

Testing models of the symmetry of the superconducting pairing state by low-temperature electron irradiation of an untwinned single crystal of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$

J. Giapintzakis and D. M. Ginsberg

*Department of Physics and Materials Research Laboratory, University of Illinois at Urbana-Champaign,
1110 West Green Street, Urbana, Illinois 61801*

M. A. Kirk and S. Ockers

*Materials Science Division, Argonne National Laboratory, 9700 South Cass Avenue, Argonne, Illinois 60439
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We have carried out *in situ* resistivity measurements in a high-voltage electron microscope to determine how the superconducting transition temperature T_c and the resistivity $\rho(T)$ change with low-temperature electron irradiation. We find that point defects introduced by oxygen displacements from the CuO_2 planes make the dominant contribution to the irradiation-induced suppression of T_c . We find that $dT_c/d\rho$ is $\sim -0.30 \pm 0.04 \text{ K}/\mu\Omega \text{ cm}$. Analysis of this result indicates the effect of strongly anisotropic superconductivity, and rules out isotropic *s*-wave pairing. Even though our data cannot determine whether the pairing symmetry is *s* or *d* wave, they provide a test for further theoretical developments.

INTRODUCTION

One of the most intriguing problems still facing theorists and experimentalists in high- T_c superconductivity is identifying the mechanism of superconductivity. Many theories claim to explain it, but none has gained general acceptance. These theories can be divided into two major groups, based on the suggested symmetry of the pairing state.¹ One group supports *d*-wave pairing symmetry, while the other supports *s*-wave symmetry. We need to eliminate one of these possibilities. To this end, several experiments have been carried out. Some of them, however, suffer from difficulties of experimental techniques or interpretation.^{2,3}

Recently, Radtke *et al.*⁴ have presented predictions of T_c as a function of planar resistivity caused by nonmagnetic impurities in a $T_c = 90 \text{ K}$ *d*-wave superconductor and in an isotropic *s*-wave superconductor. Based on these predictions, they suggested an experiment which could help find the symmetry of the pairing state. In this paper we report the results of this suggested experiment.

We have used electron irradiation to introduce the desired electronic scattering in a systematic and controlled way. By carefully selecting the energy of the electrons in the beam, we displaced only oxygen atoms. By irradiating at low temperatures ($\sim 90 \text{ K}$) and measuring the resistivity *in situ*, up to $T = 160 \text{ K}$, we have prevented these irradiation-induced defects from migrating and recovering or clustering. Thus, we are dealing with point defects. This technique has ensured that the calculation of the concentration of these defects is accurate. Also, by using a twin-free sample and measuring the ρ_{aa} element of the resistivity tensor, we have probed the contribution of only the oxygen defects on the CuO_2 planes. (The *a* axis is in-plane and perpendicular to the Cu-O chains.) Finally, we have separated the contribution to the

irradiation-induced suppression of T_c caused by the oxygen defects on the CuO_2 planes from that caused by the oxygen defects on the chains.

SAMPLE

High-quality single crystals of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO) were grown in yttria-stabilized zirconia crucibles by using the self-flux growth technique. The method of sample preparation (including growth and oxygenation) is described in detail elsewhere.⁵ The sample used in this study was cleaved to a rectangular shape with dimensions of $1.11 \times 0.44 \times 0.009 \text{ mm}^3$. The as-grown sample had many twin boundaries. They were removed by applying a uniaxial stress at 410°C in flowing oxygen on the hot stage of an optical microscope having crossed polarizers, as described elsewhere.⁶ The crystal was configured for *a*-axis resistivity measurements by applying the stress in the long direction. The crystal was then reannealed to relieve possible strains introduced by that procedure. The transition temperature of the annealed sample was 90.5 K , with a superconducting transition width of less than 0.8 K in an applied field of 1 Oe . The sample's resistivity at 100 K was $45.6 \mu\Omega \text{ cm}$, one of the lowest values reported in the literature.⁷

RESISTIVITY MEASUREMENT

The resistivity of the sample was measured with the four-probe technique. Gold stripes were evaporated onto the sample through a mask made of glass microslides. These stripes had a width of approximately $30 \mu\text{m}$, and they extended across the whole sample. The distance between the voltage contacts was 0.62 mm . Annealing the sample at 410°C for 24 h in flowing oxygen made the gold pads stick to the surface, and reduced the contact resistance from $\sim 1 \text{ k}\Omega$ to less than 0.4Ω .

Only the ends of the sample were mounted on two small pieces of MgO with silver paste. This was done to reduce sample heating from electron irradiation of the MgO. The MgO pieces were thermally anchored to a Cu-grid with silver paste.

Gold wires with 25 μm diameter were attached to the pads with silver paste. The width of contact of the silver paste was approximately 70 μm , larger than the 30 μm width of the annealed gold stripe. Since silver paste does not make good electrical contact to YBCO crystals without a heat treatment, the effective contact width was that of the gold stripe.

The assembled sample and holder, consisting of the YBCO crystal, the MgO pieces, and the Cu-grid were mounted in the 3-mm sample space of the side-entry, single-tilt, low-temperature specimen stage of the High-Voltage Electron Microscope (HVEM) at Argonne National Laboratory. A small amount of silver paste was used to assure good thermal contact between the sample and the stage. This sample stage has the capability of *in situ* electrical-resistivity measurements.

A flow cryostat using helium gas as a coolant was employed to reach the desired low temperatures. The sample's resistance was measured upon slow warming, which was manually controlled by regulating the flow of the cold helium gas. To avoid migration and recovery of the point defects induced by low-temperature electron irradiation, the measurements were carried out while the sample remained in the range 75 K < T < 160 K. No thermal lags were found; the resistance curve was the same when it was measured during the slow warming that we used and remeasured with fast cooling.

The data were collected by a computer-controlled dc-resistivity system, with a current of 1 mA. The sensitivity of the dc apparatus was about 20 nV. The results were insensitive to the size of the current. Each data point represents the average of 15 readings, with several current reversals per point to cancel thermoelectric effects.

All of the data were taken with the sample in a magnetic field of about 0.1 T induced by the current in the objective lens of the electron microscope.

IRRADIATION CONDITIONS

In addition to having the capability of *in situ* electrical-resistivity measurements, the HVEM has the advantages of continuous energy selection, accurate dosimetry, and easy control of the beam's position and shape.

The irradiations were performed in a vacuum of 4×10^{-7} Torr at ~ 90 K. The irradiation temperature was determined by measuring the resistance of the sample during irradiation, which took 2 h. The irradiation direction in the single crystal was ~ 10 degrees from the c -axis direction to avoid electron channeling. The dosimetry was performed by measuring the total beam current entering a removable Faraday cup located just above the sample beam. We used the microscope's low-magnification mode to spread the beam sufficiently to obtain uniform irradiation of the area of interest. The beam

was uniform along its diameter to within 95%. The diameter of the beam was slightly larger than the separation of the voltage leads (0.70 ± 0.05 mm). The flux of the beam was 2.3×10^{15} e/cm²/sec.

The energy of the electron beam, 350 keV, was chosen to selectively displace oxygen atoms. The thickness of the sample ($t = 9$ μm) was chosen to allow penetration by the electron beam, with a reasonably small average energy loss, about 10% of the initial energy.

RESULTS

In Fig. 1 we have plotted the $\rho(T)$ curves measured prior to irradiation, after low-temperature irradiation to doses of 0.98×10^{19} and 1.96×10^{19} e/cm², and following subsequent annealing to room temperature for 12 h. In Fig. 2 we display a magnification of the part of Fig. 1 near T_c , and in Fig. 3 we show a magnification of the part of Fig. 1 at high temperatures (130 K < T < 160 K).

There are several interesting features that one should note. As the irradiation dose increases T_c decreases while $\rho(T)$ increases. Following annealing to room temperature, both T_c and ρ recover, i.e., T_c increases and $\rho(T)$ decreases. Table I shows T_c and $\rho(145$ K) values at different doses and following annealing to room temperature. We have identified T_c as the temperature at which the resistance falls below our resolution. We have chosen to show the resistance at $T = 145$ K to avoid complications from thermodynamic fluctuations, which are important near T_c . Nevertheless, we could have looked at any temperature above T_c since the thermodynamic fluctuations near T_c seem not to be affected by the irradiation. The width of the transition is also unaffected by the irradiation and the subsequent annealing.

The resistivity above T_c exhibits a linear temperature dependence, both after each irradiation dose and after warm up to room temperature. Because the $\rho(T)$ curves are parallel before and after irradiation, we conclude that no detectable recovery of defects in the planes takes place between about 90 and 160 K, the temperature range over which the normal-state resistivity was measured. In Table I we display the linear fit parameters of the resistivity to the equation $\rho(T) = \rho_0 + bT$ for 130 K < T < 160 K. We always observe ρ_0 to be negative, so the resistivity in the absence of superconductivity could not remain linear all the way down to zero temperature, but must turn over to obey some higher power law.⁴

We chose to collect data for only two relatively low irradiation doses because we were interested in producing low defect concentrations so that we would be able to observe significant annealing effects upon warming to room temperature, and to simplify the interpretation of the data by avoiding interactions between neighboring defects.

DISCUSSION

Several *in situ* TEM studies suggest that the incident electron energy E_i needed to displace a chain oxygen is ~ 130 keV.^{8,9} The threshold energy absorbed to displace a chain oxygen atom is therefore $E_d(0) = 20$ eV. We have

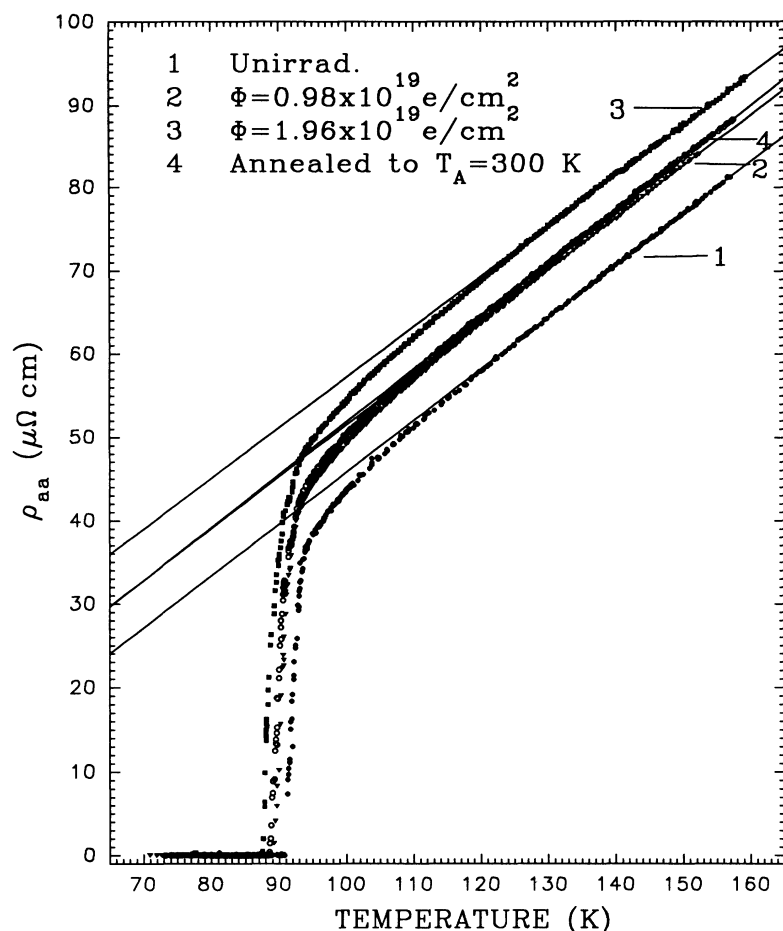


FIG. 1. $\rho(T)$ curves measured prior to irradiation, after low-temperature irradiation to doses of 0.98×10^{19} and 1.96×10^{19} e/cm², and following subsequent annealing to room temperature for 12 h. The lines are the result of the linear fitting of the data to the equation $\rho(T) = \rho_0 + bT$ for $130 \text{ K} < T < 160 \text{ K}$.

reported^{10,11} that the pinning capability of YBCO crystals, similar to the ones used in this study, was enhanced only if the energy of the incident electron beam was greater than 500 keV. We suggested that this was the threshold incident electron energy for producing displacements of copper atoms from the CuO₂ planes, so $E_d(\text{Cu}) = 25$ eV. We chose E_i to be well above the energy needed to displace oxygen atoms from the chains, to take into account the fact that E_d of the oxygen atoms on the CuO₂ planes could be higher than that of the oxygen atoms on the chains, but kept E_i below the energy required to displace copper atoms from the CuO₂ planes. By using a 350-keV electron beam we insure that only oxygen atoms were displaced, both in the chains and the planes.

To distinguish the contribution of planar-oxygen defects to the measured resistivity from the contribution of chain oxygen defects, we carried out the experiment in a crystal free of twins. The long edge of the sample (the direction of the electrical current) was along the a axis. This geometry assured that we measured the ρ_{aa} element of the resistivity tensor, to which the chains should not contribute. The increase of $\rho_{aa}(T)$ upon irradiation with 350-keV electrons enables us to set an upper limit on the incident electron energy needed to produce displacements of oxygen atoms from the CuO₂ planes. Thus, E_d (O in CuO₂ planes) ≤ 64.5 eV.

While the measured resistivity can be attributed to the planar-oxygen defects, we need to separate the contributions of the defects occurring at the two oxygen sites to the observed shift in T_c . To accomplish this, we assume that the change in T_c (ΔT_c) induced by the irradiation can be written as the sum of two components: $\Delta T_c = \Delta T_c(\text{planes}) + \Delta T_c(\text{chains})$. Each of the two components can be written as the product of T_c change per defect per unit cell times the number of defects per unit cell (n). We use the following equation to find n :

$$n^i = \sigma_d^i \Phi c^i, \quad (1)$$

where the superscript index i labels the two sites; we chose $i \equiv c$ for chains, and $i \equiv p$ for planes. σ_d^i is the cross section for producing a Frenkel pair at the i th site, Φ is the dose of the irradiation, and c^i is the number of i atoms per unit cell ($c^c = 1$ and $c^p = 4$). We use the McKinley-Feshbach approximation¹² to calculate σ_d . This approximation is sufficiently accurate in the case of light atoms ($Z < 29$) such as oxygen. As we mentioned above, the majority of studies, including one of ours,⁸ suggest that E_d (O in the chains) ~ 20 eV, but a recent study¹³ suggests that E_d (O) = 10 eV. We feel this low threshold value may not be accurate, since considerable transmission electron microscopy at 100 keV [$E_d(\text{O}) = 15$ eV] has shown no evidence for displacement damage in

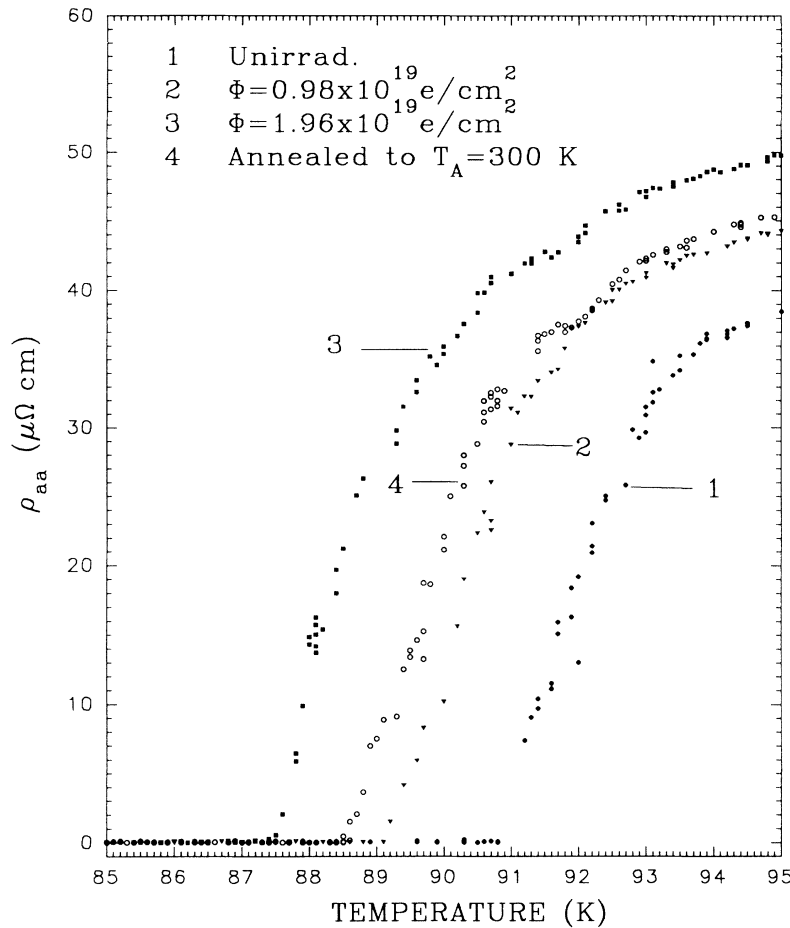


FIG. 2. Magnification of the part of Fig. 1 near T_c .

this material.⁸ Therefore we present the results from calculations in which we used $E_d(\text{O})=20$ eV. In using this value we assume the threshold energy for displacement of oxygen from the planes to be the same as from the chains. We also assume that the displacement of the apical oxygen does not play a significant role in either the planar resistivity or T_c .

Arguments supporting the latter assumption will be provided when we discuss the fractional recovery of ρ and T_c following a warmup to room temperature. We therefore calculate σ_d^i using $E_d=20$ eV and find that $\sigma_d^i(20 \text{ eV})=20.5$ b. Using Eq. (1) with this value for σ_d^i and using $\Phi=1 \times 10^{19} \text{ e/cm}^2$, we find values for n^p (the number of planar-oxygen defects per unit cell) and n^c (the number of chain-oxygen defects per unit cell): $n^p(20 \text{ eV})=8.19 \times 10^{-4}$ and $n^c(20 \text{ eV})=2.05 \times 10^{-4}$.

Previous irradiation studies using the same electron energy have indicated only a small ($\sim 5\%$) recovery in T_c upon annealing to 160 K.¹³ Our data indicate that this recovery in the normal-state resistivity must be even smaller. We were careful to minimize the migration and hence the recovery of defects (at least in the CuO_2 planes) between irradiation steps and during irradiation by measuring $\rho(T)$ only up to a maximum $T=160$ K and keeping $T_{\text{irrad}} \sim 90$ K, respectively. The concentrations of defects we have calculated should therefore be fairly accurate. Table I shows that $\Delta T_c = -1.68 \pm 0.17$ K per

$1 \times 10^{19} \text{ e/cm}^2$. The contribution of the chain-oxygen defects, ΔT_c (chains), has previously been estimated by using the results of oxygen gettering and desorption experiments.¹⁴⁻¹⁷ Those studies report a rate of decrease of T_c between 5 and about 36 K per chain-oxygen defect per unit cell. Using the maximum value of 36 K per chain defect per unit cell, we find that ΔT_c (chains; 20 eV) $= -0.007$ K for an electron dose of $1 \times 10^{19} \text{ e/cm}^2$. Since this value has much smaller magnitude than does the measured ΔT_c , we can attribute the total suppression of T_c to displacements of planar-oxygen atoms. Thus, we have experimentally confirmed the idea suggested by Weaver *et al.*¹⁸ that displacements of atoms from the CuO_2 planes dominate the suppression of T_c . (Using a 10-eV threshold energy increases the displacement cross section and the calculated defect concentration for oxygen by about a factor of 3, but it does not change this conclusion.)

Our conclusion that the planar-oxygen displacements are almost entirely responsible for degrading T_c upon irradiation is also confirmed by our following results. By analyzing our annealing data we found that the recovery fraction of T_c and $\rho(T)$ are almost the same: $[T_c(\text{ann}) - T_c(\text{irr})]/[T_c(\text{unirr}) - T_c(\text{irr})] = 0.333$ and $[\rho(\text{ann}) - \rho(\text{irr})]/[\rho(\text{unirr}) - \rho(\text{irr})] = 0.373$. (Keep in mind that ρ_{aa} probes only the CuO_2 planes.) This result indicates that the T_c suppressions caused by displace-

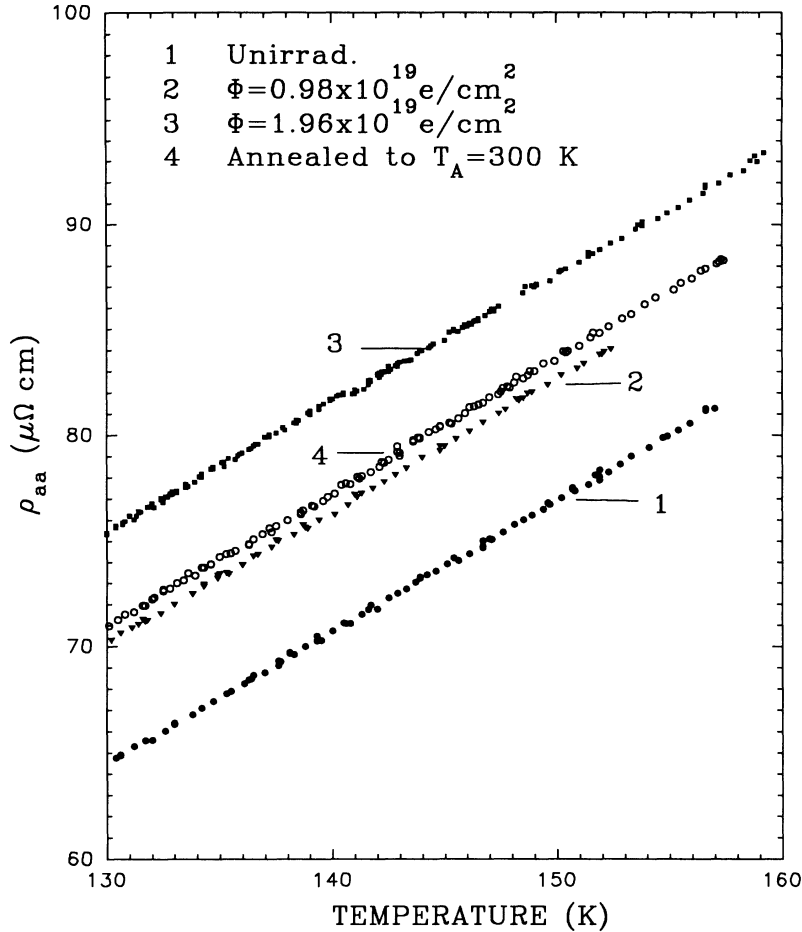


FIG. 3. Magnification of the part of Fig. 1 at high temperatures.

ments of apical oxygens and chain oxygens are not important. In addition, the linear reduction of T_c with dose would not have been observed if the chains were the dominant contribution to T_c suppression, since thermal data¹⁴ would require T_c to change in a nonlinear fashion.

There are several mechanisms leading to T_c suppression. For our experimental results, we can exclude the mechanisms involving the reduction of charge carriers and the introduction of magnetic centers. Legris *et al.*¹³ have reported that electron irradiation does not appreciably modify the number of charge carriers. (This was concluded from Hall-effect measurements of YBCO single crystals before and after irradiation with 420-keV electrons to doses higher than the ones used in our work.) Welp *et al.*¹⁹ found that the defects induced in YBCO crystals by irradiation with 1-MeV electrons to high dose are nonmagnetic. They measured the normal-state susceptibility before and after irradiation, and found no evidence of a Curie-Weiss tail. This result is in contrast with the findings of Hofmann *et al.*,²⁰ who reported that the electron irradiation of YBCO pellets resulted in the creation of magnetic centers. One should, however, keep in mind that the irradiation damage at the grain boundaries could be responsible for that observation.

By examining Table I we find that irradiation induces an increase in $\rho(T)$ at a linear rate of approximately $5.51 \pm 0.55 \mu\Omega \text{ cm}$ per $1 \times 10^{19} \text{ e/cm}^2$. Therefore $dT_c/d\rho$

is $\sim -0.30 \pm 0.04 \text{ K}/\mu\Omega \text{ cm}$.

Recently, Radtke *et al.*⁴ suggested that systematic experiments, such as the one reported here, in which the change of T_c is related to the change in ρ , could determine the symmetry of the order parameter in the cuprates. Their theory addresses only the case of s -wave scattering by impurities. It predicts that the initial slope $dT_c/d\rho$ for a d -wave superconductor should be $-0.74 - 1.2 \text{ K}/\mu\Omega \text{ cm}$ (for the best estimates of the plasma frequency currently available for YBCO: $1.1 \leq \omega_{pl} \leq 1.4 \text{ eV}$), whereas an s -wave isotropic superconductor should show no suppression of T_c . Our experimental value of $-0.3 \text{ K}/\mu\Omega \text{ cm}$ therefore indicates that the order parameter of the cuprates is certainly not an isotropic s wave. Next we examine whether we can distinguish between a d -wave and an *anisotropic* s -wave or-

TABLE I. Sample T_c , resistivity at 145 K, and linear fit parameters, $\rho(T) = \rho_0 + bT$ for $130 \text{ K} < T < 160 \text{ K}$.

	T_c (K)	$\rho(T=145 \text{ K})$ ($\mu\Omega \text{ cm}$)	ρ_0 ($\mu\Omega \text{ cm}$)	b (K/ $\mu\Omega \text{ cm}$)
Unirradiated	90.8	73.83	-16.44	0.62
$\Phi = 0.98 \times 10^{19} \text{ e/cm}^2$	89.1	79.52	-10.77	0.62
$\Phi = 1.96 \times 10^{19} \text{ e/cm}^2$	87.5	84.62	-3.59	0.61
Annealed to 300 K	88.6	80.49	-11.68	0.64

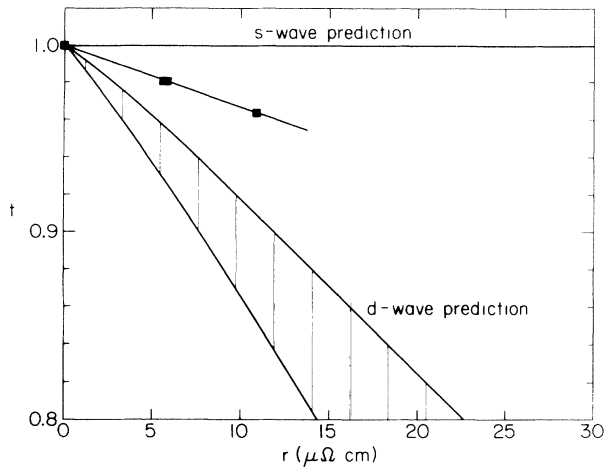


FIG. 4. The ratio t of the sample's transition temperature T_c to that of the unirradiated sample vs r , the difference between the sample's resistivity at $T = 145$ K and that of the unirradiated sample. The solid rectangular points show the experimental data. The uncertainties (plus to minus one standard deviation) of the experimental data are equal to the widths of the points and one half of the heights of the points. The s - and d -wave predictions are from Ref. 4. The indicated ranges of theoretical values for the d -wave case stem from an uncertainty of the plasma frequency. (See text.)

der parameter, based on our experimental results.

According to the theory of Millis, Sachdev, and Varma,²¹ anisotropic impurity scattering is characterized by a dimensionless parameter Q . This parameter may take any value between -1 and 1 , and it enters into the equations determining the suppression of T_c , but not the equations that determine the residual resistivity. Radtke *et al.*⁴ considered the case $Q = 0$, which assumes an isotropic impurity scattering. The abscissa of their predicted T_c vs ρ_0 plot is modified by the anisotropic scattering, since ρ_0 is replaced by $\rho_0/(1-Q)$. Thus, the initial change in T_c with residual resistivity for a d -wave superconductor could take any value between 0 and -2.4 K/ $\mu\Omega$ cm. (Note: as $Q \rightarrow 1$, the response of a d -wave superconductor to impurities is similar to that of an isotropic s -wave superconductor.) As Fig. 4 shows, our data fall about half as fast as their prediction, so it would be roughly consistent with a d -wave superconductor with anisotropically scattering impurities and $Q = 0.5$. Hence,

if the sample is a d -wave superconductor, our results indicate that the impurity scattering has a strong d -wave component.

Alternatively, an *anisotropic s-wave* superconductor can have a very similar T_c suppression. As one increases the s -wave anisotropy of the order parameter, the T_c vs ρ_0 curve changes from the isotropic s -wave case to something that looks like the d -wave case.²² Thus, we conclude that our data clearly show that the cuprates are strongly anisotropic superconductors, but we cannot determine whether the symmetry of the pairing state is s or d wave. On the other hand, our data should provide a critical test for any theory identifying the symmetry of the pairing state and the partial-waves phase shifts for the impurity scattering.

CONCLUSIONS

We have carried out low-temperature *in situ* resistivity measurements on a high-quality detwinned single crystal of YBCO in a high-voltage electron microscope. We have examined how T_c and $\rho(T)$ change as a function of electron irradiation dose at an electron energy low enough to produce only oxygen displacements and at temperatures low enough to avoid defect recovery. We find that oxygen displacements from the CuO_2 planes rather than the chains are the dominant contribution to the irradiation-induced suppression of T_c .

In addition, we find that $dT_c/d\rho = -0.30 \pm 0.04$ K/ $\mu\Omega$ cm which indicates that the cuprates are strongly anisotropic superconductors, but not whether the pairing symmetry is s or d wave. Isotropic s -wave pairing can be, however, ruled out. Our data provide a test for further development of the present theories addressing the symmetry of the pairing state.

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