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Observation of superconductivity in $Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$

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$Eu_{2-z}Ce_zSr_2Cu_2TiO_x$ with $x \approx 10$ is shown to exhibit predicted superconductivity, as detected (i) by a sudden drop in surface resistance at $T_c \approx 22$ K, (ii) by vortex dissipation in an applied field at temperatures less than T_c, and (iii) by measurements of diamagnetism (Meissner effect). This is our fourth successfully predicted high-temperature superconductor, based on a model with the primary superconducting layers in the charge-reservoirs, not in the cuprate planes.

I. INTRODUCTION

One of the primary tasks of a successful theory of high-temperature superconductivity is the prediction of which materials will superconduct (and which will not). We are aware of only three materials (i) that originally did not superconduct, (ii) that were predicted to be capable of superconducting at high temperatures, and (iii) that were subsequently shown to superconduct: $PrBa_2Cu_3O_7$,[1–11] $Gd_{1.4}Ce_{0.6}Sr_2Cu_2TiO_{10}$,[12–14] and $Pr_{1.5}Ce_{0.5}Sr_2Cu_2NbO_{10}$.[15–17] (All were first *granular* superconductors; subsequently, refined preparation techniques yielded *bulk* superconducting $PrBa_2Cu_3O_7$ [6].)

Here we report a fourth superconductor, one which was predicted to superconduct on the basis of the charge-reservoir oxygen model,[15,18,19] and, until the present work, has not been observed to superconduct. This superconductor is $Eu_{2-z}Ce_zSr_2Cu_2TiO_x$ with $x \approx 10$ (Fig. 1) and its superconductivity has been detected (i) by observation of a sudden drop of surface resistance in zero applied magnetic field $\mathbf{H}$ at $T_c \approx 22$ K (Ref. 20) (Fig. 2), (ii) by vortex dissipation in applied fields $\mathbf{H}$ at temperatures less than T_c (Fig. 2), and (iii) by a Meissner effect (Fig. 3). The dc resistance also shows evidence of granular superconductivity (Fig. 4). Thus $Eu_{2-z}Ce_zSr_2Cu_2TiO_{10}$ is definitely a *granular* superconductor from a class of materials that has previously yielded other granular superconductors: $Gd_{1.4}Ce_{0.6}Sr_2Cu_2TiO_{10}$ (Refs. 12–14) and $Pr_{1.5}Ce_{0.5}Sr_2Cu_2NbO_{10}$.[15–17] (This means that at least $Nd_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$ and $Sm_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$, should also superconduct, if fabricated correctly.)

II. EXPERIMENTAL DETAILS

Ceramic samples with nominal composition $Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_x$ were prepared using a solid state reaction technique. Prescribed amounts of Eu_2O_3, CeO_2, TiO_2, $SrCO_3$, and CuO were mixed, pressed into pellets, and preheated at 1000 °C for about 1 day in the presence of flowing oxygen at atmospheric pressure. The reaction products were cooled, reground, and sintered at 1050 °C for 72 h in a slightly pressurized oxygen atmosphere, and then furnace cooled to ambient temperature.

Powder x-ray diffraction measurements confirmed the purity of the compounds (~90%) and indicated that the material has a tetragonal-type structure with lattice parameters $a = 3.863(1)$ Å and $c = 28.50(5)$ Å. The extra diffraction peaks probably belong to the $Eu_2Sr_2Cu_2Ti_2O_{11}$ phase (the so-called 2222 phase); all attempts to eliminate this phase were unsuccessful.

The dc magnetic measurements on solid ceramic pieces in the range of 5 to 100 K were performed in a commercial [21] superconducting quantum interference device magnetometer

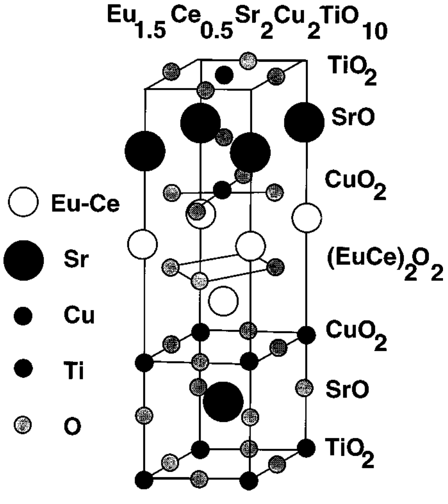

FIG. 1. Crystal structure of $Eu_{2-z}Ce_zSr_2Cu_2TiO_{10}$.

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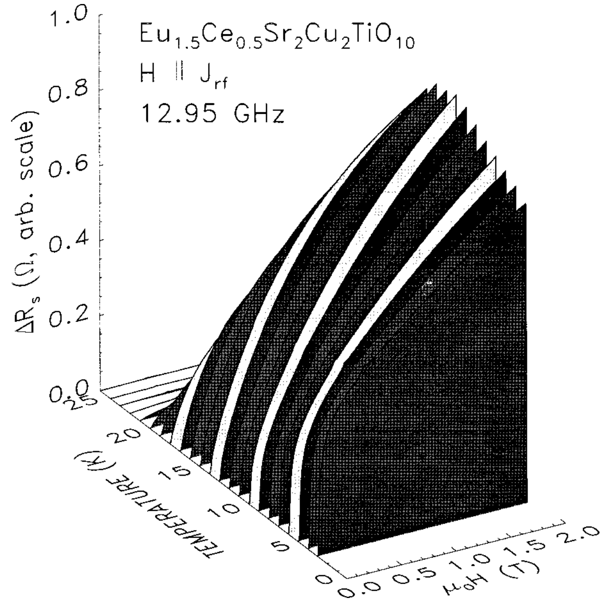

FIG. 2. Measured $Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$ microwave dissipation, $\Delta R_S(\mathbf{H},T)$, as a function of temperature and applied field with $\mathbf{H}\|\mathbf{J}_{\mathrm{rf}}$. The field-dependent dissipation is typical of what is observed for high-temperature superconductors. The broad signal in $\Delta R_S(\mathbf{H},T)$ for $T<T_c$ is due to vortex dissipation.

(SQUID). The magnetization was measured by two different procedures: (i) the sample was zero-field cooled to 5 K, a field was then applied, and finally the magnetization was measured as a function of temperature and (ii) the sample was field cooled from above 250 to 5 K and the magnetization was measured. Resistivity measurements were performed using the standard four-point technique.

Mössbauer spectroscopy studies of ^{151}Eu were carried out using a conventional constant-acceleration spectrometer and a 50 mCi $^{151}SmF_3$ source, and the isomer shifts were reported with respect to this source. Mössbauer spectroscopy performed at 300 K on ^{151}Eu shows a single narrow line of width 2.15(1) mm/s. The fit yields an isomer shift of 0.01(2) mm/s and a quadrupole splitting in the range of 5.58 mm/s. The small isomer shift values obtained indicate that the Eu ions are trivalent with a nonmagnetic $J=0$ ground state.[22]

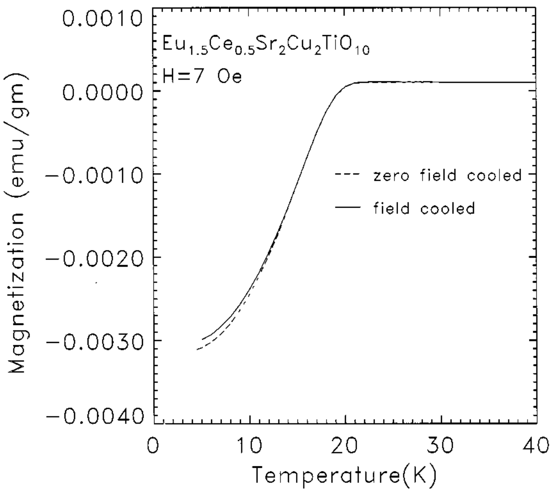

FIG. 3. Bulk magnetization (in emu/g) versus temperature T (in K) of $Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$ illustrating the onset of a Meissner effect at $T\approx 22\,\mathrm{K}$. The upper line represents the field-cooled material.

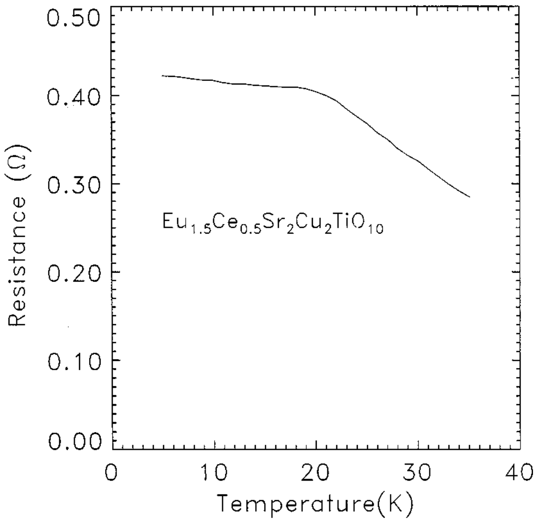

FIG. 4. The dc resistance (in ohm) of $Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$ as a function of temperature (in K).

III. SUPERCONDUCTIVITY

$Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$ is a type-II superconductor according to the following measurements.

(i) Our first method of detecting the superconductivity was to measure the magnetic-field induced change in the microwave surface resistance, $\Delta R_S \equiv R_S(\mathbf{H},T)-R_S(\mathbf{H}=0,T)$, of $Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$ as a function of both temperature T and applied magnetic field $\mathbf{H}$ (Fig. 2). The microwave frequency was 12.95 GHz. Figure 2 displays data which are typical of measurements on other high-temperature superconductors,[23] data taken for $\mathbf{H}\|\mathbf{J}_{\mathrm{rf}}$, where $\mathbf{J}_{\mathrm{rf}}$ is the rf current density. These data show an onset in the field-induced change of the surface resistance $\Delta R_S(\mathbf{H},T)$ at $T_c\approx 22\,\mathrm{K}$, which is characteristic of vortex dissipation at and below T_c in a type-II superconductor.[20]

(ii) A bulk Meissner effect, corresponding to a 6% Meissner fraction, is observed at superconducting temperatures (Fig. 3).

(iii) As expected, for typical p-type doping, the Eu is in the Eu^{+3} charge state, as determined by Mössbauer isomer shift measurements.

Furthermore the dc resistance of the sample becomes flat as a function of temperature below T_c, as indicated in Fig. 4. We speculate that this behavior is a consequence of a granular material that has not reached the percolation threshold of its superconductivity. The microwave data are unambiguous in detecting superconductivity in a fraction of the sample. Because we have a finite Meissner effect, it is clear that we are rather close to percolation. Clearly $Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$ is a type-II granular superconductor.

IV. CRITICAL TEMPERATURE AND VALENCE

The superconducting critical temperature observed for $Eu_{1.5}Ce_{0.5}Sr_2Cu_2TiO_{10}$ was ~22 K, and should be compared with the prediction of the empirical rule $T_c=(15\,\mathrm{K/Å})d$,[15] which gives a predicted value of about 30 K for optimally doped material. We do not know if our material is optimally doped, but suspect that it is not—because no effort has been made to optimize the Ce doping. Hence, although the agreement of the measured T_c with the predicted empirical rule is reasonably good, improved doping is likely to increase T_c to a value nearer the prediction.

Ti is known to prefer the Ti^{+4} valence state, which appears to be consistent with the potential at its ideal site. If this charge state does indeed occur, then the material is different from some of its homologous compounds, such as $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{RuO}_{10}$, which has its Ru in the Ru^{+5} state, according to x-ray absorption spectroscopy[24] and Mössbauer measurements.[25] This valence difference would also imply that the two compounds have significantly different charge distributions, although both superconduct.

V. EVIDENCE THAT Eu21-4 IS NOT PRESENT AS THE MAIN MINORITY SUPERCONDUCTING PHASE

The 22 K superconductor $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ can be viewed as a superlattice of $\mathrm{Eu}_{1.5}\mathrm{Ce}_{0.5}\mathrm{CuO}_4$ (Eu21-4 with $z=0.5$) and the layers $/\mathrm{SrO}/\mathrm{TiO}_2/\mathrm{SrO}/\mathrm{CuO}_2/$. Potentially $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ could be present as a superconducting impurity phase. There are several facts which are inconsistent with such an interpretation, however.

First, in order to produce superconductivity in $R_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ materials, one must anneal them at $\sim 950\,°\mathrm{C}$ under a reducing atmosphere of flowing Ar, and then quench them to room temperature in the same atmosphere. Otherwise, bulk superconductivity is not achieved. These conditions have not been met in the samples discussed here: For example, in producing $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$, oxygen at a higher pressure than 1 atm was employed.

Second, the x-ray spectrum features extra peaks which do not match those expected for a $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ minority phase of $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$; they do match to $\mathrm{Eu}_2\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{Ti}_2\mathrm{O}_{11}$, even better than to $\mathrm{Eu}_2\mathrm{CuO}_4$.

Third, the observed T_c of the $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ is too high to be caused by $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$: $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ has a critical superconducting transition temperature of only 7.5 K,[26,27] too low to account for the observation of 22 K superconductivity of $\mathrm{Eu}_{1.5}\mathrm{Ce}_{0.5}\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ as due to a $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ substructure.

Fourth, the doping z is inconsistent with the superconductivity being due to $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$: $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ superconducts for $z=0.5$, and $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ superconducts optimally for $z=0.15$. Moreover, the possibility that z exceeds 0.2 or 0.3 is effectively ruled out by the fact that Ce has limited solubility in $R_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ compounds, where R is a rare-earth ion.[28]

Fifth, the relations of $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ and $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ to $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ and $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ lend support to the picture that predicted the superconductivity of both $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ and $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$. We studied $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ first, because $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ does not superconduct, making it unnecessary for us to prove that the superconductivity we observe in $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ is caused by $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$, which does not superconduct. ($\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ has $T_c\approx 11\,\mathrm{K}$.[14]) Indeed the charge-reservoir oxygen model's argument, that $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ fails to superconduct because Gd (i) is an $L=0$ (and $J\neq 0$) ion, (ii) is unaffected by crystal-field splitting, and hence (iii) is a Cooper pair breaker, also implies that the superconducting condensate of $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ cannot be in the cuprate planes: those planes are adjacent to the Gd, and should not superconduct (because of Gd pair-breaking) if the superconducting condensate is scattered by the Gd, as it is in $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$, which has the same local structure as in $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$. Consequently, the cuprate planes of $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ cannot carry the primary superconducting condensate. Because $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ is isostructural to $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$, its cuprate planes must not carry the primary superconductivity either. The superconducting condensate must be adjacent to the rare-earth R in $R_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ compounds, but remote from it in $R_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$. This is consistent with previous assignments of the superconductivity (i) to the vicinity of both the rare-earth site and the interstitial oxygen in the $R_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ compounds[29] and (ii) to the SrO layers of the $R_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ materials.[14,19] It is also consistent with differences in critical temperatures, such as observed in $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ and in $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$. [We are unaware of any explanation of the nonsuperconductivity of $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ (Ref. 26) and $(\mathrm{La}_{1-x}\mathrm{Gd}_x)_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ (Ref. 30) in terms of a cuprate-plane model.]

The differences between non-superconducting $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ and superconducting $\mathrm{Gd}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ indicate that *different* layers provide the primary superconductivity in the $R_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ homologues and in the $R_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ compounds. (Previous work has shown that the $R_{2-z}\mathrm{Ce}_z\mathrm{CuO}_4$ compounds superconduct in the vicinity of both the rare-earth and the interstitial-oxygen sites,[29] while $R_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ superconducts primarily in its SrO layers.[14,19])

Since Eu^{+3} is not a pair breaker in lowest order, we cannot conclude that the superconductivity is exclusively in the SrO layers of $\mathrm{Eu}_{2-z}\mathrm{Ce}_z\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$. However, any superconductivity in the cuprate-planes must be secondary.

VI. CONCLUSION

$\mathrm{Eu}_{1.5}\mathrm{Ce}_{0.5}\mathrm{Sr}_2\mathrm{Cu}_2\mathrm{TiO}_{10}$ superconducts with a critical temperature of $T_c=22\,\mathrm{K}$,[20] as predicted by the charge-reservoir model of superconductivity. The fact that it does superconduct, lends support to the charge-reservoir oxygen model which (i) predicted the superconductivity and (ii) assigned the primary superconducting condensate to the SrO layers (which are the charge reservoirs), rather than to the cuprate planes.

ACKNOWLEDGMENTS

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