Non-Uniform Transition Conductivity of Superconducting Ceramic

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SUMMARY

In this work we investigate the effect of microstructural variations on the superconducting properties of $SmBa_2Cu_3O_x$. A scanning eddy current probe revealed the onset and growth of a normal conducting region. Resistance versus temperature measurements taken at different regions of the sample support the concept of a physically mixed state system. Regional variations in porosity and grain size distributions affect the observed superconducting transition.

INTRODUCTION

There is an extensive international research effort underway to understand the origin of high critical temperature (T_c) superconductivity. A considerable amount of this research is directed toward increasing both the critical current density (J_c) and T_c [1-4]. In order to increase both T_c and J_c further we must understand the mechanisms supporting the superconducting state and the mechanisms that degrade this state.

An important difference between ceramic and metallic superconductors is that the microstructural variations throughout bulk superconducting metals are generally small when compared to variations in ceramics. For example, porosity variations exist to some degree in bulk superconducting ceramics [5]. It has been found that the transition width, ΔT_c is dependent on the bulk porosity [6]. However, the effect of porosity variations, existing within the bulk of these ceramics, on the superconducting properties is unknown.

In this work we investigate the effect of porosity and grain size variations on the superconducting properties of $SmBa_2Cu_3O_x$. We show that regional variations in porosity and grain size distributions affect the observed superconducting transition.

EXPERIMENTAL

The $SmBa_2Cu_3O_x$ ceramic sample was a uniaxially cold pressed, 2.54 cm diameter, 3.0 mm thick pellet sintered for 8 hr at 930 °C and annealed for 5 hr at 440 °C under O_2. The resulting grain microstructure was orthorhombic, quasi-isotropic (i.e., randomly textured), and single phase as determined from x-ray diffraction analysis. One quadrant of the pellet was used for this investigation. The quadrant was radiographed

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and polished for optical examination after eddy current and resistance measurements were completed. A tone-pulse encoding technique [7] was used to determine porosity and grain size distributions at selected regions of the sample.

The use of eddy currents for the detection of surface flaws is well known. In brief, a coil excited by a continuous high frequency electrical signal yields an output signal that has a magnitude sensitive to changes in the inductance of the coil. A change in the coil inductance is brought about by placing materials that have different conductivities (assuming that we are working with nonmagnetic materials) within the range of the electromagnetic field produced by the coil (this distance called lift-off is usually held constant for scanning systems). Eddy current surface probe evaluation does not yield the actual value of the conductivity where the probe is positioned, but it does identify changes in conductivity. For normal materials the depth of penetration of the magnetic field that induces eddy currents is given by [8],

$$d = [3.14\ \mu_0\mu\sigma f]^{-1/2}$$

where μ_0, μ, σ, and f are the permeability of free space, relative permeability, conductivity and ac frequency, respectively. $SmBa_2Cu_3O_x$ is a paramagnetic material. The relative permeability is approximately 1, therefore, in its normal state the eddy current response is expected to be due to only changes in conductivity. For $SmBa_2Cu_3O_x$ in its normal state the penetration depth is about 250 μm at 4 MHz.

For superconductors the depth of penetration of the magnetic field is given by the London relation [9],

$$d = \frac{d_0}{\left[1 - \left(\dfrac{T}{T_c}\right)^4\right]^{1/2}}$$

where d_0 is the penetration depth of the magnetic field at $T = 0$ Kelvin, and d_0 is estimated to be 0.1 μm for superconducting ceramics [10]. As $T \to T_c$ then the magnetic field completely penetrates the material. The London relation is only dependent on temperature. However, it has been found experimentally [9] that the London depth is dependent on frequency, geometry of sample, surface roughness and the applied magnetic field strength.

The depth of penetration between T_c and $T_c + \Delta T_c$ for ceramic superconductors is unknown. It is expected to be a relatively smooth function having a maximum value of about 250 μm in its normal state and a minimum value of about 0.1 μm in its superconducting.

A commercially available eddy current deflectometer was used for this work. The probe was operated at 4 MHz and had a 1 mm diameter coil. The probe spatial resolution, determined by detecting and imaging a surface crack in $SmBa_2Cu_3O_x$ in its normal state, is about 1 mm.

The experimental setup is shown in figure 1. The sample was placed on a leveled supporting table that is located at the bottom of a large liquid nitrogen dewar. An eddy current probe, attached to a support arm, is scanned over the sample. The probe scans a 10- by 10-mm array (fig. 2) in 1 mm increments. One complete scan of the area could be completed in 10 min. The temperature is continuously measured by a thermocouple placed next to the sample.
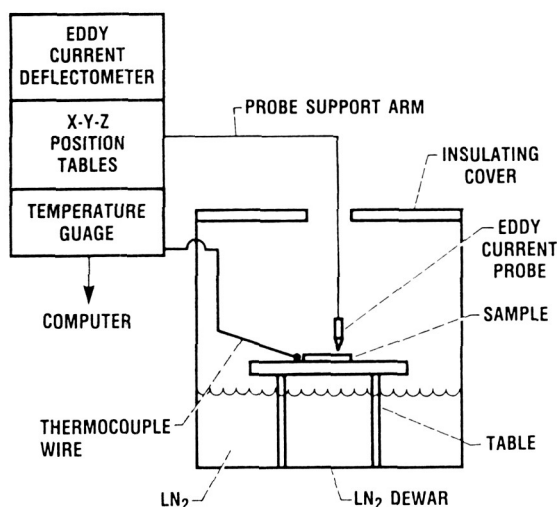
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FIGURE 1. - DIAGRAM OF EXPERIMENTAL SETUP.

During a run the dewar is filled with liquid nitrogen so that the sample is completely immersed. The probe continuously scans the same area of the sample in a repeated fashion while the liquid nitrogen evaporates from the dewar. A square hole in the insulating cover allowed the probe to be moved while limiting turbulent exchange of room air with the nitrogen gas. After the liquid nitrogen level drops below the base of the sample, the temperature of the sample uniformly increased at a rate of 1 K in 10 min. The temperature difference between the top and bottom surface and center to edge of the sample was less than 0.3 K.

Resistance versus temperature measurements were generated during a separate run by using a modified four point measurement arrangement. Six indium contacts [11] were made at the locations shown in figure 2. The extreme two contacts were connected to a constant current source. No "resistive heating" effects were observed for currents below 35 mA. The resistance at the center, R_1, and at the edge, R_2 were simultaneously measured as a function of temperature.
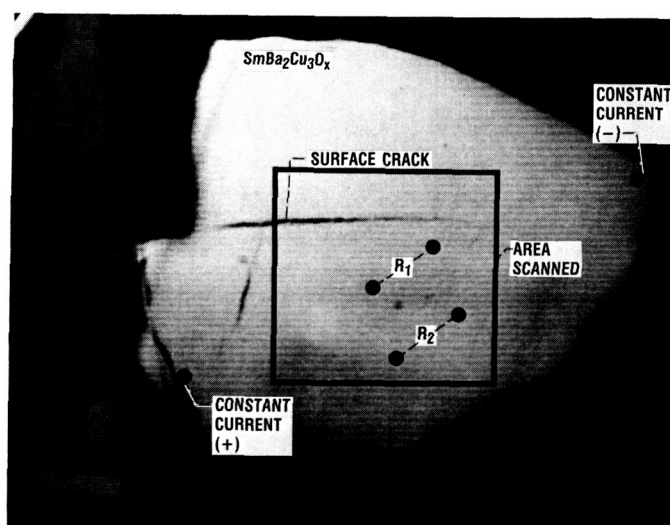

FIGURE 2. - RADIOGRAPHIC IMAGE OF SAMPLE SHOWING THE AREA SCANNED, ELECTRICAL CONTACT POINTS, AND SURFACE CRACK.

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The eddy current response images and the resistance versus temperature data are shown in figure 3. The resistance data are normalized, for
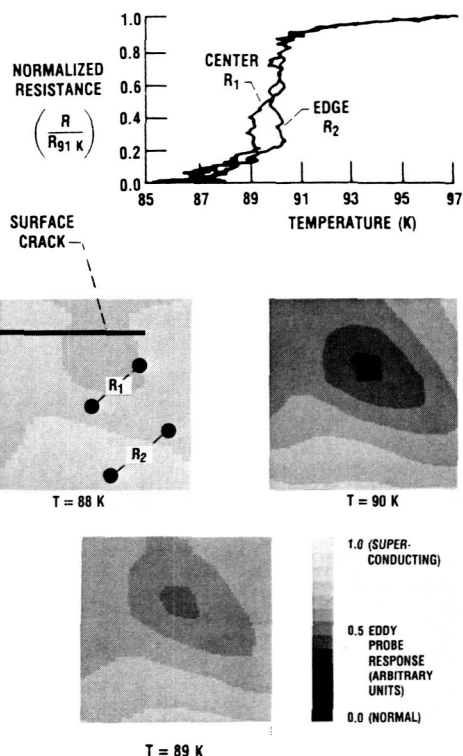

FIGURE 3. - EDDY CURRENT RESPONSE IMAGES, AND RESISTANCE VERSES TEMPERATURE DATA AT THE CENTER AND EDGE OF THE SAMPLE.

comparison, by dividing the measured value by the value at 97 K. The resistance versus temperature data exhibit temperature fluctuations of about ±0.25 K. These fluctuations are due to the actual temperature drifts of the exchange gas within the large dewar. At 85 K, the sample is completely superconducting. At 88 K there is change in the conductivity of the sample. The superconducting-to-normal transition begins at about the center of the image as indicated by the darkest area. The resistance, R_1, at the center is slightly higher at the center than at the edge, R_2, of the sample at this temperature. At 89 K an isolated region of high resistance is identified near the center of the image. At 89.5 K the resistance at the center exceeds that of the edge by about a factor of 2. The eddy current response at 90 K indicates that the center of the sample is almost completely normal and surrounded by a region having some residual superconductivity.

A surface crack developed during the thermal cycling of the sample. The surface crack was located about 2 mm above the central normal zone (figs. 2 and 3) and was detectable only at temperatures above 91 K when the sample was everywhere in its normal state.

The grain and porosity size distributions at the center and edge of the sample are shown in figure 4. No anisotropy was observed for either the grain or porosity distributions. The porosity fractions at the center and edge of the sample are 0.24 and 0.23, respectively. The mean pore sizes are 34±1.5 and 31±1.8 μm at the center and edge, respectively. The mean grain sizes are 7.3±0.35 and 6.2±0.32 μm at the center and edge,
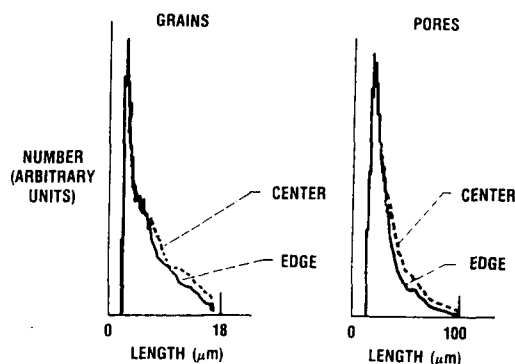
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FIGURE 4. - GRAIN AND POROSITY DISTRIBUTIONS
AT THE CENTER (DASHED LINE) AND THE EDGE
(SOLID LINE) OF THE SAMPLE.

respectively. The results indicate that the mean pore and grain sizes are about 9 and 18 percent, respectively, greater at the center of the sample than at the edge.

DISCUSSION

The eddy current response images (fig. 3) showed that as the sample temperature increases, the center of the sample begins to go normal before the outer edge. Between 89 and 90 K, both the eddy current response and the resistance measurements indicate that the superconducting-to-normal transition begins at and grows from the center of the sample.

Previous work [6] indicated that the superconducting transition temperature decreases and the transition width increases with increasing bulk porosity fraction and grain size. Figure 3 indicates that the total transition width is about the same for both the center and edge, however, the data for the central region does exhibit a much sharper transition jump at 90 K. The increase in mean pore and grain size at the center of this sample is likely to be responsible for the different transition structure in this region. It should be noted that these microstructural variations may have affected the degree and uniformity of oxygenation throughout the bulk of the sample.

The shape of the resistance versus temperature curves in figure 3 resemble that of the superconducting bolometers used widely in superfluid physics research [12]. These bolometers are electronically biased into their transition region for the measurement of temperature variations in the microkelvin range. Wherever there is a nonzero resistance supporting a current there is joule heating. For superconducting metals, joule heating effects are minimized by their high thermal conductivity (1.0 W $cm^{-1}K^{-1}$) and by use of the appropriate thermal sinking methods. A typical metallic sample was, throughout the bulk, at the same temperature within a few millikelvin. In contrast, the low thermal conductivity (5.0×10^{-3} W $cm^{-1}K^{-1}$) of bulk high temperature ceramics [13] is expected to promote local joule heating. It has been speculated [6] that superconducting ceramics may exhibit varying local superconducting properties or a physically mixed state system. Our results indicate that regional nonuniformity of porosity and grain size distributions encourage localized superconducting transitions to take place over different regions of a high critical temperature ceramic superconductor. We believe that local heating on a millikelvin level, at the porous or granular areas, is the mechanism responsible for affecting the observed superconducting transitions.

CONCLUSION

In this work we investigate the effect of microstructural variations on the superconducting properties of $SmBa_2Cu_3Ox$. A scanning eddy current probe revealed the onset and growth of a normal conducting region. Resistance versus temperature measurements taken at different regions of the sample support the concept of a physically mixed state system. Regional variations in porosity and grain size distributions affect the observed superconducting transition.

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15. Supplementary Notes

16. Abstract

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