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RF MEASUREMENTS ON HIGH T_c MATERIALS

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1. Introduction

Measurements of the microwave surface resistivity of high T_c materials were made in a superconducting niobium $6Ghz$ TE_{011} resonant cavity and a niobium $8.6Ghz$ TM_{010} elliptical cavity. The TE_{011} apparatus permits variation of the temperature of the sample independently of that of the niobium cavity. The Q of the host cavity thus remains at a high value while the resistivity of the sample is measured from $4.2K$ to above the transition ($\approx 100K$). The resistivity of a $1.27cm$ diameter ceramic pellet is observed to fall from 1Ω above transition to 0.02Ω at $4.2K$. A set of nine single crystals of total area $6.5mm^2$ is measured to have a broad transition near $75K$. The surface resistivity of a single $3mm^2$ crystal is measured in the ellipitical cavity to be $\leq 9.8 \times 10^{-5}\Omega$ at $1.5K$.

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2. TE_{011} Cavity

A TE_{011} niobium cavity was employed to measure the microwave resistivity of samples of high T_c superconducting material. The samples are introduced into the high Q cavity via a sapphire rod through a niobium cutoff tube aligned along the cavity axis. The rod is coupled by a thermal resistor to the helium bath. A heater at the base of the sapphire rod and thermometers in contact with the rod control and monitor the temperature of the sample. The heater and both thermometers are placed well beyond RF cutoff.

The surface currents excited by the TE_{011} mode are exclusively circumferential and vanish at the joints. Dissipated power is thus restricted to the niobium cavity and sample surfaces rather than the details of the cavity joints. The port for the sample introduction is a rather large perturbation and is therefore located on the cavity axis to preserve the cylindrical symmetry and avoid large spurious joint currents. The fields at the surface of the sample are presumably well known except for enhancements due to the annular space between the sample and the cavity or edge effects from the sample. A disadvantage of the configuration is that the fields vanish at the center of the cavity. The impact of the resistivity of the pellet on the cavity Q is thereby depressed. As a consequence, unless the resistivity of the sample is at least an order of magnitude higher than the resistivity of the $4.2K$ niobium ($10^{-5}\Omega$), the effect on the unloaded Q is not measureable.

Good sensitivity to very low resistivity is provided by calorimetry. The temperature rise of the sample above that of the He bath is measured for a particular transmitted power. The same temperature rise is reproduced by a measured heater power. We thus correlate dissipated power with transmitted power. Knowledge of the effective magnetic field on the sample surface yields a resistivity. Resolution is limited by background losses (heating of the sapphire and grease, etc.) to $\pm 10^{-6}\Omega$. The apparatus thus permits a measure of the temperature dependence of the resistivity.

2.1. Cavity Design

The TE_{011} niobium cavity is shown in Fig. 1. The cavity consists of a $5.02cm$ long by $6.98cm$ diameter cylinder with removable endplates. A groove is placed in one of the endplates to split the degeneracy with the TM_{111} mode. The geometry factor for the cavity in the TE_{011} mode is 757.5Ω. Two coupling loops are located in an endplate at a radius consistent with the peak magnetic field in the TE_{011} mode. One is fixed while the other remains adjustable during the experiment. The fixed coupler is set to yield an external Q of 2×10^{10} and during the tests is used to measure transmitted power and decay time.

A $1.27cm$ diameter pellet of high T_c material is introduced into the opposite endplate through the cutoff tube. The sample rests on the end of a $0.635cm$ diameter sapphire rod. Sapphire is distinguished by its high thermal conductivity, low RF losses, and low thermal expansion coefficient. Good thermal contact between sapphire and sample is guaranteed by a small dab of Apiezon N grease. A thermometer is located half way up the rod to monitor the temperature of the rod and the sample. At the base of the rod is a heater to raise the temperature and an additional thermometer. Due to the excellent conductivity of sapphire we find the two thermometers agree to within $0.1 \rightarrow 0.2K$ over the temperature range between $4.2K$ and $100K$. A thermal resistor connects the rod to the helium bath. A thermometer is also located in the bath. The combination of heater, bath, and distributed thermometers provide for a flexible control of the temperature of the sample without disturbing the cavity. The distance that the rod extends into the volume of the cavity is externally adjustable. The cavity is evacuated through the cutoff tube.

Fig. 1. TE_{011} Cavity.

2.2. Baseline Measurements

In order to test the precision measurement capability of the apparatus, its characteristics were studied empty of a sample and also with a niobium sample. The cavity assembly including the sapphire rod but no sample was cooled to $4.2K$. The unloaded Q_0 was measured to be 7.9×10^7 in good agreement with the BCS value at $4.2K$ of 7.894×10^7. The impact of losses in the joints and sample holder clearly have a negligible impact on the cavity Q_0. The Q_0 was found to be independent of the temperature of the sapphire rod as expected. There was some heating of the sapphire in the presence of RF indicating dissipation in the dielectric.

Niobium Pellet

A niobium pellet with dimensions that matched our high T_c sample was mounted for insertion into the cavity. Just as with the high T_c pellet a small dab of grease effectively glued the niobium to the end of the sapphire rod and established thermal contact. The penetration of the rod was set so that the surface of the pellet was flush with the surface of the cavity. We were wary of pushing the pellet further into the cavity for fear of exposing the grease to the RF.

The ratio of incident to transmitted power as a function of the temperature of the pellet yields the temperature dependence of the cavity Q_0. Calorimetry establishes the relationship between Q_0 and a quantity proportional to the resistivity of the pellet. The absolute value of the resistivity requires a calibration of the field strength and is effected by heating the niobium pellet to room temperature at which its resistivity is known.

In particular the power dissipated in the cavity walls and pellet is

$$P_{c+p} = \frac{\omega U}{Q_0} = \frac{1}{2} R_c \int_c H^2 da + \frac{1}{2} R_p \int_p H^2 da. \tag{2.1}$$

We define the effective areas $< A_c >$ and $< A_p >$ so that

$$\frac{1}{Q_0} = \frac{1}{2} R_c < A_c > + \frac{1}{2} R_p < A_p > . \tag{2.2}$$

The power dissipated in the pellet that corresponds to a temperature rise of ΔT (typically $\Delta T \approx 0.5K$) above the temperature of the bath is

$$P_p = \frac{1}{2} R_p(T_h) < A_p > P_{trans} Q_{ext}, \tag{2.3}$$

where P_{trans} and Q_{ext} are the transmitted power and the Q associated with the ouput coupler respectively. P_p is also the power required of the heater in the absence of RF to raise the temperature to $T_h = T_b + \Delta T$ and T_b is the temperature of the bath. A measurement of Q_0 at T_h and solution of (2.3) and (2.2) yields the temperature independent quantity

$$\frac{1}{2} R_c < A_c >= \frac{1}{Q_0(T_h)} - \frac{P_p(T_h)}{P_{trans}(T_h) Q_{ext}}. \tag{2.4}$$

Finally a rearrangement of (2.2) and computation of the cavity losses according to (2.4) gives:

$$R_p(T) = \frac{1}{< A_p >} \left(\frac{1}{Q_0(T)} - \frac{1}{2} R_c < A_c > \right). \tag{2.5}$$

Equation (2.5) is an expression of proportionality of the resistivity of the pellet and a function of the Q_0 of the cavity. The constant of proportionality is $\frac{1}{<A_p>}$. The Q_0 is computed from the ratio of incident to transmitted powers which are measured as a function of temperature according to:

$$Q_0(T) = \frac{Q_{inp}}{2\sqrt{\frac{Q_{inp}}{Q_{ext}}}\sqrt{\frac{P_i(T)}{P_{trans}(T)}} - 1}. \tag{2.6}$$

Q_{inp} is the external Q corresponding to the input coupler and P_i the incident power. The relative measurement error in the Q_0 from one point to the next is a fraction of a per cent. Knowledge of the resistivity of niobium at $4.2K$ and at room temperature, ($9.6 \times 10^{-6}\Omega$ and 0.059Ω respectively) permits the solution of (2.5) for $< A_p >$ and the proportionality becomes an equality. Note that in the BCS theory resistivity scales as an exponential function of the energy gap and the

inverse temperature. Therefore the constant $< A_p >$ is irrelevant to a measure of the energy gap and no prior knowledge of resistance is required to determine the gap.

The measured resistivity of the niobium pellet versus temperature is shown in Fig. 2. The errors are a consequence of uncertainty in the field distribution at the pellet, systematic uncertainty in absolute determination of the transmitted power, and systematic uncertainty in the calibration of dissipated power with respect to pellet temperature. Point to point error is due to random fluctuations in the measurement of the transmitted power. The prediction of BCS theory is computed[1] and there is good agreement with the data as indicated in Fig. 2.

Fig. 2. Surface resistance of a niobium pellet. The points corresponding to the measured resistivity of the niobium are accompanied by error bars. The smooth curve indicates BCS theory.

3. Sintered Y-Ba-Cu-O Pellet

A sintered ceramic pellet of high T_c material was prepared by grinding and sintering the raw materials several times. An SEM micrograph of the $1.27cm$ diameter pellet is shown in Fig. 3. The crystal morphology was uniform throughout the pellet and there were no large holes. Using EDS it was determined that the yttrium, barium and copper content were in the stochiometric ratios of 1:2:3. The oxygen content was not determined. One region of the sample was found to contain finer grains than elsewhere and EDS analysis of the region indicated that it was barium rich and copper poor.

Fig. 3. SEM micrograph of sintered Y-Ba-Cu-O pellet.

The transition temperature T_c was determined by an inductive measurement. The magnetic coupling of a pair of coils with the high T_c sample placed between the coils was measured as a function of temperature. The transition to the superconducting state is evident in Inductive measurement of the transition temperature. Fig. 4 at about $87K$ and is consistent with the observed RF transition.

Fig. 4. A transition in the AC flux linkage of two coils with the high T_c material between them occurs at $87K$.

The pellet was placed in a location in the cavity identical to that of the niobium pellet for which the field distribution was measured. That the niobium and high T_c pellet are identically located is guaranteed by matching the cavity frequency. Again thermal contact is assured by Apiezon N grease between pellet and sapphire rod. Decay time and thus Q is measured as a function of the temperature of the pellet and the resistivity of the sample is extracted from the data according to (2.5). The results are shown in Fig. 5. The errors are predominately due to the uncertainty associated with measuring decay times. All measurements are at fixed transmitted power and therefore at fixed field level. (The Q_{ext} of the output coupler is also fixed.) The ratio of transmitted to incident power yields a more self consistent and reproducible measurement of the cavity Q than does the decay time. However it was desireable to measure the temperature dependence of the Q at a fixed field level and variations in transmitted power were thus not tolerable. For

Fig. 5. Resistivity of Y-Ba-Cu-O sintered pellet. Representative error bars are indicated. Error is associated primarily with the fluctuations in the measurement of the decay time.

comparison, niobium and Y-Ba-Cu-O data are plotted simultaneously in Fig. 6.

A strong magnetic field dependence of the resistivity of the sintered pellet at fixed temperature is observed. The data for $4.2K$ are indicated in Fig. 7. For the measurement of the field dependence helium gas was admitted into the cavity and the RF duty cycle reduced in order to prevent heating of the pellet. There was no observed dependence of the resistivity of the niobium pellet over the range of magnetic fields accessible with our apparatus. The temperature dependence of the resistivity (Fig. 5) is determined at a fixed magnetic field level of about $1.0G$.

Fig. 6. Data for the high T_c pellet appear in the upper left of the figure. In the lower right are the data for the niobium pellet. The apparatus is capable of yielding resistivity over a range of five decades.

3.1. Energy Gap

We assume that for $T \ll T_c$ the resistivity is described in terms of the energy gap Δ according to $R - R_{res} \propto e^{-\Delta/kT_c}$. R_{res} is a residual resistivity that we approach asymptotically as $T \to 0$. Then $\log(R - R_{res}) \propto -\Delta/kT_c$. A fit to the data are shown in Fig. 8. Error bars are indicated at the extremes of the points used in the fit. The fit yields a value of $2\Delta/kT_c = 2.4 \pm 1.0$ which is consistent with a gap measurement [2]P.E.Sulewski, T.W.Noh,J.T.McWhirter,and A.J.Sievers, Laboratory of Atomic and Solid State Physics and Materials Science Center, Cornell University, Ithaca, New York 14853.2501 based on the infrared reflectivity of similarly prepared samples.

Fig. 7. A strong field dependence of the Q_0 of the cavity is observed at $4.2K$.

4. Single Crystals

Ten single crystal flakes of Y-Ba-Cu-O were prepared in crucibles using the technique described by Schneemeyer, et. al.[3] These crystals had the correct yttrium,barium and copper compositions. They were not annealed in oxygen after extraction from the crucible and were probably oxygen deficient. The crystals were planar with the c-axis of the orthorhombic structure perpendicular to this plane. The crystals were typically about $10\mu m$ thick. Some aluminum particles were embedded in the crystal flakes. The source of the contamination was probably the alumina crucible in which the crystals were grown.

Nine crystals of total area $6.5mm^2$ were placed on the end of the sapphire rod in the TE_{011} cavity. A small amount of paraffin fixed the crystals to the rod and provided thermal contact. The cavity Q was measured as a function of crystal temperature. The data are shown in Fig. 9. There is a broad transition about $75K$. The field distribution at the surface of the crystals is critically dependent

Fig. 8. Surface resistance of the Y-Ba-Cu-O pellet with residual subtracted. Representative error bars are indicated.

on the highly irregular crystal boundaries and is essentially unknown. In addition there can be significant RF dissipation in the paraffin and the sapphire and in consequence we did not extract a surface resistivity from the data.

A complimentary experiment using an x-band ($8.6Ghz$) elliptical cavity yields an upper limit for the resistivity of a single $3mm^2$ crystal at $1.5K$. The experimental technique is similar to that used by Hein et. al.[4] The crystal was placed in a high magnetic field region of the cavity and the cavity cooled to $1.5K$. Cold helium gas was added to the interior of the cavity to assure that the crystal was kept cold in the presence of RF. We measured the difference in the perturbed and unperturbed Q.

Consider that $P_{diss} = P_{flake} + P_{niobium}$, where P_{diss} is the total dissipated power and P_{flake} and $P_{niobium}$ the power dissipated in the crystal flake and the niobium respectively. Normalization with respect to ωU where U is the stored

Fig. 9. The Q of the cavity rises rapidly as the single crystals become superconducting. The dashed line indicates the Q that is attained if the crystals and paraffin on the sapphire rod are replaced by a $1.27cm$ diameter niobium pellet at $4.2K$.

energy yields:

$$1/Q =< A_{flake} > R_f + (< A_{cav} > - < A_{flake} >)R_{cav}, \tag{4.1}$$

where $< A_{flake} >= \frac{\frac{1}{2}\int_{flake} H^2 da}{\omega U}$ and similarly for $< A_{cav} >$. R_f and R_{cav} are the resistivities of the flake and cavity walls. A measurement of the Q of the unperturbed cavity gives $1/Q_0 =< A_{cav} > R_{cav}$. Then (4.1) can be solved for the ratio

$$\frac{< A_{flake} >}{< A_{cav} >} = \frac{(Q_0/Q)}{(R_f/R_{cav} - 1)}. \tag{4.2}$$

The ratio defined in (4.2) is determined by measurement of the effect of a perturbation in a $1500Mhz$ elliptical copper cavity. The perturbative effect of the crystal flake in the $8.6Ghz$ cavity is reproduced by a titanium alloy foil cut in the same

shape as that of the crystal with dimensions scaled to match the larger copper cavity. The resistivity of the foil is 90 times that of copper. Now we measure Q_0 and Q where R_f is the resistivity of the foil and R_{cav} the resistivity of copper and substitution into (4.1) gives $\frac{<A_{flake}>}{<A_{cav}>} = 1.27 \times 10^{-2}$. Finally we invert (4.1) and solve for R_f.

$$R_f = (\frac{Q_0/Q - 1}{\frac{<A_{flake}>}{<A_{cav}>}} + 1)R_{cav}. \tag{4.3}$$

We measure in the x-band cavity $\frac{Q_0}{Q} = \frac{1.81\times10^9}{2.1\times10^8}$. Again Q corresponds to cavity including crystal flake and Q_0 the unperturbed cavity. Then $R_{crystal} = 6.13 \times 10^2 R_{cav} = 9.8 \times 10^{-5}\Omega$. We estimate the measurement error of about 15% to be due almost exclusively to uncertainties in measuring decay times and input coupling.

In the TE_{011} cavity the crystal was exposed to a range of magnetic fields and no Q dependence was observed thus demonstrating that there was sufficient cold helium gas in the cavity to prevent RF heating of the crystal. Limitations of the technique include uncertainty in the resistivity of niobium at $1.5K$ and losses through the boundary between crystal and the surface of the cavity.

5. Conclusions

A method was developed to measure the temperature dependence of the microwave surface resistance of $1.27cm$ diameter high T_c pellets from $100K$ to $4.2K$. A superconducting niobium host cavity is maintained at $4.2K$ throughout the measurement providing a baseline resistance of $10^{-5}\Omega$. Using a niobium pellet the method has proven to give reliable values of absolute surface resistance down to a few $\times10^{-5}\Omega$. It is estimated that resistance values down to a few $\times10^{-6}\Omega$ can be determined by calorimetry in the same apparatus.

A sintered high T_c pellet was measured and the residual resistance of 0.02Ω and an energy gap of $2\Delta/kT_c = 2.4\pm1$ determined. The surface resistance is observed to

depend strongly on magnetic field and to increase sharply to the normal conducting value of 1.0Ω in a field of $15G$.

Characterization of single crystal material has started. A broad transition near $75K$ is observed and a residual resistance of $10^{-4}\Omega$ measured.

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