Ag SCREEN CONTACTS TO SINTERED YBa$_2$Cu$_3$O$_x$ POWDER
FOR RAPID SUPERCONDUCTOR CHARACTERIZATION


Abstract

We have developed a new method for making current contacts and voltage taps to YBa$_2$Cu$_3$O$_x$ sintered pellets for rapid superconductor characterization. Ag wire screens are interleaved between calcined powder sections and then fired at 930°C to form a composite pellet for resistivity and critical current measurements. The Ag diffuses into the powder during the sintering process forming a proximity contact that is permeable to O$_2$. Contact surface resistivities (area-resistance product) range from 1 to 10 $\mu\Omega$-cm$^2$ at 77 K for the Ag-powder interface. In this configuration, current can be uniformly injected into the ends of the pellet through the bonded Ag screen electrodes. Also, Ag screen voltage contacts, which span a cross section of the pellet, may provide an ideal geometry for detecting voltage drops along the pellet, minimizing current transfer effects.

Introduction

Ag is a good candidate for contacts to YBa$_2$Cu$_3$O$_x$. The melting point of Ag is 960°C, close to the YBa$_2$Cu$_3$O$_x$ annealing temperature range of 900°C to 950°C. There is evidence that, even at temperatures as low as 600°C, Ag films can diffuse into the surface of sintered powder YBa$_2$Cu$_3$O$_x$ samples without substantially harming the superconducting properties of YBa$_2$Cu$_3$O$_x$. The oxides of Ag are unstable at high temperatures, preventing the formation of an insulating layer at the Ag-YBa$_2$Cu$_3$O$_x$ interface. Ag is permeable to O$_2$ at high temperatures, allowing the YBa$_2$Cu$_3$O$_x$ in the vicinity of the contact to be fully oxygenated.

In this paper we discuss the use and potential uses of Ag screen contacts that have been laminated into YBa$_2$Cu$_3$O$_x$ pellets for making low resistance contacts for rapid superconductor characterization. Ag screens can be used for making external as well as internal contacts to samples of YBa$_2$Cu$_3$O$_x$. We have found that surface resistivities as low as 1 $\mu\Omega$-cm$^2$ can be obtained for Ag screen contacts to YBa$_2$Cu$_3$O$_x$ that were annealed at 930°C. One of the advantages of Ag screen contacts is that the sample electrodes are an integral part of a YBa$_2$Cu$_3$O$_x$ pellet incorporated during the O$_2$ annealing step without requiring additional process steps.

The contacts have been used to measure the $J_c$ of differently shaped samples of the same materials to investigate geometry effects. Apparently, the self field limits the $J_c$ of samples with larger cross sectional area. Some other potential applications of Ag contacts to YBa$_2$Cu$_3$O$_x$ include transport resistance detection of higher $T_c$ fractions in sintered powder samples and reinforcement and grounding of superconducting YBa$_2$Cu$_3$O$_x$ electromagnetic shields.

Sample Preparation

Laminated YBa$_2$Cu$_3$O$_x$-Ag screen pellets were prepared from reground calcined powder and Ag wire mesh. The YBa$_2$Cu$_3$O$_x$ powder was made by heating a stoichiometric mixture of BaCO$_3$, Y$_2$O$_3$, and CuO to 930°C, holding for 10 h, and then cooling at a rate of 2.4°C/min to 450°C in air. We used two sizes of square Ag mesh to make the screen contacts. The first had 31.5 wires per cm (80 mesh, 0.11 mm wire diameter). The second had 7.87 wires per cm (20 mesh, 0.30 mm wire diameter). The Ag screens and YBa$_2$Cu$_3$O$_x$ powder were pressed together using a circular pellet die with a diameter of 1.905 cm. The pressure used to compress the laminated composites was 20 MPa. We made pellets with 80 mesh external end contacts (the source for the sample type in Fig. 1a), and pellets with 80 mesh external end contacts and 20 mesh internal contacts (the source for the sample type in Fig. 1b). One pellet had an annular 80 mesh screen annealed into its top surface (the source for the sample type in Fig. 1c). After the composite pellets were pressed they were sintered at 930°C for 10 h in flowing O$_2$ gas followed by slow cooling to 450°C at a rate of 2.4°C/min. We found that lower sintering temperatures gave mechanically unreliable contacts that easily delaminated. Samples were dry cut from the pellets into the configurations shown in Fig. 1 with a diamond saw. The samples were cut so that they had square cross sections. Surface voltage contacts were ultrasonic solder bonds made directly to the YBa$_2$Cu$_3$O$_x$ surface using InAg(2% Ag) solder. Contact to Ag screens was also made using the ultrasonic soldering method. Typically voltage contacts were several millimeters apart along samples that were around 1 to 2 cm long.

Figure 1. The Ag electrode configurations used for the contact resistance and $J_c$ measurements of the laminated samples. Figure 1a shows a sample with end contacts and surface voltage taps. Figure 1b shows a sample with end contacts and internal voltage screen electrodes. Figure 1c shows surface screen electrodes and surface voltage taps. Extra voltage taps on the sample in Fig. 1c are used to measure current transfer voltages near the current injection point of the sample. Voltage taps on current leads in the vicinity of the current contacts were employed during V-I measurements of the contacts themselves.

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The 80 mesh wire was used for current contacts on the surface of the samples because it provided more contact area per unit cell to the YBa$_2$Cu$_3$O$_y$. We tried firing silver foil (0.1 mm thick) onto the samples but found that the contacts delaminated easily unless the samples were fired near 960°C (near the melting point of Ag) where partial melting occurred and the Ag visibly diffused into the pellets as much as 0.2 mm. We therefore chose the wire mesh to avoid excessive diffusion and yet maintain electrical and mechanical integrity. The 20-mesh screens were chosen for internal voltage contacts to minimize the effective reduction of superconductor cross sectional area: 23% reduction for 20 mesh compared to 33% for the 80 mesh.

**Apparatus**

The voltage current curves for the voltage tap pairs on the sample were recorded simultaneously with a digitizing oscilloscope. An analog nanovoltmeter, with an amplified output, was used to measure the voltage at each tap pair. Two battery-powered supplies having 0 to 10 A and 0 to 1000 A ranges were used to supply sample current. The sample voltages were measured at numerous sample currents. Several electric field criterion were employed when determining $I_c$. We found that the 1 $\mu$V/cm electric field criteria was a reasonable choice for comparison purposes.

Another parameter that was determined during the $I_c$ measurement is $n$, defined by the approximate relationship, $V = V_o (I/I_o)^n$, where $I_o$ is a reference critical current at a voltage criterion $V_o$. $V$ is the sample voltage, and $I$ is the sample current. $n$ has a typical value of 20 to 60 for conventional superconductors (a higher value means a sharper transition). A lower value of $n$ can be caused by sample inhomogeneities or self-field quenching (depending on sample geometry).

All measurements were performed at 77 K in liquid $N_2$. The samples were not shielded from the earth's magnetic field.

**Contact Resistance Measurements**

Along with the voltage contacts on and in the samples we also soldered voltage taps to the current leads attached to the Ag screen contacts as shown on all of the sample types in Fig. 1. In this way, we could measure the contact resistance and determine the surface resistivity (the area-resistance product or "$\rho_s$") of the contact. Figure 2 shows a typical R-I ($R = V/I$) curve of two Ag-screen current contacts on one sample. The resistance is roughly constant for currents below the $I_c$ of this sample of 5.03 A. At $I_c/2$ the resistance of the two contacts was 11 and 31 $\mu$V-m, corresponding surface resistivities of 1.7 and 6.8 $\mu$V-cm$^{-2}$. Above $I_c$ the voltage across the contact voltage taps markedly rises due to the included section of sintered YBa$_2$Cu$_3$O$_y$ becoming normal. Of the nine samples tested thus far (two contacts per sample), the value of the surface resistivity for this type of contact using the 80 mesh screen is $3 \pm 2 \mu$V-cm$^{-2}$. We think that the variability of the surface resistivity may be due to partial delamination of the Ag screen.

**Critical Current Measurements**

Figure 3 shows a log-log plot of the electric field as a function of current for a YBa$_2$Cu$_3$O$_y$ sample with external Ag screen current contacts and external InAg voltage taps (see Fig. 1a). The nanovoltmeter was operated at several sensitivities. Figure 4, on the other hand, shows the data for a YBa$_2$Cu$_3$O$_y$ sample with external Ag screen current contacts and internal Ag screen voltage taps (see Fig. 1b). The noise at the lower ends of the voltage ranges for a given nanovoltmeter setting for the sample with the internal Ag screen voltage taps was less than that observed in the samples with external InAg contacts. This is probably due to the relatively high surface contact resistivity of the InAg contacts (about 0.01 n-cm$^2$) compared to that of the Ag screen contacts. Also, the critical current in Fig. 3 is higher than that of the critical current in Fig. 4 even though the cross sectional area of the samples was about the same. This was because the internal Ag screen voltage taps decrease the effective superconducting cross sectional area.

**Self-Field Effects**

$J_c$ as a function of the inverse of the thickness of the YBa$_2$Cu$_3$O$_y$ samples (square cross sections) is plotted in Fig. 5. Samples represented by squares are of the type shown in Fig. 1a with external voltage taps. Samples represented by circles are of the type shown in Fig. 1b with internal voltage taps. Instead of being independent of the size of the sample, $J_c$ is larger for smaller cross sections. This is probably due to self field quenching of the superconductor at the surface of the sample. This is not surprising considering the weak link nature of these materials.

**n value effects**

Figure 6 shows the $n$ value as a function of the inverse of the sample size. Again, the sharpness of the transition, represented by the $n$ value, depends on sample size, as in the case of $J_c$. Here however the $n$ value decreases for the samples having internal Ag screen voltage taps and increases for the samples with external voltage taps as the size of the sample is reduced. The $n$ value increasing with decreasing sample size is consistent with self field quenching. We believe that the reduction of the $n$ value for internal voltage tap samples is due to the effective reduced cross sectional area of the superconductor within the internal screen. This has its greatest effect on the smallest sample where the total cross sectional area of the sample is close to that of the unit cell of the mesh of the screen.
Figure 3. The electric field versus current for a sample of the type shown in Fig. 1a (T = 77 K). The sensitivity of the nanovoltmeter ranged from 10 μV to 10 mV.

Figure 4. The electric field versus current for a sample of the type shown in Fig. 1b (T = 77 K). The sensitivity of the nanovoltmeter ranged from 10 μV to 10 mV.

Figure 5. $J_0$ as a function the inverse of the thickness of the sample (square cross section).

Figure 6. $n$ value as a function of the inverse of the thickness of the sample (square cross section).
Possible Applications

High Current Contacts for Jc Measurements: Surface versus End Geometries

We have shown data for Ag screen contacts designed for current injection into the ends of a sample. As discussed in the introduction, the main advantage of this geometry is to insure that there is no current transfer in the sample that could give erroneous \( J_c \) results, especially for extremely short samples, or samples with large aspect ratios (diameter to length). We found, however, that samples tested using the weak link nature of the sample that sufficiently for current injection into the ends of a sample. As discussed in the introduction, the main advantage of results, especially for extremely short samples, this geometry is to insure that there is significant current transfer between opposed voltage taps on either side of the sample mounted near the current injection point. This may be due to the weak link nature of the sample that sufficiently randomizes the current immediately after it enters the sample.

In fact, the end contacts have some disadvantages over the surface contacts. For small cross sectional areas the current contacts are mechanically weak and easily delaminate from the sample. The self-field effects in the weakly linked materials limit the size of the sample. Finally, for higher \( J_c \)'s, above 100 \( \text{A/cm}^2 \), the surface resistivity of the Ag screen contacts may be large enough to cause heating at the contacts (depending on the stabilizing cooling power of the liquid \( N_2 \) - \( \text{YBa}_2\text{Cu}_{3}\text{O}_y \) interface).

Internal Ag screen voltage contacts provide low noise measurements of \( I_c \). However, the meshes used here displaced enough of the superconductor from the cross section of the sample to severely affect the resulting \( J_c \) measurements. Still, the data are inconclusive as to the potential benefits of internal voltage taps. Changing the mesh so that the fraction of superconductor displaced from the cross section of the sample is reduced to an acceptable level may result in n value of the transition that is independent of the size of the sample.

Contacts for Resistivity Measurements: The Search for Higher \( T_c \)'

Transport resistivity measurements as a function of temperature are very similar to \( J_c \) measurements. One might say that all conductors are superconductors even at room temperature - it's just that their critical currents are very low. With this in mind, we propose the following experiment. Using the internal voltage contact configuration (Fig. 1b) in a resistivity measurement should allow detection of small, low-\( I_c \), fibril superconducting links of higher \( T_c \) material buried in the interior of a sample.

Contacts for Superconducting Shields

\( \text{YBa}_2\text{Cu}_{3}\text{O}_y \) sintered powder has the potential of becoming a useful shield material. At 77 K it presently has the same shielding capability as copper at an equivalent weight. For low dc magnetic field shielding it may already have some uses. However, the matter of grounding or biasing the shield is still unresolved. Also, \( \text{YBa}_2\text{Cu}_{3}\text{O}_y \) is a low-strength brittle material. We propose that the Ag screens be laminated into a \( \text{YBa}_2\text{Cu}_{3}\text{O}_y \) shield to provide good grounding paths within the sintered powder, as well as to improve the structural integrity of the shield.

References


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