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TITLE: SUPERCONDUCTING PROPERTIES OF Tl-Ba-Ca-Cu-O FILMS ON SILVER SUBSTRATES

AUTHOR(S): R. C. Dye, P. N. Arendt, J. A. Martin, K. M. Hubbard, N. Elliott and G. Reeves

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SUPERCONDUCTING PROPERTIES OF Tl-Ba-Ca-Cu-O FILMS ON SILVER SUBSTRATES

R. C. Dye, P. N. Arendt, J. A. Martin, K. M. Hubbard, N. Elliott and G. Reeves, Los Alamos National Laboratory, Los Alamos, NM 87545

Abstract

Films of Ba-Ca-Cu-O have been rf magnetron sputtered onto Consil 995 substrates. A post deposition anneal in an over pressure of Tl produces the superconducting 1212 and 2212 phases. Varying the annealing procedures changes the electrical properties of the final films dramatically. Dynamic impedance, a novel approach to the electrical characterization of these films on a conductive substrate is discussed and compared with SEM, XRD and RBS measurements as a function of differing annealing protocols.

Introduction

High-temperature superconducting (HTS) materials on metal substrates have a wide range of potential applications. These applications include HTS tapes, rf and microwave accelerating cavities.[1] Because the Tl based HTS materials have the highest known operating temperatures, these materials show the most promise for immediate device development.

In choosing a substrate for HTS one must consider: the substrate thermal conductivity to the coolant medium; the capability of the material to be machined or shaped into non-planar geometries; its bonding properties to the HTS; the oxidation layer that may form at elevated temperatures; and how the electrical properties of the HTS material are effected. Also, for practical applications the expense of the substrate material should be taken into account. These stringent requirements are met by Consil 995 which is a silver alloy consisting of 99.5% Ag, 0.25% Ni and 0.25% Mg.

Furthermore, to optimize the quality of the HTS films on a metal substrate a rapid and reliable diagnostic technique is needed. Currently the most widely used technique to routinely analyze the electrical properties of high temperature superconductors is the four-point probe resistivity measurement. In many instances, however, a simple d.c.-percolating path measurement may give misleading results. The optimization of the processing parameters can be hindered due to limited information produced by insufficient material characterization. For instance, if a sample of Tl-Ba-Ca-Cu-O contains material which has varying amounts of oxygen a typical four-point probe measurement would only detect the presence of the higher temperature transition. Also, if the material contains the (1212) phase and the (2212) phase only the transition for the (2212) phase will be detected by the four-point measurement. Relying only on a four-point probe measurement to set an experimental procedure can cause insufficient oxygen pressures and inadequate temperatures to be used in the material processing. In general, resistivity through a percolation pathway may not be representative of the global electrical properties of the superconducting material. Additionally, analysis of a HTS on a metal substrate is difficult using the four-point probe measurements due to the current shorting through the conducting substrate material.

To further advance the processing of high temperature superconductors it is desirable to develop more reliable, non-destructive, techniques to analyze the quality of the materials produced.

In this study, the electrical response of Tl-Ba-Ca-Cu-O films on Consil 995 were followed as a function of annealing procedure using a novel inductive technique, dynamic impedance (DI), which can monitor the response of HTS on metal substrates. With this capability a better understanding of the electrical properties and film growth can be obtained.

Experimental Procedures

A metal oxide target with a nominal composition of $Ba_2Ca_1Cu_2O_x$ was used in a 10 cm diameter rf magnetron sputter gun. Substrates were placed 35 mm from the target on a planetary style (dual-axis) rotator. During deposition, as many as five 38 mm diameter substrates may be coated simultaneously. Deposition conditions are; input power = 1.7 W/cm^2, deposition rate = 8 nm/min, film thickness = 0.8×10^4 to 1.0×10^4 nm, and sputter gas composition-partial pressures of $Ar/O_2 = 8 \times 10^{-3}/4 \times 10^{-3}$. The background plasma heats the substrates to over 200° C during deposition. Ion backscattering spectrometry shows the composition of the films to be within 10% of the composition of the target. The substrates used were either 1 cm^2 or 38 mm in diameter.

The post-deposition annealing apparatus used to generate the Tl over pressure has been described previously.[2] Annealing temperatures varied from 885° to 910° C. The dwell times at these temperatures were also varied from a few minutes up to thirty minutes. Several 1 cm^2 films could be annealed simultaneously but only one of the 38 mm diameter films could be annealed at a time.

The dynamic impedance technique (DI) is similar to ac susceptibility measurements. However, the DI technique uses a single inductive coil and measures the out-of-phase (reactive) component at a set drive frequency. One obtains a direct measure of the impedance change in the coil caused by the coupling between the coil and the supercurrents induced in the film.[3]

The measurement technique uses a lock-in amplifier, ac-volt meter, and a temperature control system. The data is collected with a data acquisition system connected to a personal computer. The combination sense/drive coil is 6 mm in diameter and is placed 0.05 mm above the HTS sample on a helium, flow-through, cold-finger. The coil is maintained at a temperature of 28° C with a temperature control system throughout the data collection period.

Results

The results of this study can be summarized by analyzing three representative films grown under three annealing procedures. Film "A" was held at a temperature of 890° C for 1 hour. Film "C" was held at 910° C for 2 min., dropped to 895° C for 12 min., and then raised back to 910° C for 5 min.. Film "B" had the same protocol as "C", however, this sample had a 1 mm gold buffer layer applied between the silver and superconducting layer. The gold film was annealed at 910° C for 15 min. before application of the HTS precursor and final annealing for thallination. All of these samples were heated at a rate of 20° C/min. until 800° C. The rate was then slowed to 10° C/min. due to power limitations of the heating supply. The furnace cooling to room temperature was at 20° C/min..

Comparing the SEM photographs, film A (fig. 1) shows a highly granular texture with very little continuous material. The morphology of film B (fig. 2) is smoother over that of film A. This film is less granular and more continuous. The best morphology is observed in film C (fig. 3). The micrographs show that the film morphology is improving from A to C.

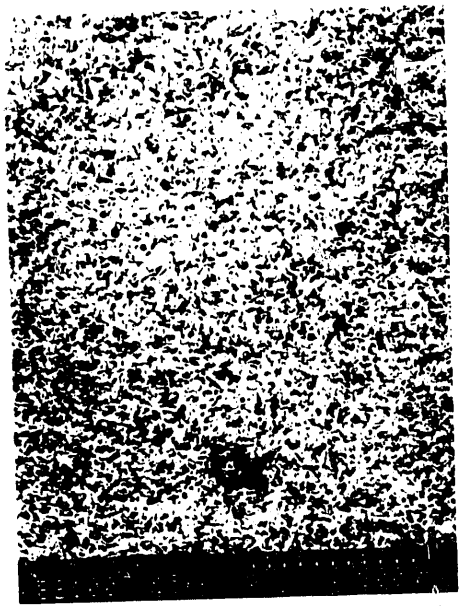

Figure 1. Scanning electron micrograph of HTS film with annealing protocol A.

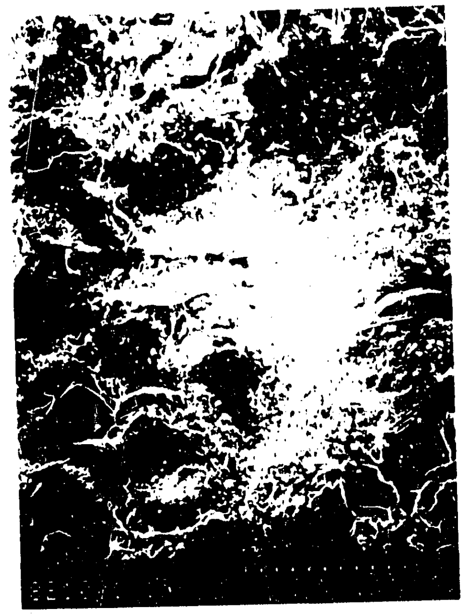

Figure 3. Scanning electron micrograph of HTS film with annealing protocol C.

Figure 2. Scanning electron micrograph of HTS film with annealing protocol B.

To confirm the material changes observed in the SEMs, X-ray diffraction (XRD) was also performed. The XRD of film A (fig. 4) has very little of the HTS phases. In fact, several peaks associated with non-superconducting material are observed. The XRD of film B (fig. 5) shows considerable improvement in crystallinity with more c-axis oriented material. Three phases of the Tl material can be assigned from this XRD; 2212, 1212, and 2223. The XRD of film C (fig. 6) does not show a substantial improvement over the XRD of film B. However, the XRD of film C shows that slightly less material is in the 2223 phase.

For a direct measure of the superconducting properties of these films the DI responses are plotted in figures 7, 8, and 9. The DI response for film A shows no superconducting transition implying that this film can not support macroscopic supercurrents. A superconducting transition is observed in film B with the onset of supercurrents at 105 K and a transition width of 15 degrees. Film C shows the best superconducting transition with an onset at 107 K. This film also has the largest inductive response, implying that more material under the coil has gone into the superconducting state.

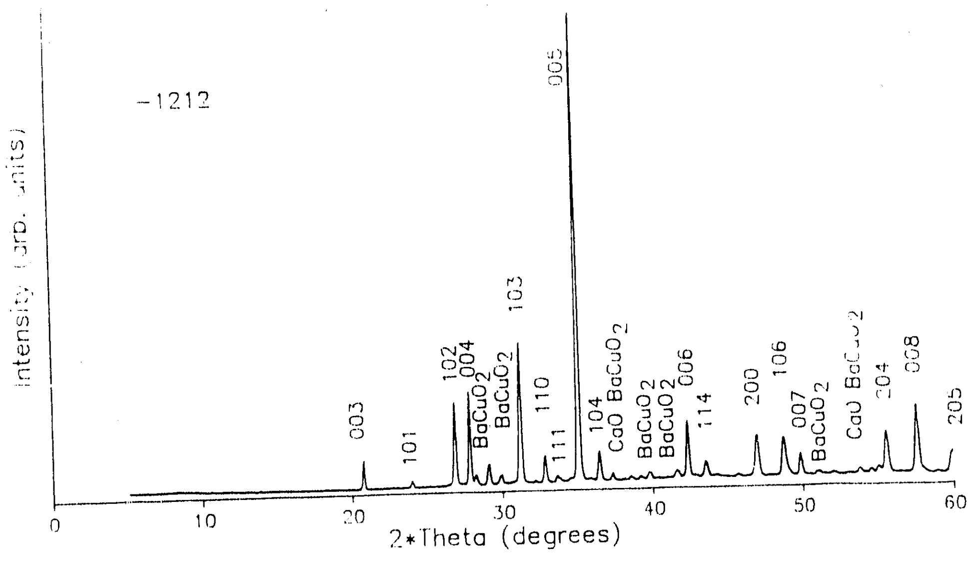

Figure 4. X-ray diffraction pattern of HTS film A.

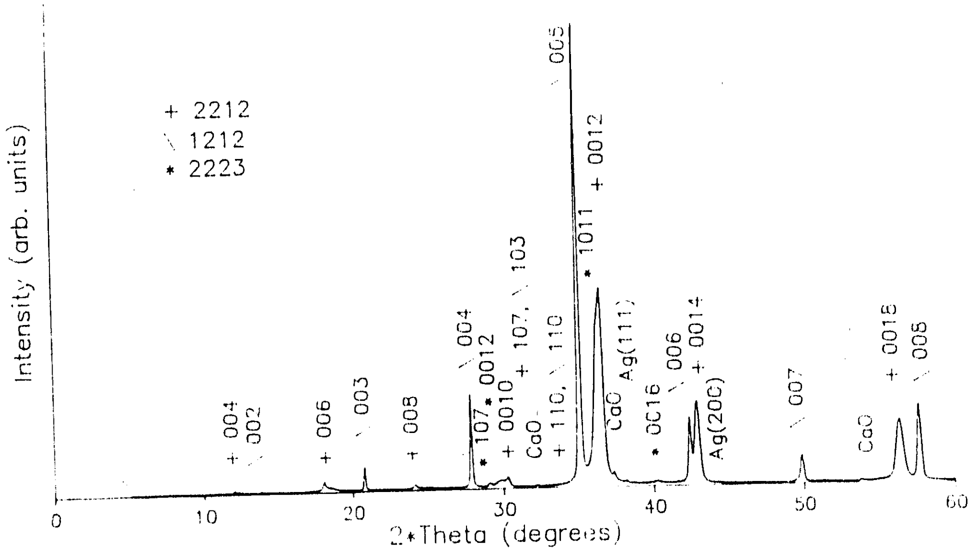

Figure 5. X-ray diffraction pattern of HTS film B.

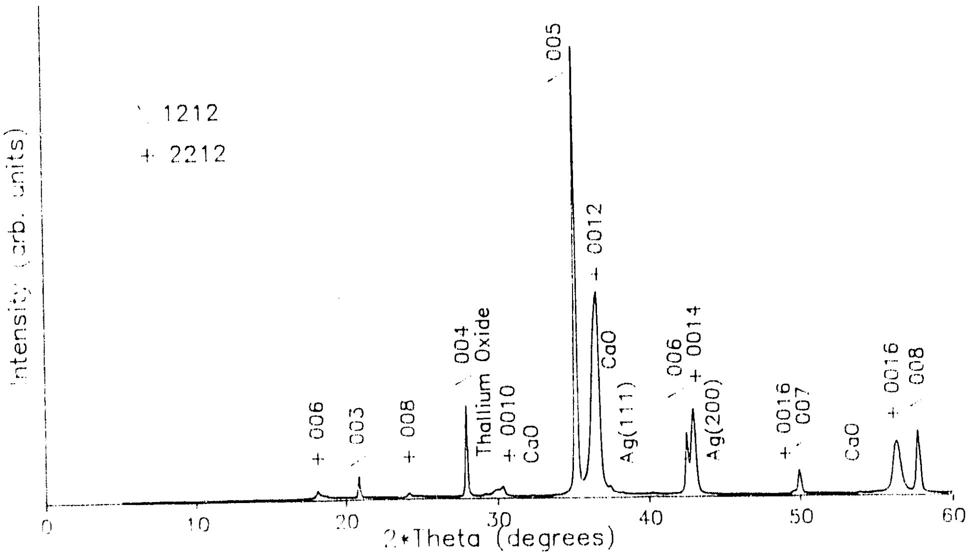

Figure 6. X-ray diffraction pattern of HTS film C.

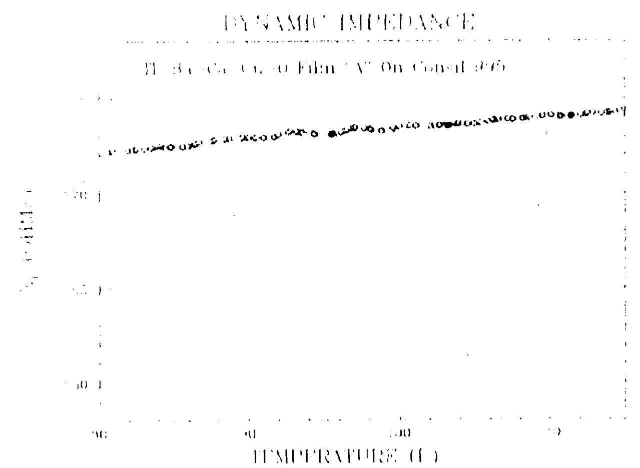

Figure 7. Dynamic impedance measurement of film A on a Consil substrate.

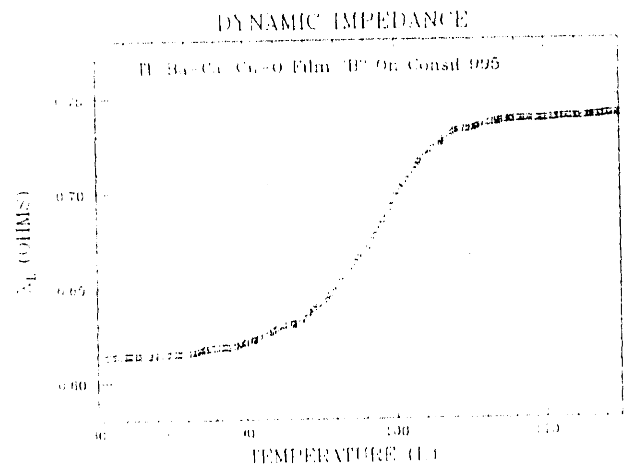

Figure 8. Dynamic impedance measurement of film B on a Consil substrate.

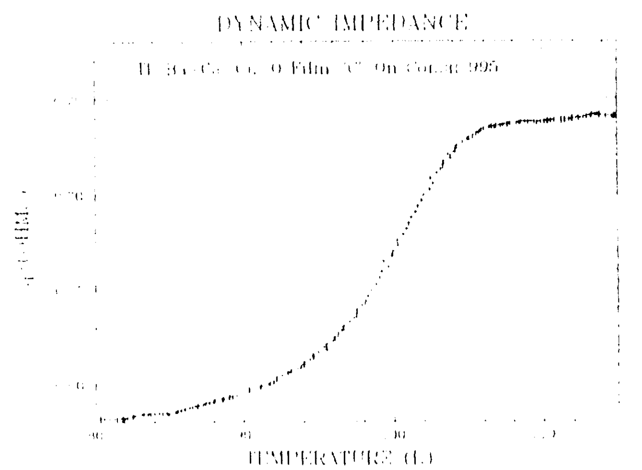

Figure 9. Dynamic impedance measurement of film C on a Consil substrate.

Conclusion

We have presented the results of different annealing procedures of Tl-Ba-Ca-Cu-O films on Consil 995. The superconducting responses were measured inductively with a single coil system. This system provides a rapid and reliable method of optimizing the superconducting properties of a material even on a metal substrate. The improvement in the superconducting responses as a function of annealing procedures correlates well with the improved morphology observed in the SEM photographs. Also, the XRD data shows increased c-axis material with the "C" annealing protocol.

References

1. D. W. Cooke and E. R. Gray, "Conference on High Temperature Superconductivity", Huntsville, AL, May 23-25, (1989).

2. P. Arendt, W. Bongianni, N. Elliott, and R. Muenchausen, "Science and Technology of Thin Film Superconductors", R. D. McConnell and S. A. Wolf eds., Plenum New York, NY (1989).

3. See, for example, H. L. Libby, "Introduction to Electromagnetic Nondestructive Test Methods", Robert E. Krieger Publishing Company, Malabar, Florida, 1971.

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