ABSTRACT

We have studied the behavior of high-quality YBa2Cu3O7-x (YBCO) thin films with Ag over-layers. We chose to study Ag in detail because of its widespread use as contact metalization and our earlier studies of proximity effects in YBCO. The details of transport critical current measurements are presented. The Ag coatings can reduce normal state resistance while not degrading the critical current density, Jc.

I. INTRODUCTION

Practical applications of high temperature superconductors such as YBa2Cu3O7-x (YBCO) will probably be demonstrated first using high quality, nearly epitaxial thin films. Such films have crystal current densities (Jc) in excess of 106 A/cm² at 77 K. The films are, however, difficult to contact and have high normal state resistivities (50-100 μΩ-cm at 100 K). High-current applications such as ground planes and power busses for integrated circuits will require not only high Jc, but reasonable thermal stability and ease of external contact. A thin film of Ag, deposited on the YBCO and annealed, can provide these necessary properties without degrading the Jc. We have demonstrated this by measuring transport Jc in epitaxial films with and without Ag over-layers. A normal metal over-layer (N) can affect a superconducting film (S) by straightforward thermal and/or electrical parallel shunting; this has been exploited in practical superconductors for stabilization in high-current applications [1]. S-N bilayers can also exhibit the superconducting proximity effect, which is crucial to many device applications for thin film superconductors, for example, in Josephson S-N-S (superconductor-normal-superconductor) junctions, proximity effect junctions, and low resistance ohmic contacts.

II. EXPERIMENTAL

We grew the YBCO films using an in situ laser ablation process [2]. The films were patterned into microbridges which range in width from 4 to 500 μm. The critical current density was measured as a function of magnetic field, field orientation, and temperature. Presently, the best films have current densities over 10⁷ A/cm² at 4 K and up to 2 T. A thin over-layer of Ag (10-200 nm) can decrease the composite resistivity and still have little or no effect on Jc. The field dependence of Jc was measured up to 10 T in films with 10-20 nm Ag layers. An increase in Jc at 1 T was observed and reported [3] for the case where the field was oriented in the plane of the YBCO film. The Ag layers did not appreciably affect Jc at 4 K in any field orientation.

We used a standard pulsed laser ablation process to grow our YBCO films in situ. The laser was a frequency tripled Nd:YAG laser (λ = 355 nm), with a 10 ns pulse width, 200 mJ pulse, and a 10 Hz repetition rate. The laser spot was focused to produce a fluence of 1-2 J/cm² on a stoichiometric YBCO target. A single cryostat substrate of SrTiO3 or LaAlO3 was heated to 700-750°C in O2 at a pressure of 27-33 Pa (200-250 mTorr). The films were grown at a rate of 1-2 nm/s to final thicknesses ranging from 200 to 400 nm. The resulting zero-resistance transition temperatures, Tc, were between 87 and 91 K. The films were patterned by a wet etch: saturated ethylenediaminetetraacetic acid (EDTA) [4] in H2O with an etch rate of 50 nm/min. The resist process consisted of a conventional positive photoresist and aqueous developer [5]. After etching, the resist was stripped in acetone and Ag pads were deposited through a metal stencil. In some cases, the Ag pads were lifted off using a photoresist mask. Contact resistances were reduced by annealing at 500°C in flowing oxygen for 15-60 min. A drawing of our sample geometry is shown in Fig. 1.

The measurements were carried out in three different experimental cryostats with different room temperature electronics. The low-current, variable temperature probe was designed for low-noise device testing and was equipped with filtered room temperature leads. The high-current, ac cryostat incorporated a novel temperature control scheme and was able to measure Jc on the fly. The high-current dc cryostat was used for extremely careful dc measurements of Jc in external fields up to 10 T.

The low-current filtered apparatus (cryostat I) is shown schematically in Fig. 2. Temperature control is maintained by first stationing the probe in the cold gas above the liquid He. Then a calibrated carbon-glass resistor is used as a thermometer, and a feedback loop controls current to the heater resistor. Between 70 and 90 K, the temperature can be stabilized to better than 10 mK. Resistance vs. temperature data can be obtained by measuring the sample resistance as the probe cools to the temperature of the He gas. Critical currents are taken from current-voltage characteristics obtained using a dc preamplifier and a swept dc current source. An electric field criterion of 10 μV/cm was used.

The high-current dc testing apparatus (cryostat II) was designed to measure the V-I characteristic using a computer-controlled data acquisition system [6]. The current was ramped to predetermined set points and held constant while the sample voltage and current were measured and averaged. Set points which allowed for a logarithmically smooth V-I curve were established. A linear fit on the logarithmic scale was used to determine Jc at an electric field criterion of 1.0 μV/cm. The sample was immersed in liquid helium (4 K) or liquid nitrogen (76 K). Either soldered or pressure contacts were used for current and voltage leads. The
samples were tested in a superconducting magnet for the 10 T data and between the poles of a copper electromagnet for the data to 1 T at 76 K.

![Diagram of the low-noise, variable temperature experimental apparatus (Cryostat I).](image)

The third cryostat (III) incorporated a cryogenic bathysphere for measuring $J_c$ versus $T$ curves for samples in high magnetic fields [7]. The essential feature is an inverted stainless steel dewar that insulates a helium vapor from surrounding liquid helium in the bore of the magnet. The sample, thermometers, and a heater are located in the vapor space thermally anchored to one another through a copper holder. Platinum and carbon glass resistor thermometer sensors are used for operation in magnetic fields up to 7 T. We estimate the temperature error to be less than 1 K. Figure 3 shows the $J_c(H,T)$ characteristic for an uncoated sample, representative of the data taken in cryostat III.

Contact resistance is a limiting factor in $J_c$ measurements of these high quality films. The general approach to transport current measurements in high-$J_c$ films is to make a narrow constriction in the film by any of a number of patterning techniques. For example, a 0.1 mm wide strip of 250 nm thick YBCO would have a critical current of 2.5 A for a $J_c$ of $10^7$ A/cm². This would dissipate over 600 mW if the contact resistance were as high as 0.1 $\Omega$. By narrowing the line to 10 $\mu$m in width, the power dissipation can be reduced to 6 mW for the same contact resistance. The approach we adopted was to use the widest line convenient for measurement, on the general premise that patterning damage effects become less and less important as the linewidth increases. We also optimized our contact schemes to obtain the contact resistances needed to do the experiments. The results of one optimization experiment are shown in Fig. 4. The experimental geometry consists of a thin film Ag strip bridging the gap. The contacts were 16 $\mu$m in area; the Ag was annealed in a series of tests to determine the resistance of the contact as a function of annealing time. The $R(T)$ characteristics show that the resistance decreased after each anneal. The resistance shows evidence of proximity coupling at temperatures below 30 K as seen in Fig. 4(b). The flat $R(T)$ curve begins to bend down with decreasing temperature, probably due to the contact region being driven superconducting through proximity with the underlying YBCO. In another test, the resistance of another composite structure approached the resistance of the Ag bridge, approximately 1 $\Omega$, after 8 anneals, corresponding to a specific resistance of the contact ($R_{contact} \times area$) less than $10^{-9}$ $\Omega$-cm².

$J_c(H)$ in external field is shown in Fig. 5 for a Ag coated sample. The plot contains data at 4 K in fields up to 10 T and data at 76 K in fields up to 1 T. The lack of weak link behavior is evident from the high $J_c$ values, and the lack of substantial roll-off of $J_c$ up to 1 T. The $J_c$ of this sample (L77.2) at 1 T was higher than the $J_c$ of samples which were not Ag coated. This phenomenon is discussed in detail elsewhere [3]. In Fig. 5, the squares are $J_c$ data when the external field was aligned perpendicular to the c axis (in the plane of the film). The circles represent data for the case where the field was parallel to the c axis (perpendicular to the film surface).
The data presented in Reference 3 include only films where $I_c$ of coated samples was greater than or equal to the $I_c$ of uncoated films. Figure 6 shows the effect of reduced $H$ dependence for a coated film in terms of $I_c$ normalized to the zero field value. In this case, the $I_c$ after coating was lower than before coating. However, the normalized values are larger for the Ag-treated sample at 1 T. Thus, even though the sample was damaged during the Ag processing, there is still a reduced dependence of $I_c$ on field perpendicular to the c axis.

Figure 6. A comparison of normalized $I_c$ for sample L2-96B before and after Ag deposition. The $I_c$ values are normalized to the zero field value. This sample was tested, then was coated with 10 nm of Ag.

The effect of a thick layer of Ag on the sample resistance can be seen in Fig. 7, a plot of the R(T) characteristic of two different samples. The hope is that the composite resistance would decrease without affecting the $T_c$. In the comparison of two similar $I_c$ samples, it is evident from the inset of Fig. 7 that in fact the $T_c$ and transition widths are very similar. $I_c$ is a much more sensitive measure of film quality. In Fig. 8, $I_c$ is plotted vs. temperature for sample L3-11B after 200 nm of Ag was deposited. The square data point represents the value of $I_c$ after 10 nm of Ag, before the final thick layer was deposited. The zero-field value of $I_c$ actually increased after Ag processing. We can explain some of
the improvement as a result of the more complete re-oxidation of the YBCO after the Ag anneal. All samples had Ag pads deposited and annealed. The measured \( J_c \) could be diminished if the pad anneal was insufficient to restore oxygen lost in the vacuum evaporation of the Ag.

The temperature dependence of \( J_c \) has been explained by weak link effects [9] as well as the more conventional flux motion voltages [8]. We have studied \( J_c(T) \) in both cryostats I and III, as seen in Figs. 3, 8 and 9. Further work in analyzing our \( J_c \) data in terms of flux creep models and weak link models is in progress.

IV. DISCUSSION

Three measurement systems, with different contacting methods and temperature control, were used in this study. A comparison of the results shows that these systems together form a uniquely well-qualified test bed for the characterization of patterned thin films. Figure 9 shows data obtained in cryostats I and II. The open circle represents \( J_c \) obtained in liquid nitrogen; the closed circles are data obtained in He gas in cryostat I. The measurements were made more than one month apart. The similarity in \( J_c \) values implies that the different measurement set-ups were consistent, and that there is little sample degradation in a one month period.

The key technological result is that the various thicknesses of Ag that we used did not reduce \( J_c \) or \( J_c(H) \). Critical current densities in excess of \( 10^6 \) A/cm² have been achieved at temperatures greater than 76 K. An unusual effect was seen in \( J_c(H) \) when the field was oriented perpendicular to the c axis of the film. The \( J_c \) at 1 T was higher in samples with 10 nm coatings of Ag than in similar uncoated samples [3].

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REFERENCES

5. The photoresist used was Shipley 1400-31 with the 0-30 developer. (This statement is included for the important chemical information it contains, and is neither an endorsement of the products by the U.S. Government nor does it imply that the materials are necessarily the best available for the purpose.)
8. Campbell and Evetts, *op. cit.* Ch. 2.