Ag SCREEN CONTACTS TO SINTERED $\text{YBa}_2\text{Cu}_3\text{O}_x$ POWDER
FOR RAPID SUPERCONDUCTOR CHARACTERIZATION†

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Abstract

We have developed a new method for making current contacts and voltage taps to $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 $10 \mu\Omega\text{-cm}^2$ 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$.^{1,2} The melting point of Ag is 960°C , close to the $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$ samples without substantially harming the superconducting properties of $\text{YBa}_2\text{Cu}_3\text{O}_x$.³ The oxides of Ag are unstable at high temperatures preventing the formation of an insulating layer at the Ag- $\text{YBa}_2\text{Cu}_3\text{O}_x$ interface. Ag is permeable to O_2 at high temperatures, allowing the $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$ pellets for making low resistance contacts for rapid superconductor characterization. Ag screens can be used for making external as well as internal contact to samples of $\text{YBa}_2\text{Cu}_3\text{O}_x$. We have found that surface resistivities as low as $1 \mu\Omega\text{-cm}^2$ can be obtained for Ag screen contacts to $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$ include transport resistance detection of higher T_c fractions in sintered powder samples and reinforcement and grounding of superconducting $\text{YBa}_2\text{Cu}_3\text{O}_x$ electromagnetic shields.

Sample Preparation

Laminated $\text{YBa}_2\text{Cu}_3\text{O}_x$ -Ag screen pellets were prepared from reground calcined powder and Ag wire mesh. The $\text{YBa}_2\text{Cu}_3\text{O}_x$ powder was made by heating a stoichiometric mixture of BaCO_3 , Y_2O_3 , and CuO to 930°C , holding for 10 h, and then cooling at a rate of $2.4^\circ\text{C}/\text{min}$ 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 $2.4^\circ\text{C}/\text{min}$. 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$ surface using $\text{InAg}(2\% \text{ Ag})$ 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$. 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 digital processing 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.⁴ The sample voltages were measured at numerous sample currents. Several electric field criterion were employed when determining J_c . We found that the $1 \mu\text{V}/\text{cm}$ 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, $V = V_0(I/I_0)^n$, where I_0 is a reference critical current at a voltage criterion V_0 , 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 " ρl ") 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 these two contacts was 11 and 31 $\mu\Omega$ with corresponding surface resistivities of 1.7 and 4.8 $\mu\Omega\text{-cm}^2$. Above I_c the voltage across the contact voltage taps markedly rises due to the included section of sintered $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 $3 \pm 2 \mu\Omega\text{-cm}^2$. We think that the variability of the surface resistivity may be due to partial delamination of the Ag screen.

Critical Current Measurements

V-I Curves

Figure 3 shows a log-log plot of the electric field as a function of current for a $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$ sample with external Ag screen current contacts and internal Ag

Figure 2. The resistance versus current of a Ag screen current contact to $\text{YBa}_2\text{Cu}_3\text{O}_x$ ($T = 77 \text{ K}$).

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 $0.01 \Omega\text{-cm}^2$) 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 $\text{YBa}_2\text{Cu}_3\text{O}_x$ 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 meter ranged from 10 μ V 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 meter ranged from 10 μ V to 10 mV.

Figure 5. J_c 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 J_c 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 configuration shown in Fig. 1c, where current was injected into the surface of the sample, did not show significant current transfer voltage 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 A/cm², 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 N₂ - YBa₂Cu₃O_x 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 an n value of the transition that is independent of the size of the sample.

Contacts for Resistivity Measurements: The Search for Higher T_c 's

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

YBa₂Cu₃O_x 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, YBa₂Cu₃O_x is a low-strength brittle material. We propose that the Ag screens be laminated into a YBa₂Cu₃O_x shield to provide good grounding paths within the sintered powder, as well as to improve the structural integrity of the shield.

Summary

Ag screen contacts can be used to make low resistance contact to sintered YBa₂Cu₃O_x powder superconductor. The following comments summarize the results obtained when using the screens for J_c measurements.

1. The Ag screen contacts can be incorporated into the sample during the O₂ annealing step without additional processing steps.
2. The surface resistivity of the Ag screen contacts can be as low as 1 $\mu\Omega$ -cm². Care must be taken, however, to avoid delamination of the contacts.
3. End contacts provide an ideal geometry for injecting current into YBa₂Cu₃O_x samples. However, we did not find significant current transfer problems in top-surface-mounted current contacts.
4. Surface resistivities for the Ag screen contacts are sufficient for lap joints but may be insufficient for end current injection into samples with higher J_c 's.
5. Internal Ag screen voltage taps provide low resistance, low noise contact to the sample. However, coarse mesh size can result in a substantial reduction in the effective cross sectional area of the sample.

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