Surface resistance measurement of superconducting YBa₂Cu₃O₇ in a magnetic field

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We report on the magnetic-field dependent surface resistance of polycrystalline $YBa_2Cu_3O_7$ ($T_c \approx 92$ K), measured using a brass cylindrical cavity resonator, operating at 16.5 GHz in the TE_{011} mode. A dc magnetic field H_{app} is applied parallel to the superconducting sample surface, and the temperature dependence of the surface resistance is measured for four different values of H_{app} (0 T, 0.22 T, 1 T, 5 T). An effective medium theory and the two-fluid model are used to fit the surface resistance versus temperature measurements both in zero field and for various applied fields. These results are applied to characterize the microwave properties of a polycrystalline ceramic superconductor.

I. INTRODUCTION

The microwave properties of superconductors are important for scientific reasons to enable study of the electromagnetic response of a superconductor for frequencies near but below the superconducting gap and temperatures below T_c . The nonvanishing values of the surface resistance under these conditions further allow exploration of the effect of an external magnetic field on the electromagnetic response. From a practical standpoint, the surface resistance of a superconductor suggests use of superconductors in microwave resonant cavities to achieve very high Q values. The high T_c superconductors open up such possibilities in the liquid nitrogen temperature range.

The temperature dependence of surface resistance is an important quantity for describing the microwave properties of high T_c materials. The surface resistance, $R_s(T)$, can be described as a sum of $R_{th}(T) + R_{res}$, where $R_{th}(T)$ is the intrinsic temperature dependent surface resistance and R_{res} is the residual surface resistance and is measured to be relatively independent of temperature. In this paper the temperature dependence of $R_{th}(T)$ is examined as a means to characterize polycrystalline ceramic samples, providing information on the temperature and magnetic field dependence of the characteristic parameters: the penetration depth λ , the fraction of the sample that is superconducting, and the residual surface resistivity. We assume that a fraction of the polycrystalline ceramic sample consists of nonsuperconducting components, and we use an effective medium approximation¹ and the two-fluid model to calculate $R_s(T)$ over the whole temperature range of interest. A fit of the experimental data to this model yields specific values for the three characterization parameters and their magnetic field dependence.

Experimentally, the magnetic field dependence of the dc resistivity R(H) versus T curves is broad,² and the phase transition at the zero resistance point is not well defined. Recent studies of the high T_c materials³⁻⁵ suggest that flux creep⁶⁻¹⁰ must be explicitly included in the analysis of the experimental data in order to determine quantities such as H_{c2} .¹¹ Flux creep considerations suggest that the phase transition is unambiguously measured at high frequencies ($v > v_c$), so that flux pinning effect should be unimportant above v_c (the critical damping frequency associated with flux pinning in the samples). It has been suggested that $v_c \sim 1$ GHz^{12,13} in high T_c materials. The 16.5 GHz cavity measurements reported here suggest that $H_{c2}(T)$ near T_c can be extracted from the surface resistance data.

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A large number of workers have reported results on the microwave characterization of high T_c materials. These experiments include resonant cavities^{14–21} and stripline resonators,²² where values of R_s are now approaching that of Nb at 16 GHz. In addition to the temperature dependence, a number of workers have reported the magnetic field dependence.^{14,17,18,23}

II. EXPERIMENTAL DETAILS

The microwave cavity experiments reported here make use of a polycrystalline ceramic sample of YBa₂· Cu₃O₇ that was prepared from sintered powder. Y₂O₃, BaCuO₃, and CuO powders were ground together and reacted at 920 °C. The resultant material was both ground and heat treated at 930 °C two more times to improve homogeneity. Next, the material was ground, cold pressed (4 GPa), and sintered at 940 °C in oxygen. The sintered material was ground into a disk two inches in diameter and 5 mm thick, and post-annealed in O₂ at 450 °C for 8 h.

Powder x-ray diffraction patterns for the sample used in this experiment show it to be "123" stoichiometry with a small admixture (~1%) of BaCO₃. The dc resistivity measurement show the onset of the superconducting transition at 93 K and zero resistivity at 90 K, which compares with the $T_c \approx 92.8$ K determined from microwave measurements reported here. SQUID susceptibility measurements show that from the Meissner effect the sample consists of ~80% superconducting material. Though higher quality pellets have been fabricated and characterized by microwave measurements,^{23,24} the focus of this work is on the orientational averaging which is required for all but the oriented films and single crystal samples.¹⁷

To determine the surface resistance of the superconductor, two disks were placed as end walls on a brass cylindrical cavity that is resonant at 16.5 GHz in the TE₀₁₁ mode. The resonant frequency of the TM_{111} mode (degenerate with the TE_{011} mode) was shifted by chamfering the edge of the cavity walls, a standard technique. A Janis superconducting cryostat was used to cool the cavity and to apply a dc magnetic field parallel to the surface of the superconducting end-plates. An F.W. Bell cryogenic Hall probe and a model 615 gaussmeter were used to monitor the magnetic field at the sample. The sample temperature was controlled and monitored by a Lake Shore Cryotronics DRC 82C temperature controller. Two types of temperature sensors were used. Below 80 K, one carbon-glass resistor was employed. Above 80 K, two platinum sensors were used because of their higher sensitivity in this temperature range. The two platinum sensors were placed so as to indicate the maximum possible temperature difference across one of the samples, and their

magneto-resistance was accounted for in a calibration procedure. Using this method, the temperature stability and maximum temperature difference across the sample were found to be within ± 0.1 K.

Microwave measurements were made with an HP 8341B synthesized sweeper, an HP 8510 network analyzer, and an HP 8516A S-parameter test-set. In order to isolate the analyzer from any stray magnetic fields, a 3 meter coaxial cable was used to connect the analyzer to the cryostat and cavity. The network analyzer was calibrated using standards (short, open, and load terminations). The surface resistance was measured as a function of temperature for four different values of dc magnetic field, i.e., 0, 0.22, 1, and 5 tesla. In each case, the cavity and samples were zero-fieldcooled (ZFC) from above T_c to 5 K, where the dc magnetic field was turned on, and data were taken at about twenty-five or so different temperatures. After the temperature had become stable (typically an hour), the S_{11} scattering parameter, or reflection coefficient, of the cavity was measured on and near resonance. The unloaded Q of the cavity was found from the reflection coefficient by using equations described in Ref. 25. To account for the surface resistance of the brass cavity wall, brass end-plates were substituted for the superconducting plates and the Q of the cavity was measured as a function of temperature. This Q measurement was found to be independent of the dc magnetic field, as expected for brass. The surface resistance of the superconducting end-plates was determined using conventional analysis.16,26,27

III. EXPERIMENTAL PROCEDURE

To minimize the possibility of trapping magnetic flux in the superconducting end-plates, we chose to take data under constant magnetic field, rather than at constant temperature, and to zero-field cool the sample before applying any dc magnetic field. We are aware that there is a time constant involved with the decay of any trapped flux to its equilibrium value.

In this paper, we curve-fit the surface resistance versus temperature data using an effective medium theory and the two-fluid model. To verify that our experimental procedure would result in a determination of the characteristic parameters mentioned above, and that the surface resistance data were obtained at equilibrium, we performed hysteresis measurements. A dc magnetic field of 0.22 tesla was chosen for these studies so as to easily observe hysteresis at the higher temperatures (>10 K).¹⁰

The hysteresis studies were performed as follows: the cavity and end-plates were zero-field cooled to 5 K, a field of 0.22 tesla was applied, and the surface resistance was measured as the temperature of the cavity and end-plates was increased above T_c . The magnetic field was left on, and the surface resistance measured as the temperature was decreased from above T_c to 5 K. For both runs, data were taken at 16 different temperatures. An hour was typically required for the temperature to stabilize at a given value. This hysteresis measurement was designed to compare surface resistance data for the zero-field cooled case, where flux trapping should be reduced, to that obtained for the field-cooled case, where a deliberate attempt was made to trap flux. The results of the hysteresis measurements are shown in Fig. 1. Hysteresis is noticeable for temperatures below 30 K. The difference in R_s at 30 K between the zero-field and field-cooled experiments is about 10%. Since our curve-fitting procedure relied most heavily on temperatures near T_c , we conclude that under the conditions of the magnetic field dependent surface resistance studies, we have presumably avoided any serious problems with flux trapping.

IV. DISCUSSION OF RESULTS

The experimental results of the temperature dependence of the surface resistance for the polycrystalline ceramic YBa₂Cu₃O₇ sample are presented in Fig. 2 at $H_{app} = 0$, 1 tesla, and 5 tesla. We next describe how the model calculation was carried out to fit these data.

The upper critical field of YBa₂Cu₃O₇ is highly anisotropic. Any direct measurement of H_{c2} on a polycrystalline sample would represent an average value of H_{c2} over different directions. For the anisotropic superconducting YBa₂Cu₃O₇ end-plate placed in a dc magnetic field H_{app} , the transition temperature, $T_c(\theta, H_{app})$, will depend on the angle, θ , of the *ab* plane of each grain relative to the dc magnetic field. The angular



FIG. 1. Plot of $R_s(H_{app}, T)$ versus T at constant magnetic field $H_{app} = 0.22$ T of data for both zero field cooled (ZFC) and field cooled (FC) traces. The small hysteresis of these measurements can be noted in the region below ~ 30 K.



FIG. 2. Plot of $R_s(H_{app}, T)$ versus T at zero field and at constant magnetic fields of $H_{app} = 1$ tesla and 5 tesla. The points correspond to the experimental measurements and the curve to the effective medium theory.

and temperature dependence of $H_{c2}(\theta)$ near T_c is given by Ginzburg-Landau theory

$$H_{c2}(\theta) = \frac{H_0}{\sqrt{\sin^2\theta + \left(\frac{m_{\parallel c}}{m_{\parallel ab}}\right)^2 \cos^2\theta}} (1-t), \qquad (1)$$

where the reduced temperature is written as

$$t = \frac{T}{T_c(\theta, 0)} \tag{2}$$

and $T_c(\theta, 0)$ is the critical temperature in zero magnetic field at angle θ . From Eq. (1), the transition temperature, $T_c(\theta, H_{app})$, under a dc magnetic field, H_{app} , is thus given by

$$\frac{T_c(\theta, H_{app})}{T_c(\theta, 0)} = \left[1 - \frac{H_{app}}{H_0} \sqrt{\sin^2 \theta + \left(\frac{m_{\parallel c}}{m_{\parallel ab}}\right)^2 \cos^2 \theta} \right]$$
(3)

It is more convenient to relate the effective mass parameter $m_{\parallel c}/m_{\parallel ab}$ and H_0 with $dH_{c2}^{\parallel c}/dT$, and $dH_{c2}^{\parallel ab}/dT$:

$$H_0 = \left(\frac{dH_{c2}^{|c|}}{dT}\right) T_c(\theta, 0) \tag{4}$$

$$\left(\frac{m_{\parallel c}}{m_{\parallel ab}}\right)^2 = \frac{dH_{c2}^{\parallel ab}}{dT} \left/ \frac{dH_{c2}^{\parallel c}}{dT} \right.$$
(5)

1601

By the two-fluid model, the conductivity of the superconducting component is $\sigma_s(\theta, H_{app})$ given by

$$\sigma_s(\theta, H_{app}) = \sigma_1 - j\sigma_2$$

= $\sigma_n t_H^4 - j(1 - t_H^4) \left(\frac{1}{\omega \mu_0 \langle \lambda(0) \rangle^2}\right)$ (6)

where σ_n is the normal state conductivity of the grain at temperatures above the superconducting transition at $T_c(\theta, H_{app})$. The reduced temperature in an applied field $t_H = T/T_c(\theta, H_{app})$ is dependent on θ and H_{app} , where $\langle \lambda(0) \rangle$ is the average London penetration depth (averaged over the crystalline orientations) at zero temperature. Hence, the conductivity $\sigma_s(\theta, H_{app})$ of a grain will depend both on the magnitude of the applied field H_{app} and on the orientation of the crystal axes of the grain relative to \mathbf{H}_{app} .

The temperature dependence of surface resistance R_s can be written as²⁷

$$R_s(T) = R_{th}(T) + R_{res} \tag{7}$$

where $R_{\rm res}$ is the residual surface resistance, which we assume to be temperature independent, and R_{th} can be described by the two-fluid model. The residual surface resistance, $R_{\rm res}$, presumably comes from the resistive barriers between grains^{28,29} and also from nonsuperconducting regions in the sample. We assume that the two effects result in an effective residual conductivity, $\sigma_{\rm res}$, which is temperature independent, and an effective fraction δ_n of nonsuperconducting material. The resistive barrier, nonsuperconducting regions, and the angular dependence of the superconducting conductivity of the sample result in a rather complex averaging problem.

A similar percolation problem, concerning the dc electrical conductivity of a binary alloy, has been studied by Bruggeman,³⁰ and by Garner and Stroud,³¹ who used an effective medium approximation theory to handle the absorption of infrared radiation by granular NbN. We have here extended the formulation of Garner and Stroud. In our case, the effective conductivity $\langle \sigma \rangle$ is determined from the relation

$$\delta_{n} \left(\frac{\sigma_{\text{res}} - \langle \sigma \rangle}{\sigma_{\text{res}} + 2\langle \sigma \rangle} \right) + \frac{1}{2} (1 - \delta_{n}) \int_{0}^{\pi} \left(\frac{\sigma_{s}(\theta, H_{\text{app}}) - \langle \sigma \rangle}{\sigma_{s}(\theta, H_{\text{app}}) + 2\langle \sigma \rangle} \right) \sin(\theta) \, d\theta = 0$$
(8)

together with the conductivity of the superconducting component $\sigma_s(\theta, H_{app})$ given by Eq. (6)

Once the effective conductivity is known, we can find the surface impedance of a conductor in the local limit using the relation:

$$Z_s = \left(\frac{j\omega\mu_0}{\langle\sigma\rangle}\right)^{1/2}.$$
(9)

The effective medium model¹ [Eq. (8)] above which was applied by Rao *et al.*^{32,33} to the ir assumes that the London penetration depth and the surface resistance of superconducting grains in the normal state are isotropic. This model represents a significant simplification of the actual YBaCuO material which is known to be anisotropic. However, the surface resistance of the polycrystalline ceramic depends more strongly on the anisotropy of $T_c(\theta, H_{app})$ than on the anisotropy of the London penetration depth and of the normal surface resistance. Hence, we have used an effective value of the London penetration depth and of the normal surface resistance, and have assumed these parameters to be isotropic.

Values for σ_n in Eq. (6) can be found from the difference between the observed surface resistivity in the normal state and the residual surface resistivity. The fraction of normal phase, δ_n , and the conductivity, σ_{res} , of the nonsuperconducting components can be found by solving Eq. (8) self-consistently and requiring the calculated residual surface resistance at zero temperature to be equal to the observed residual surface resistance. The fit is sensitive to δ_n , $\langle \lambda(0) \rangle$, and σ_{res} , but is much less sensitive to $dH_{c2}^{\parallel c}/dT$ and even less sensitive to $dH_{c2}^{\parallel ab}/dT$. The parameters for the best fit are summarized in Table I. It should be noted that since fit parameters such as $\langle \lambda(0) \rangle$ represent angular averages of an anisotropic quantity, the values listed in Table I may not be consistent with single crystal values given in the literature. The parameters δ_n and σ_{res} are sample specific parameters that may be useful in comparing various samples that may be used for microwave applications, and in characterizing the magnetic field dependence of the microwave properties. In the microwave experiment δ_n is found to be approximately 61%, which compares to the SQUID measurements which give $\sim 20\%$. This apparent inconsistency arises because

TABLE I. Parameters used for the effective medium approximation calculation.

H_{app} (T)	(λ(0)) (Å)	δ_n	$\frac{\sigma_{\rm res}}{\omega\mu_0}$ (Ω sec)	$\frac{-dH_{c2}^{\ ab}}{dT} (T/K)$	$\frac{-dH_{c2}^{\parallel c}}{dT} (\mathrm{T/K})$
0 T	3570 ± 860	0.61 ± 0.02	5.8 ± 0.1		
0.22 T	4140 ± 710	0.65 ± 0.01	5.0 ± 0.1	28 ± 22	4.2 ± 2.0
1 T	4630 ± 790	0.64 ± 0.02	5.0 ± 0.1	28 ± 22	4.2 ± 2.0
5 T	6260 ± 1400	$0.65~\pm~0.02$	4.5 ± 0.2	28 ± 22	4.2 ± 2.0

the dc SQUID measurements include currents associated with Josephson junction weak links between grains for the polycrystalline sample.^{28,29}

A good fit is obtained between the model calculation and the experimental points (see Figs. 1 and 2) as a function of T at zero magnetic field and for the various field values shown. The variation of the London penetration depth at zero temperature $\langle \lambda(0) \rangle$ with applied magnetic field H_{app} can easily be explained. $\langle \lambda(0) \rangle$ is inversely proportional to the density of superconducting electrons, which decreases with an increase in applied magnetic field. Recently Sridhar et al.¹⁷ showed that the penetration depth λ increases from 1400 Å by ~1000 Å for $H_{app} \sim 0.015$ tesla for a single crystal sample. However, our data show that λ changes by less than a factor of 2 for fields up to 5 tesla. This apparent discrepancy may be due to the saturation of the increase in penetration depth at high fields, as discussed by Sridhar et al.¹⁷ The effect of the change in λ for single crystal samples at high field should be of great interest to explore. The surface resistance R_s (4 K) at $H_{app} = 0, 0.22, 1, and 5$ tesla are 32 m Ω , 61 m Ω , 68 m Ω , and 70 m Ω , respectively. Our value of R_s (4 K) at zero magnetic field is slightly above that for Cu, which is approximately equal to 10 m Ω . The slight increase in R_s (4 K) and in δ_n and the small decrease in $\sigma_{\rm res}$ with increasing magnetic field $H_{\rm app}$ is perhaps due to vortex penetration into the superconducting grains.

The small hysteresis effects observed below 30 K show that flux trapping is not a serious problem in the surface resistance measurements reported here (see Fig. 1). We have also measured R_s versus H at constant temperature and find a very large change in R_s at fields <200 Oe, as has also been reported by a number of workers.^{14,17} The residual fields in the superconducting magnet did not permit a careful study of this low field anomaly. This initial rapid increase is followed by a much smaller increase up to H_{c2} (provided $T \approx T_c$ so that H_{c2} is accessible). Hence, the measurements reported in Figs. 1 and 2 can be considered to correspond to thermal equilibrium conditions. By decreasing the microwave power from 25 μ W to 0.2 μ W for various temperatures and magnetic fields, no change in the O of the cavity was observed, showing that the Lorentz force induced by the dc magnetic field acting on the microwave current does not introduce flux motion. We thus conclude that our operating frequency (16.5 GHz) was above the critical frequency for flux creep, in agreement with single crystal estimates of Worthington et al.¹² based on single crystal materials. Thus accurate measurements of $H_{c2}(T)$ near T_c by microwave methods in a regime where flux pinning effects are not important should be of great interest. The surface resistance curves in Fig. 2 can be fitted with slopes $dH_{c2}^{\|ab}/dT =$ 28 ± 22 T/K, and $dH_{c2}^{\parallel c}/dT = 4.2 \pm 2.0$ T/K. However, the present experimental accuracy is not sufficient to yield quantitative information about the functional form of $H_{c2}(T)$.

These results show that the temperature dependence of the surface resistance of the polycrystalline sample in zero field can be explained using a two-fluid model in conjunction with an effective medium approximation. From this result we can conclude that granularity is an important factor in the analysis of the microwave properties of polycrystalline ceramic samples. This result also implies that detailed information on the superconducting mechanism for intrinsic high T_c materials cannot be extracted from the temperature dependence of the surface resistance of polycrystalline ceramic samples. A sample of higher quality (e.g., single crystal or oriented thin film) must be used in order to learn something about the superconducting mechanism of the high T_c superconductors.

In summary, microwave measurements of the temperature dependence of the surface resistance of the ceramic sample can be described within the framework of an effective medium model and using a two-fluid model for the normal and superconducting electrons. From this model, we find that the microwave properties of polycrystalline ceramic samples can be characterized by an average penetration depth at T = 0, the fraction of sample that remains in the normal state, and the effective residual microwave conductivity σ_{res} . We further show that this model can describe the effect of a magnetic field on the microwave properties of high T_c polycrystalline samples and the model can be used to characterize such samples in a magnetic field.

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