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TITLE MICROWAVE CHARACTERIZATION OF HIGH-TEMPERATURE SUPERCONDUCTORS

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Microwave characterization of high-temperature superconductors

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ABSTRACT

Thick (10-15 μm) Tl-Ba-Ca-Cu-O films have been deposited onto yttria-stabilized zirconia and Ag substrates by d.c. magnetron sputtering techniques. Direct deposition onto 1" diameter yttria-stabilized zirconia yields films with typical 22-GHz surface resistance (R_s) values of $5.2 \pm 2 \text{ m}\Omega$ and $52 \pm 2 \text{ m}\Omega$ at 10 K and 77 K, respectively. For comparison, R_s of Cu at this same frequency is 10 $\text{m}\Omega$ at 4 K and 22 $\text{m}\Omega$ at 77 K. Tl-Ba-Ca-Cu-O films have also been deposited onto 1" diameter Ag substrates using Au/Cu, Cu, and BaF_2 buffer layers. The lowest R_s values were obtained on films with a BaF_2 buffer layer, typical values being $7.8 \pm 2 \text{ m}\Omega$ and $30.6 \pm 2 \text{ m}\Omega$ (measured at 22 GHz) at 10 K and 77 K, respectively. Larger films (1.5" diameter) with similar R_s values were prepared using this same technique, demonstrating that the fabrication process can be scaled to larger surface areas. These films are promising for radiofrequency cavity applications because they are thick (50-75 times the London penetration depth), have relatively large surface areas, are fabricated on metallic substrates, and have R_s values that are competitive with Cu at 77 K and are lower than Cu at 4 K. Because they are polycrystalline and unoriented, it is anticipated that their R_s values can be lowered by improving the processing technique. High-quality films of $\text{YBa}_2\text{Cu}_3\text{O}_7$ have been electron-beam deposited onto 1" LaGaO_3 and 1.5" LaAlO_3 substrates. The 1" sample is characterized by R_s values of $0.2 \pm 0.1 \text{ m}\Omega$ at 4 K and $18.6 \pm 2 \text{ m}\Omega$ at 77 K. The 4-K value is only 2-4 times higher than Nb. The 1.5" sample has R_s values (measured at 18 GHz) of $0.93 \pm 2 \text{ m}\Omega$ and $71 \pm 3 \text{ m}\Omega$ at 10 K and 77 K, respectively.

1. INTRODUCTION

In the approximately three years since the discovery of high-temperature superconductivity, experimentalists have made tremendous progress in their ability to fabricate these materials in the form of bulk ceramic pellets, thin and thick films, and twinned crystals. Much of the motivation for this work stems from the envisioned potential applications of these high-temperature superconductors (HTS). One of the most important parameters that will ultimately determine the suitability of these materials for various applications is high-frequency surface resistance (R_s). Recognizing this fact, many research groups worldwide are now engaged in measuring R_s of HTS material. To date, R_s measurements have been made on bulk ceramics,¹⁻⁴ thin and thick films,⁵⁻⁷ and single (twinned) crystals.^{8,9}

The first applications will surely involve HTS material deposited onto films of various thickness and geometry. For example, microwave transmission lines, filters, resonators, and other passive components are currently approaching the realm of practicality.¹⁰ Another exciting potential application of HTS is radiofrequency (RF) accelerating cavities.^{11,12} This particular application is different from others in that it requires a low value of R_s in the presence of relatively large surface magnetic fields. This is certainly a more stringent requirement than demanding only that R_s be lower than Cu at some given temperature.

Niobium is the present industry standard for accelerating cavities. Typical accelerating gradients are 5-10 MeV/m with Q-values of 10^9 - 10^{10} at 2-4 K. These gradients correspond to typical surface RF magnetic fields H_{rf} of 250-500 Oe for electron acceleration, and values that are 2-3 times higher for protons or ions.⁶ From theoretical considerations, and using the experimental value of the critical magnetic field, the maximum accelerating field for Nb is 50 MeV/m. Similar calculations suggest that the HTS critical magnetic field should be $\sim 11,000 \text{ Oe}$ (4 times that of Nb), corresponding to an accelerating

gradient of 200 MeV/m! Unfortunately the present performance of HTS material falls far short of these lofty predictions. For example, high-quality crystals exhibit a dramatic increase in R_s for H_{rf} exceeding ~ 90 Oe.⁸ On the other hand, bulk ceramic $\text{YBa}_2\text{Cu}_3\text{O}_7$ (YBCO) has been found to retain its superconducting properties ($R_s = 0.05 R_n$) up to H_{rf} values of 640 Oe, a value that is limited by the dielectric breakdown of the surrounding cryogen.¹³ Further measurements of R_s vs. H_{rf} , especially on high-quality films, will be required to elucidate the materials properties that cause HTS material to quench in rather modest surface magnetic fields.

Given the above assessment of HTS surface resistance, it is fair to state that considerable improvement in materials properties will be required before an HTS accelerating cavity can be realized. However, there is no present theoretical or experimental evidence suggesting that such a goal cannot be attained. In fact, considering that these materials have been under investigation for less than 3 years it is remarkable that R_s values have gone from $500 \mu\Omega$ at 4 K (measured at 3 GHz)¹ to $9 \mu\Omega$ at 77 K (measured at 86.5 GHz and scaled by ω^2 to 3 GHz)⁵, i.e., a reduction of more than a factor of 50!

2. R_s MEASUREMENTS OF THALLIUM-BASED FILMS

2.1. 1"-diameter thallium films

Surface resistance measurements have been made on several Tl-Ba-Ca-Cu-O thick films using a cylindrical 22-GHz Cu cavity (TE_{011} fundamental mode) whereby the superconducting film and substrate form the cavity end wall. In this configuration the microwave surface currents are minimal at the junction between the cavity and superconductor thereby minimizing joint losses. The electromagnetic field distribution for this fundamental mode is easily calculable from Maxwell's equations with appropriate boundary conditions, yielding the result that 26% of the cavity losses occur at the end wall, which is normally replaced by the superconducting film and substrate. Measurement of the Q value of the cavity with sample, and subsequent computation of R_s is as follows. The reciprocal Q of the sample $1/Q_s$ is obtained by subtracting the reciprocal Q of the empty cavity $1/Q_c$ from that of the cavity with sample $1/Q_{c+s}$.

$$\frac{1}{Q_s} = \frac{1}{Q_{c+s}} - \frac{1}{\eta Q_c} \quad (1)$$

where η is an enhancement factor (1.26) that artificially increases the bare cavity Q to a value consistent with no losses in the end wall. The surface resistance is computed from the measured Q values for the sample and a known material, stainless steel in the present case,

$$R_s = \frac{Q R_s'}{Q_s} \quad (2)$$

where the primes stand for the appropriate values of the standard material.

Shown in Fig. 1 are the temperature-dependent surface resistance curves obtained at 22 GHz for four 1" Tl-based films that have been magnetron sputtered onto either yttria-stabilized zirconia (YSZ) or Ag substrates. The curve labeled YSZ corresponds to a 10 μm -thick Tl film deposited directly onto YSZ. Following deposition it was annealed in O_2 at 900°C for 18 minutes. X-ray diffraction data show this film to be comprised primarily of the 2212 phase; scanning electron micrographs reveal a random structure comprised of platelets. R_s values are $5.2 \pm 2 \text{ m}\Omega$ and $52 \pm 2 \text{ m}\Omega$ at 12 K and 77 K, respectively. A summary of all R_s values discussed in this work, as well as values measured by other workers, and a comparison with Cu will be given later.

Fig. 1. Surface resistance data for 1"-Ti films deposited onto YSZ; Ag with either Cu, Au/Cu, or BaF₂ buffer layers. The microwave frequency is 22 GHz.

Ti films deposited onto 1" Ag substrates with buffer layers of either Cu, Au/Cu, or BaF₂ are labeled in Fig. 1 according to the buffer layer material. A typical film is composed of a 0.125" Ag substrate with a 1 μ m buffer layer and a 10 - 15 μ m Ti layer. The best film was fabricated with a BaF₂ buffer layer separating the Ag substrate from the 15 μ m-thick Ti film. R_s values at 12 K and 77 K are 6.5 ± 2 m Ω and 30.6 ± 2 m Ω , respectively. Recall that respective R_s values for Cu at these temperatures are 11 m Ω and 22 m Ω . X-ray data show that these films are unoriented and multi-phased. However, it is significant that the post-deposition annealing temperature required to obtain these films is now less than 800°C.

2.2. 1.5"-diameter Ti films

Based on the previous promising results obtained by using a BaF₂ buffer layer to separate the Ag substrate from the 1"-Ti film, a 1.5" film was fabricated. The temperature dependence of R_s for the resulting film is shown in Fig. 2. These data were obtained in a way similar to that described above for the 22 GHz cavity, the exception being that an 18-GHz cavity was employed to accommodate the larger diameter films. Although this film is multi-phased it contains primarily the 2223 phase rather than the 2212 phase of the 1" films. At 11 K R_s is 15.4 ± 2 m Ω and the value increases to 38.6 ± 2 m Ω at 77 K, similar to the values previously described for the 1" Ti film with a BaF₂ buffer layer. Again note that the R_s values are similar to Cu. These results are significant for accelerator cavity applications in three respects: 1) The composite films are deposited onto a metallic substrate that has good thermal conductivity and machinability--properties that are extremely important when considering the fabrication of a cavity. 2) The R_s values are approximately equal to Cu both at 4 K and 77 K. However, as stated in the introduction most accelerators operate at frequencies lower than 18 or 22 GHz, the typical value being 500 MHz. Therefore if we assume quadratic scaling¹⁴ of our present 18-GHz R_s values, they become approximately 12 $\mu\Omega$ and 30 $\mu\Omega$ at 10 K and 77 K, respectively--more than two orders of magnitude lower than Cu. 3) The fabrication process is amenable to scaling to larger surface areas. In fact, an elliptical-shaped substrate of dimensions 2" x 4" has recently been coated by magnetron sputtering. Although no R_s measurements have yet been made on this device, it has been demonstrated that the stoichiometry variations over this

large area are minimal. Other fabrication techniques that have been applied to Tl-based films have as yet not produced high-quality films on surface areas larger than 1 cm^2 .¹⁵

Fig. 2. Surface resistance data for a 1.5"-Tl film deposited onto a Ag substrate with a BaF₂ buffer layer. The microwave frequency is 18 GHz.

3. R_s MEASUREMENTS OF YBCO FILMS

3.1. 1"-diameter YBCO films

A complementary research effort at Los Alamos is devoted to the fabrication of high-quality YBCO films. The best results have come from $0.8 \mu\text{m}$ thick films electron-beam deposited onto single-crystal (001) LaGaO₃ substrates. The coevaporation of Y, BaF₂, and Cu was followed by anneals in oxygen and water vapor. X-ray powder diffraction analysis revealed the presence of impurity phases, principally BaCuO₂ and Y₂BaCuO₅, in all samples. Pole-figure analyses by x-ray diffraction showed that the film contained approximately equal amounts of *c/a*-axis oriented YBCO. Figure 3 shows the results of R_s measurements in a 22-GHz Cu cavity on a $0.8 \mu\text{m}$ film of YBCO deposited onto LaGaO₃. At 16 K the measured value of R_s is $0.4 \text{ m}\Omega$; however, the experimental error of this measurement is $\pm 2 \text{ m}\Omega$, as determined primarily by the sensitivity of the Cu cavity. A similar measurement was done in a Nb superconducting cavity yielding an R_s value at 4 K of $0.2 \pm 0.1 \text{ m}\Omega$. This value is only a factor of 2-4 higher than Nb at the same temperature and frequency. At 77 K the R_s value is $18.6 \pm 2 \text{ m}\Omega$, which is only slightly lower than Cu at this temperature ($22 \text{ m}\Omega$).

Surface resistance measurements on similar YBCO films deposited onto SrTiO₃ substrates show that the substrate plays an important role in determining the low-temperature R_s values.⁷ SrTiO₃ is not a good substrate for high-frequency applications because of the strong temperature dependence of its permittivity ϵ , which rises from near 1000 at 100 K to 25,000 at 4 K. In contrast LaGaO₃ and LaAlO₃ substrates exhibit weakly temperature dependent ϵ values of 15 - 25 with concomitant low microwave losses.

Fig. 3. R_s vs. T for YBCO deposited onto a 1"-single-crystal LaGaO_3 substrate. The sensitivity of the Cu cavity is $\sim 2 \text{ m}\Omega$. The Nb cavity sensitivity is $0.1 \text{ m}\Omega$. The microwave frequency is 22 GHz.

3.2. 1.5"-diameter YBCO films

Recent R_s vs. T results obtained from a 1.5"-diameter YBCO film *e*-beam deposited onto a single-crystal LaAlO_3 substrate are shown in Fig. 4. These measurements were done in an 18-GHz Cu cavity by the technique described above. At the lowest attainable temperature of our closed-cycle refrigerator, 10.9 K, the measured R_s is $0.93 \text{ m}\Omega$. Again, however, the measurement error is $\pm 2 \text{ m}\Omega$. Measurements in an 18-GHz Nb cavity are underway, but the results are not available at the time of this writing. Nevertheless, it appears that high-quality films of YBCO can be *e*-beam deposited onto relatively large surface area single-crystal LaAlO_3 , and presumably LaGaO_3 . These results are more pertinent for electronics applications than for cavity applications.

4. SUMMARY OF R_s RESULTS

A summary of the surface resistance values described in this work is shown in Fig. 5 as a function of frequency at $T = 4 \text{ K}$. A similar summary for values at $T = 77 \text{ K}$ are shown in Fig. 6. In addition to data obtained at Los Alamos, we show several results obtained by other groups. No R_s values have been scaled to lower frequencies in this plot; only measured values are shown. Furthermore, we have omitted the well-known results of R_s measurements on single (twinned) crystals. These specimens have low values of R_s that may, in fact, be presently limited by the measurement technique.^{8,9} We have chosen to concentrate on HTS film results because of their implications for near-term applications.

Some observations regarding the surface resistance values obtained to date are offered. At $T = 4 \text{ K}$ there already exists several films whose R_s values are considerably better than Cu, and are approaching that of Nb. For the more interesting temperature of 77 K (at least with respect to applications), there are several films that show great promise. The laser-ablation-produced ones show good high-frequency performance over a rather broad frequency range. Their R_s values are one to two orders of magnitude lower than Cu, which should make them amenable to immediate electronics applications. For cavity applications the picture is not as clear. The Ti-films deposited onto Ag substrates look very promising, but

the dependence of R_s on H_{rf} has not been measured. Based on results obtained from other samples, which will be discussed below, it is unclear whether large surface magnetic fields can be tolerated in the present samples.

Fig. 4. R_s vs. T for YBCO deposited onto a 1.5"-single-crystal LaAlO_3 substrate. The microwave frequency is 18 GHz.

An additional observation is that in many cases the temperature dependence of R_s shows a rather high and constant value over a large temperature range. There are several possible causes for this behavior; 1) the substrate is dominating the measurement; 2) the measurement technique is not sufficiently sensitive to the low values of R_s , and/or 3) R_s is truly limited by the temperature-independent residual resistance. This last point will be discussed below. However, it is noteworthy that highly-oriented samples exhibit sharp superconducting transitions as opposed to very broad transitions for unoriented samples although the low-temperature R_s values are essentially equal. It suggests that *intergranular* behavior dominates R_s near T_c , but that *intragranular* properties dominate R_s for $T \ll T_c$.

5. SURFACE MAGNETIC FIELD DEPENDENCE OF R_s

Displayed in Fig. 7 are the surface resistance curves as a function of surface magnetic field for high-quality crystals,⁸ a bulk ceramic pellet,¹⁶ and a laser-ablation-produced thin film.⁶ In each case R_s increases dramatically for rather low H_{rf} values. The best result is obtained for the crystals, where R_s remains approximately constant up to $H_{rf} = 90$ Oe. This field value corresponds to an accelerating field of ~ 2 MeV/m, which is much less than the 5 - 10 MeV/m routinely obtained in Nb. It is possible that these results are flawed because of the measurement technique. For each measurement the samples were mounted on a pedestal inside an evacuated cavity. This arrangement does not allow the microwave-induced heat to be readily dissipated by the samples. Thus, it is likely that increase in R_s is due to poor thermal conductivity rather than to intrinsic behavior.

Similar measurements on bulk rods placed inside a coaxial TEM resonator exhibit very different behavior.¹³ For this arrangement the rod formed the center conductor of the resonator which was bathed in cryogen, either LHe or LN_2 . Surface magnetic fields as high as 640 Oe were obtained at $T = 77$ K and 190 MHz with corresponding R_s values being 5% of the normal-state value. These results are very encouraging for accelerator applications. It is obvious, however, that proper measurements of R_s vs. H_{rf} on high-quality films need to be made.

Fig. 5. Summary of R_s vs. frequency data for selected films measured at 4 K. 1. YBCO on 1-cm² MgO (Stanford/Hewlett-Packard). 2. YBCO on 1"-diameter LaGaO₃ (LANL). 3. YBCO on 1-cm² LaAlO₃ (Cornell/Belcore). 4. Tl on 1"-diameter YSZ (LANL). 5. Tl on 1"-diameter Ag with a BaF₂ buffer layer (LANL). 6. Tl on 1.5"-diameter Ag with a BaF₂ buffer layer (LANL). 7. Tl on 1-cm² MgO (ST/UCLA).

6. DEPENDENCE OF R_s ON INTRAGRANULAR DEFECTS

We conclude by briefly discussing the effects of intragranular defects on R_s . The high-temperature superconductors, unlike conventional ones, are characterized by a large penetration depth [$\lambda(0) \approx 150$ nm] and short coherence length [$\xi_{ab} \sim 20$ - 30 Å; $\xi_c \sim 5$ - 10 Å]. Furthermore, currents flow much more readily in the ab planes than in the c direction. The superconducting structure is orthorhombic but with nearly tetragonal coordinates; typical ab unit cell dimensions are 3.9 Å. These parameters are quite different from those of Nb for example, where the lattice constant is smaller (3.3 Å), ξ is much larger (~ 380 Å), and $\lambda(0)$ is smaller (~ 390 Å).

Such a small HTS coherence length suggests that intragranular defects may be contributing to the high residual surface resistance of these materials. Recall that by definition the coherence length is the spatial range of phase coherence in the wave function for the superconducting electron pair. Disruption of this wave function destroys superconductivity on a microscopic level and thereby yields two normal conducting electrons. Obviously if one expects to attain low values of R_s it will be necessary to have all the electrons condensed into Cooper pairs, any unpaired electrons will cause R_s to become larger.

From microwave surface impedance measurements, Pippard¹⁷ demonstrated that in some superconductors (viz., those for which a nonlocal field-current relationship is applicable) the coherence length is reduced by the presence of impurities. He suggested an empirical relation for the coherence length:

$$\frac{1}{\xi} = \frac{1}{\xi_0} + \frac{1}{\alpha l} \quad (3)$$

where ξ_0 is the intrinsic coherence length, l is the electron mean free path, and α is a constant on the order of unity. Microscopic theory¹⁸ gives the value for ξ_0 as

$$\xi_0 = \frac{0.18 h v_F}{2 \pi k_B T_c} \quad (4)$$

where v_F is the electron velocity at the Fermi surface. The physical picture is one where the mean free path limits the range of the pair interaction. Defects or impurities will reduce the mean free path and thus the coherence length. Pippard concluded that although impurities introduced on a small scale (few percent) have no significant effect on T_c , the effect on his microwave measurement was a direct result of the coherence phenomena. Recognizing that HTS materials are described by a local theory rather than a nonlocal one, we suggest nevertheless that by similar arguments one might expect defects and impurities to contribute to high residual R_s values in HTS. This is especially true because of the extremely short coherence length. This may explain the experimental observation that although highly oriented films give much sharper microwave transitions, their low temperature values are similar to those of unoriented films.

Fig. 6. Summary of R_s vs. frequency data for selected films measured at 77 K. 1. YBCO on 1-cm² LaAlO₃ (Cornell/Belcore). 2. YBCO on 1-cm² SrTiO₃ (Wuppertal). 3. Tl on 1.5"-diameter Ag with a BaF₂ buffer layer (LANL). 4. YBCO on 1"-diameter LaGaO₃ (LANL). 5. Tl on 1"-diameter Ag with a BaF₂ buffer layer (LANL). 6. Tl on 1"-diameter YSZ (LANL). 7. Tl on 1-cm² MgO (STI/UCLA).

Results of a preliminary experiment in our laboratory tend to support the notion that defects and impurities may be responsible for the high residual R_s in HTS. Shown in Fig. 8 are R_s values as a function of the oxygen stoichiometry for high-quality bulk specimens. Before oxygen removal by Zr gettering, R_s of each sample was measured and found to be approximately 0.3 m Ω at 4 K and 3 GHz. Oxygen content was determined by iodometric titration. Individual samples had some fraction of the oxygen removed and the subsequent R_s value determined. The results demonstrate the sensitivity of R_s to oxygen stoichiometry. In essence, oxygen removal is increasing the concentration of defects in the sample, thereby reducing the electron mean free path and coherence length. This disruption of the order parameter yields normal conducting electrons which contribute to R_s , especially at $T \ll T_c$.

Fig. 7. Magnetic surface field dependence of R_s for YBCO bulk, crystal, and film samples. The bulk data were scaled by ω^2 to 6 GHz. Cu data are shown for comparison.

Fig. 8. Dependence of R_s on oxygen stoichiometry x in $\text{YBa}_2\text{Cu}_3\text{O}_x$. Measurements were done at 3 GHz and 4 K.

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