmetric shrinking of the superconducting dome. The $T_c$ trajectories followed in this 3D diagram for our electron irradiated samples are shown by full red lines. As we have seen hereabove, the existence of $\rho(T)$ upturns does not imply a $T = 0$ insulating state, but is associated at low defect content with inelastic scattering processes. The actual MIC (blue line in fig. 5) takes place when a divergence of $\rho(T)$ is detected, which only occurs for sufficient disorder.

In this 3D representation we have ranged the $(T,n_h)$ phase diagrams of the various pure cuprate families with respect to their optimal $T_c$ values, which can be considered as a reasonable physical quantity representative of the intrinsic disorder in these systems. The MIC locations for La-Bi2201 and LSCO were determined as for the irradiated YBCO. We can see that the La-Bi2201 SC dome and MIC match those induced by electron irradiation in YBCO. On the contrary, the fact that for LSCO the SC dome only qualitatively fits in this diagram can be assigned to the different nanoscale disorder associated with the local stripe ordering favored for $x \approx 0.12$. We have also sketched the SG phase which extends with disorder in a similar manner as the MIC.

As for the four families of rather clean cuprates Hg-1201, YBCO, Bi2212 and Tl-2201, they have been ranging as well here with respect to their optimal $T_c$, respectively 97, 93, 90 and $\sim 85\,$K [33]. At this fine level,
the ordering might not be representative of the residual disorder as it does not take into account the influence of the number of CuO$_2$ layers (and their buckling).

Let us further point out that the representation done here is yet somewhat simplified as a constant disorder is assumed throughout the phase diagram for each cuprate family. This is less critical for the lower-$T_c$ cuprate families than for the cleanest ones such as YBCO, where the hole content and chain disorder are fully interconnected. The data of fig. 3c allow us to indicate that a MIC occurs for $x \approx 0.35$ for disordered chain samples. For this oxygen content an AF phase is usually detected. So, an essential consequence of our study is that the intrinsic properties in this family are only accessible on reasonably well-ordered compounds such as YBCO$_{7_2}$, YBCO$_{6.6}$ or for the chain ordered orthorhombic YBCO$_{6.5}$ used recently to probe the Fermi surface by bulk transport measurements [34].

Conclusion. – Here we have used the progressive electron irradiation of the clean YBCO system to provide a true control of the disorder parameter for specific dopings in the 3D phase diagram that we propose. This has allowed us to demonstrate that the apparent MIC detected so far in cuprates is always driven by the existing disorder. We have thus unravelled the confusion brought about by the report of similar $-\log T$-dependences in the “insulating” state of all these compounds. Those are due for low defect contents to scattering processes intimately linked with the strong-correlation effects and incoherent magnetism. These results, together with the Curie-Weiss–like susceptibilities found by NMR for in-plane defects display a strong similarity with the Kondo effect in dilute alloys. Although the microscopic picture does differ, and no detailed theoretical justification has been proposed so far, the data can be analyzed in the frame of a Kondo-like phenomenology. We have also shown that the magnitude of the $-\log T$ variation rather depends on the morphology of the disorder.

So the physical properties of most of the “pure” cuprates families are affected by their specific disorder, its influence being quite critical in the underdoped pseudogap phase, for which those electron correlation effects are most important. We have evidenced here that, even if the true microscopic character of the disorder is not fully characterized in a given cuprate family, the actual value of its optimal $T_c$ and the hole content at which a MIC is detected are direct measures of the disorder, that allow us to position the families on the 3D phase diagram.

Contrary to the proposal that disorder and spin glass behavior is generic to the physics of the CuO$_2$ planes [35], an essential conclusion of our study is that the intrinsic properties are only accessible on reasonably well-ordered compounds. The very shape of the $(T, n_h)$ phase diagram for vanishing disorder could correspond to a merging of the SC and AF lines as found in other correlated electron systems such as heavy fermions and organic superconductors. Such a speculation is illustrated in fig. 5, by the hypothetical trend of the 3D phase diagram for vanishing disorder.

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