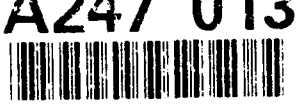

Final Report
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CHEMICAL VAPOR DEPOSITION OF $YBa_2Cu_3O_X$ THIN FILMS FOR WIRE APPLICATIONS

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Outline

LIST OF TABLES

LIST OF FIGURES

Executive Summary

The chemical vapor deposition of superconducting $YBa_2Cu_3O_X$ thin films onto continuous lengths of ceramic fiber tows has been investigated. This project couples two areas of current interest in the materials field -- continuous fiber coating and the deposition of one of the unique high temperature superconducting oxides. The $YBa_2Cu_3O_X$ material has zero resistance at temperatures above 77 K (the boiling point of nitrogen), and the thin film form has been shown to have high critical current densities ($>10^6$ A/cm^2). By coating ceramic fiber tows, a strong, flexible superconducting material in the form of wire can be developed and used for several applications, including coils for magnets and transmission lines.

Over 900 deposition runs were completed using a horizontal CVD furnace and over 150 continuous fiber coating experiments were completed. The deposition was performed on fiber tows, flat samples, and flexible tapes; the continuous substrates were moved and held stationary in the furnace. The films were characterized using optical and scanning electron microscopy, energy dispersive spectroscopy, x-ray diffraction, and critical current and resistance versus temperature measurements.

We have emphasized development of the process and equipment required for the chemical vapor deposition (CVD) of $YBa_2Cu_3O_x$ onto ceramic fiber substrates. Metal organic reagents, such as the tetramethylheptanedionates are fed into the MOCVD reactor as

powders where they sublimate/evaporate and subsequently react to permit deposition of the $YBa_2Cu_3O_x$ coating at a high rate. Both monofilament and fiber tows have been successfully coated. Emphasis has been on the use of alumina substrates in either single crystal or polycrystalline form. A continuous fiber coating furnace was designed, fabricated, installed and operated. Wires with critical temperatures as high as 82 K have been prepared. Critical current measurements have been hampered by difficulties encountered in making reliable contacts to the small diameter wires.

An MOCVD process for the preparation of silver films was developed using a reagent (perfluoro-1-methylpropenyl silver) synthesized at Georgia Tech. This process has been used to apply a protective layer to ceramic fibers (monofilaments and tows) to prevent interaction of the $YBa_2Cu_3O_x$ with the substrate fiber.

In collaboration with ORNL, equations were developed and computerized to permit calculation of fiber and coating stresses which occur as a result of mismatches in thermal expansion coefficient between the fiber and coating. The methodology is sufficiently flexible to permit anisotropy in both the fiber and coating thermal expansion. The program was extended to permit calculation of longitudinal, radial, and circumferential stresses (in the fiber and coating) as a result of bending the wire as required for the winding of magnets, motors and other devices.

The required process development for routine fabrication of superconducting wire using the MOCVD coating technique is not complete. Improved process control and higher critical currents

are needed. Because of high $YBa_2Cu_3O_x$ deposition rates the MOCVD process using powder feeding of reagents continues to appear attractive as a practical method for fabricating wire and other thick film supercondctor devices.

The following list of publications and presentations were a direct result of the DARPA Superconductivity funding.

Publications:

Shapiro, M. J., Lackey, W. J., Hanigofsky, J. A., Hill, D. N., Carter, W. B., Barefield, E. K., "Chemical Vapor Deposition of Silver Films for Superconducting Wire Applications," submitted to the J. of Less-Common Metals, July, 1991.

Lackey, W. J., Hanigofsky, J. A., Groves, M. T., Heaney, J. A., "Continuous Fiber Coating System," Ceram. Eng. Soc. Proc. 12(7-8), 1048-63, 1991.

Hsueh, C. H., Becher, P. F., and Lackey, W. J., "Thermal and Mechanical Induced Stresses in Superconducting Coatings on Fibers," J. Appl. Phys., 70(3), 1337-44, August, 1991.

Shapiro, M. J., More, K. L., Lackey, W. J., Hanigofsky, J. A., Hill, D. N., Carter, W. B., Barefield, E. K., Judson, E. A., O'Brien, D. F., Patrick, R., Chung, Y. S., and Moss, T. S., "Interaction of Chemically Vapor Deposited $YBa_2Cu_3O_x$ with Yttria-Stabilized Zirconia Substrates," J. Am. Ceram. Soc., 74(8), 2021-24, 1991.

Lackey, W. J., Hanigofsky, J. A., Shapiro, M. J., Carter, W. B., Hill, D. N., Barefield, E. K., Judson, E. A., O'Brien, D. F., Chung, Y. S., Moss, T. S., and More, K. L., "Preparation of Superconducting Wire by Deposition of $YBa_2Cu_3O_x$ Onto Fibers," Mat. Res. Soc. Symp. H Abstracts, p. 293, 1990.

Lackey, W. J., Carter, W. B., Hill, D. N., Barefield, E. K., Hanigofsky, J. A., Shapiro, M. J., Moss, T. S., Green, A. J., O'Brien, D. F., Jake, R. A., and Efferson, K. R., "Rapid Chemical Vapor Deposition of $YBa_2Cu_3O_x$ Coatings," Mat. Res. Soc. Symp. Proc. 169, 585-88, 1990.

Lackey, W. J., Carter, W. B., Hanigofsky, J. A., Hill, D. N., Barefield, E. K., Neumeier, G., O'Brien, D. F., Shapiro, M. J., Thompson, J. R., Green, A. J., Moss, T. S., Jake, R. A., and Efferson, K. R., "Rapid Chemical Vapor Deposition of Superconducting $YBa_2Cu_3O_x$," Applied Physics Letters, 56(12) 1175-77, March, 1990.

Lackey, W. J., Hanigofsky, J. A., Shapiro, M. J., Carter, W. B., Hill, D. N., Barefield, E. K., Judson E. A., O'Brien, D. F., Chung, Y. S., Moss, T. S., and More, K. L., "Preparation of Superconducting Wire by Deposition of $YBa_2Cu_3O_x$ Onto Fibers," Proc. 11th Int. Conf. on CVD, 195-210 ed. K. E. Spear and G. W. Cullen, The Electrochemical Soc., Pennington, NJ, 1990.

Lackey, W. J., Carter, W. B., Hill, D. N., Hanigofsky, J. A., Shapiro, M. J., Thompson, J. R., Green A. J., Moss, T. S., Efferson, K. R., and Jake, R. A., "Fabrication of Flexible High Temperature Ceramic Superconducting Wire by CVD," Mat. Res. Soc. Symp., November, 1988.

Theses:

"An Analysis of Preferred Orientation in $YBa_2Cu_3O_x$ Superconducting Films Deposited by CVD on Single and Polycrystalline Substrates", M.S. Thesis of Elizabeth Ann Judson, Georgia Institute of Technology, Atlanta, GA, June 1991.

"Chemical Vapor Deposition of Silver Films for Superconducting Wire Applications", Ph.D. Dissertation of Michael Shapiro, Georgia Institute of Technology, Atlanta, GA, March 1991.

"Annealing Study of $YBa_2Cu_3O_x$ Deposited by CVD on Ceramic Fiber Tows", M.S. Thesis of David O'Brien, Georgia Institute of Technology, Atlanta, GA, June 1991.

"Chemical Vapor Deposition of $YBa_2Cu_3O_x$ Thin Films for Wire Applications", Ph.D. Dissertation of John A. Hanigofsky, Georgia Institute of Technology, Atlanta, GA, in progress.

Presentations:

Lackey, W. J., Hanigofsky, J. A., Groves, M. T., and Heaney, J. A., "Continuous Fiber Coating System," presented at the American Ceramic Society Meeting on Coating and Composites, Cocoa Beach, FL, January, 1991.

Hanigofsky, J. A., Lackey, W. J., Shapiro, M. J., Hill, D. N., Carter, W. B., Barefield, E. K., Judson, E. A., O'Brien, D. F., Patrick, R. S., Chung, Y. S., Hardin, R. D., Beckloff, B. N., Emmerich, D. M., "Continuous Fiber Coating With Superconducting $YBa_2Cu_3O_x$ For Wire Applications," presented at the 93rd Annual Meeting of the American Ceramic Society, Cincinnati, Ohio, April, 1991.

O'Brien, D. F., Barefield, E. K., Carter, W. B., Hill, D. N., Lackey, W. J., and Hanigofsky, J. A., "Effects of Annealing at Reduced Oxygen Pressure on YBCO Films Deposited by CVD," presented at the 93rd Annual Meeting of the American Ceramic Society, Cincinnati, Ohio, April, 1991.

Judson, E. A., Hill, D. N., Cagle, J. R., Carter, W. B., Lackey, W. J., Hanigofsky, J. A., and Barefield, E. K., "Analysis of Preferred Orientation of $YBa_2Cu_3O_x$ Superconducting Films by CVD on Single and Polycrystalline Substrates," presented at the 93rd Annual Meeting of the American Ceramic Society, Cincinnati, Ohio, April 1991.

Lackey, W. J., Hanigofsky, J. A., Hunt, A., Carter, W. B., Barefield, E. K., and Hill, D. N., "Fabrication of $YBa_2Cu_3O_x$ Wire by Continuous MO-CVD Coating of Fiber," presented at the DARPA High Temperature Superconductor Workshop, Seattle, WA, September, 1991.

Lackey, W. J., Barefield, E. K., Hill, D. N., Carter, W. B., "Fabrication of Flexible High Temperature Ceramic Superconducting Wire by Rapid CVD," presented to Dr. Frank Patten and Dr. Mark Davis, Oak Ridge, TN, January, 1990.

Shapiro, M. J., Lackey, W. J., Hanigofsky, J. A., Carter, W. B., Barefield, E. K., Neumeier, G., O'Brien, D. F., Chung, Y. S., Green, A. J., Moss, T. S., More, K. L., "Substrate Interaction With Chemically Vapor Deposited $YBa_2Cu_3O_x$," presented at the 92nd Annual Meeting of the American Ceramic Society, Dallas, Texas, April 1990.

Hanigofsky, J. A., Lackey, W. J., Carter, W. B., Barefield, E. K., Neumeier, G., O'Brien, D. F., Shapiro, M. J., Chung, Y. S., Green, A. J., Moss, T. S., Jake, R. A., and Efferson, K. R., "Novel Processing of $YBa_2Cu_3O_x$ Films by Chemical Vapor Deposition," presented at the 92nd Annual Meeting of the American Ceramic Society, Dallas, Texas, April 1990.

Shapiro, M. J., Lackey, W. J., Hanigofsky, J. A., Hill, D. N., Carter, W. B., Barefield, E. K., O'Brien, D. F., Green, A. J., Moss, T. S., "Preparation of Superconducting Wire by MOCVD," presented at the Annual Meeting of the Southeastern Section of the American Ceramic Society, Clemson, SC, June, 1990.

Lackey, W. J., Hanigofsky, J. A., Shapiro, M. J., Carter, W. B., Hill, D. N., Barefield, E. K., Judson E. A., O'Brien, D. F., Chung, Y. S., Moss, T. S., and More, K. L., "Preparation of Superconducting Wire by Deposition of $YBa_2Cu_3O_x$ Onto Fibers," presented at the 11th International Conference on Chemical Vapor Deposition, Seattle, WA, October, 1990.

Lackey, W. J., Carter, W. B., Hill, D. N., Barefield, E. K., Hanigofsky, J. A., Shapiro, M. J., Judson, E. A., Chung, Y. S., Moss, T. S., O'Brien, D. F., Patrick, R. S., Polley, T. A., Hardin, R. D., Emmerich, D. M., Beckloff, B. N., Jake, R. A., Efferson, K. R., "Improved Method of Introducing Reagents for MOCVD of $YBa_2Cu_3O_x$," presented at the Second Annual DARPA High Temperature Superconductor Workshop, Boston, MA, October, 1990.

Lackey, W. J., Carter, W. B., Hill, D. N., Barefield, E. K., Hanigofsky, J. A., Shapiro, M. J., Judson, E. A., Chung, Y. S., Moss, T. S., O'Brien, D. F., Patrick, R. S., Polley, T. A., Hardin, R. D., Emmerich, D. M., Beckloff, B. N., Jake, R. A., Efferson, K. R., "Preparation of Superconducting Wire By MOCVD of $YBa_2Cu_3O_x$ Coatings Onto Fibers," presented at the Second Annual DARPA High Temperature Superconductor Workshop, Boston, MA, October, 1990.

Lackey, W. J., Hanigofsky, J. A., Shapiro, M. J., Carter, W. B., Hill, D. N., Barefield, E. K., Judson, E. A., O'Brien, D. F., Chung, Y. S., Moss, T. S., and More, K. L., "Preparation of Superconducting Wire by Deposition of $YBa_2Cu_3O_x$ Onto Fibers," presented in the Materials Issues in Applications of Ceramic Superconductors, the Materials Research Society, Boston, MA, November, 1990.

Hanigofsky, J. A., Lackey, W. J., Shapiro, M. J., Hill, D. N., Carter, W. B., Barefield, E. K., "Fabrication of Flexible Superconducting Wire By MOCVD," presented at the Association of Ceramic Industries, Atlanta, GA, November, 1990.

Shapiro, M. J., Lackey, W. J., Hanigofsky, J. A., Hill, D. N., Carter, W. B., Barefield, E. K., Green, A. J., Moss, T. S., "Superconducting Coatings and Wires by CVD," presented at the Association of Ceramic Industries, Atlanta, GA, January, 1989.

O'Brien, D. F., "Resistance and Magnetic Susceptibility Measurements of $YBa_2Cu_3O_x$ Coatings Deposited by CVD," presented at the Summer Meeting of the Southeastern Section of the American Ceramic Society, Boone, North Carolina, July, 1989.

Lackey, W. J., Barefield, E. K., Carter, W. B., Hanigofsky, J. A., Green, A. J., Hill, D. N., Moss, T. S., Neumeier, G., O'Brien, D. F., Shapiro, M. J., Thompson, J. R., Jake, R. A., and Efferson, K. R., "Fabrication of Flexible High Temperature Ceramic Superconducting Wire By Rapid CVD and the Manufacture of a Demonstration Magnet," presented at the DARPA High Temperature Superconductivity Workshop, Santa Barbara, CA, September, 1989.

Lackey, W. J., Carter, W. B., Hill, D. N., Barefield, E. K., Hanigofsky, J. A., Shapiro, M. J., Moss, T. S., Green, A. J., and O'Brien, D. F., "Rapid Chemical Vapor Deposition of $YBa_2Cu_3O_x$ Coatings," presented at the 1st Annual DARPA High Temperature Superconductors Workshop," Santa Barbara, CA, September, 1989.

Lackey, W. J., Carter, W. B., Hill, D. N., Barefield, E. K., Hanigofsky, J. A., Shapiro, M. J., Moss, T. S., Green, A. J., O'Brien, D. F., Jake, R. A., and Efferson, K. R., "Rapid Chemical Vapor Deposition of $YBa_2Cu_3O_x$ Coatings," presented in the Fundamental Properties and Novel Materials Processing Session, the Materials Research Society, Boston, MA, November, 1989.

Lackey, W. J., Carter, W. B., Hill, D. N., Hanigofsky, J. A., Shapiro, M. J., Thompson, J. R., Green A. J., Moss, T. S., Efferson, K. R., and Jake, R. A., "Fabrication of Flexible High Temperature Ceramic Superconducting Wire by CVD," presented in the Preparation and Materials Properties of High Temperature Superconductors Session, the Materials Research Society, Boston, MA, November, 1988.

INTRODUCTION

The discovery of high temperature oxide superconductors by Bednorz and Muller in 1985 has led to extensive research efforts to study and develop these materials for several applications. Potential applications include magnetic coils, high speed switches, antennae, RF high Q microwave cavities, and power transmission lines. The preferred oxide superconductors have high transition temperatures (above 77 K) which will allow for liquid nitrogen cooling to obtain the desired electrical properties. Extensive materials development is required, however, before any application can be realized.

The literature review consists of background on the purpose of fiber coating and the superconducting $YBa_2Cu_3O_x$ material. Next, a description of previous CVD work with this system is discussed, including work performed at Georgia Tech. The goal of the proposed research is to match the fiber coating technology with the $YBa_2Cu_3O_x$ material system, resulting in a successful deposition of the material and a process model which describes the continuous deposition onto fiber tows (bundles of filaments). The results for the 3 year project, followed by a discussion and conclusions summarize the effort completed at Georgia Tech.

LITERATURE REVIEW

A. Fiber Coating Purpose and Applications

To take advantage of the superconducting properties of $YBa_2Cu_3O_x$, specifically the high critical currents and high transition temperatures realized using thin film deposition techniques, a useful form of the material must be developed. One potential form involves coating flexible ceramic fiber tows with a thin film of $YBa_2Cu_3O_x$ surrounding each individual fiber. This will result in a large volume percent of the superconducting material being supported by the fiber. (See Figure 2-1) The ceramic fiber has two purposes: first, it provides a high temperature, small diameter substrate onto which the film deposits; second, it gives the final product mechanical strength. The mechanical strength is necessary if the coated filaments are to be wound into any useful shape, including magnet coils[1,2].

Other applications which have been cited for superconducting thin films and wire include high power transmission lines[3] and Josephson junctions[4]. The use of liquid nitrogen for cooling the material below the superconducting transition temperature represents a substantial savings ($1.41/ℓ) over presently used Nb_3Ge superconducting materials which are cooled using liquid helium ($4.00/ℓ)[5]. Liquid nitrogen also requires minimal refrigeration cost and has little boil-off loss.

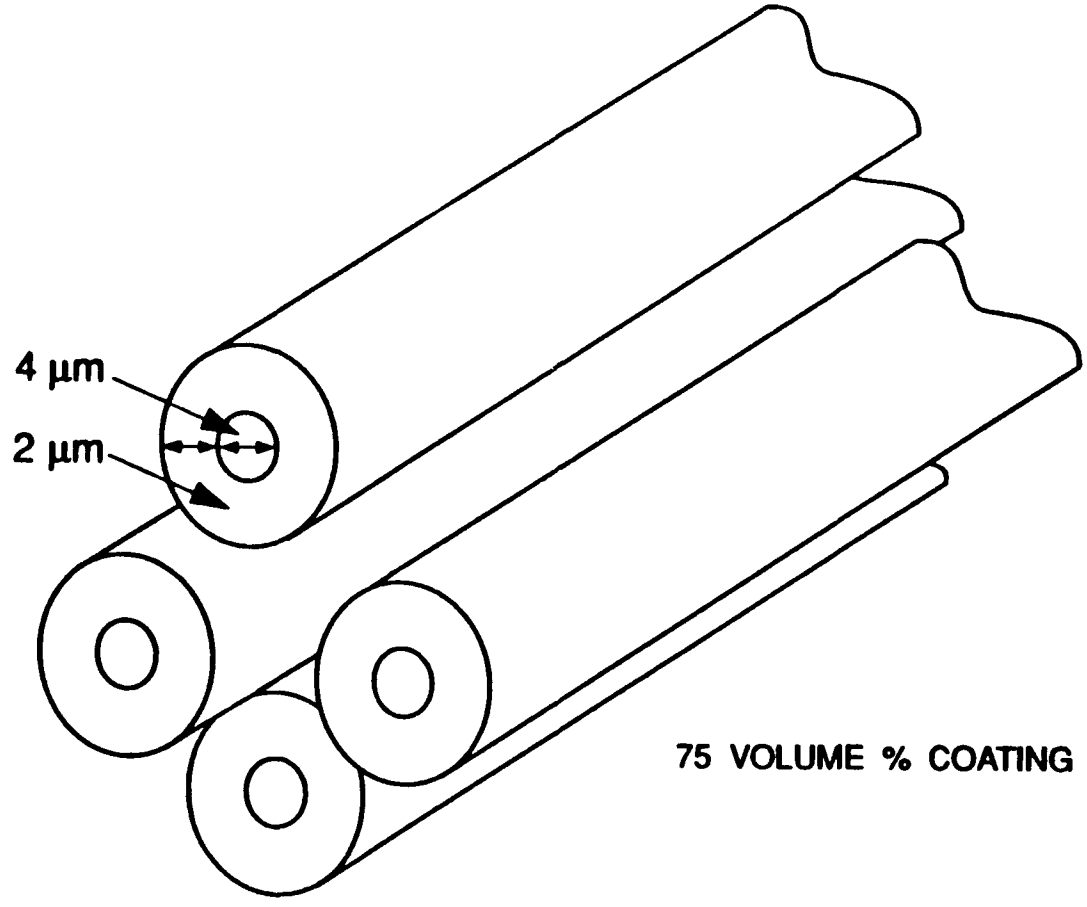


Figure 2-1. Schematic describing the objective of $YBa_2Cu_3O_x$ coating on fiber tows for wire applications.

B. Superconducting $YBa_2Cu_3O_x$

1. Structure

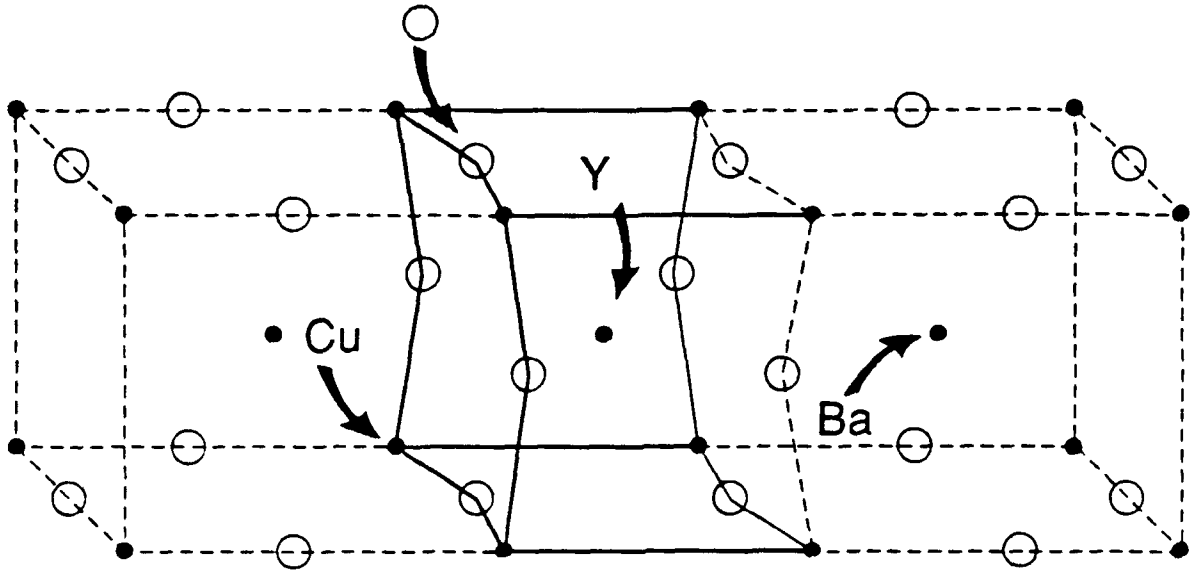
One of the earliest identified new high temperature superconductors was $YBa_2Cu_3O_x$[4]. This material has an open, layered structure which has been described as a distorted, oxygen deficient perovskite structure. The lattice constants calculated for the material by x-ray diffraction are a=3.82 Å, b=3.89 Å, and c=11.69 Å[7]. A formally filled perovskite structure would have the chemical formula $YBa_2Cu_3O_9$, with the oxygen octahedrally coordinated. The $YBa_2Cu_3O_x$ compound (referred to as 123) has a triple unit cell along the c-axis with two inequivalent copper planes sandwiched between the Y-Ba and Ba-Ba planes. (See Figure 2-2) The oxygen content of the superconducting phase ranges between 6.3 and 7.0 for x in the formula $YBa_2Cu_3O_x$. This corresponds to the orthorhombic crystal system with the space group Pmmm[8]. If the compound is oxygen poor (x<6.3), the phase is tetragonal, with the space group P4/mmm[8].

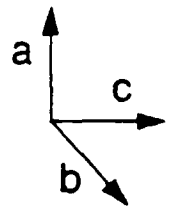
2. $YBa_2Cu_3O_x$ Phase Relations

A 950°C section of the ternary phase diagram for the BaO-$YO_{1.5}$-CuO system is shown in Figure 2-3[9]. The diagram predicts the $YBa_2Cu_3O_x$ compound to exist as an invariant point, surrounded by three ternary compatibility regions consisting of:

1. $YBa_2Cu_3O_x$ + $BaCuO_2$ + CuO
2. $YBa_2Cu_3O_x$ + Y_2BaCuO_5 + $BaCuO_2$ and
3. $YBa_2Cu_3O_x$ + Y_2BaCuO_5 + CuO.

Clark et al.[9] presented microstructural differences caused by slight adjustments of the cation ratios. The samples were sintered





a = 3.8203 Å a/b = 0.982

b = 3.8906 Å c/b = 3.005

c = 11.6915 Å V = 173.77 Å^3

x-ray density = 6.33 g/cm^3

Figure 2-2. Crystal structure and lattice parameter data for the superconducting $YBa_2Cu_3O_x$ compound.

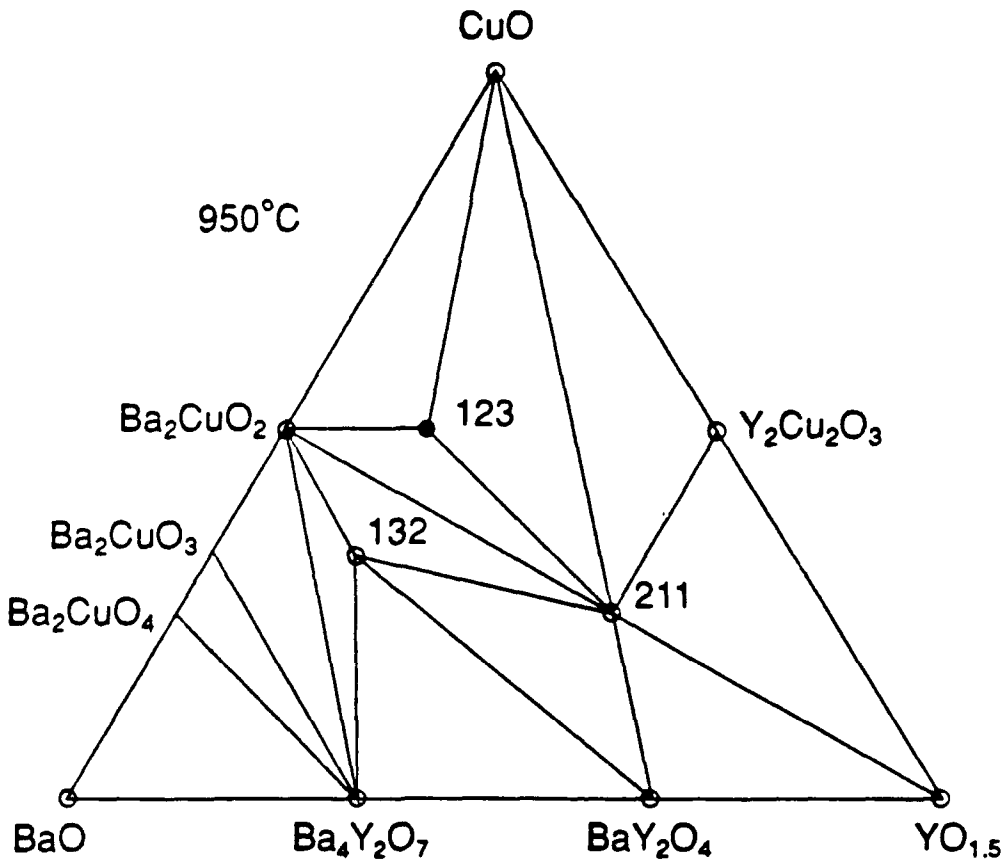


Figure 2-3. Ternary phase diagram (950°C section) for the CuO-BaO-$YO_{1.5}$ system.

in flowing oxygen at 950°C. Dense materials were observed in the ternary regions containing CuO (regions 1 and 3), due to some melting during sintering. Melting was not observed in region 2 ($YBa_2Cu_3O_x$ + Y_2BaCuO_5 + $BaCuO_2$), where a very fine grained morphology was obtained.

Annealing studies in argon, air, and oxygen were used to determine eutectic and peritectic reactions in the BaO-$YO_{1.5}$-CuO system[10]. The major conclusions from this study indicated that the material melted at lower temperatures (90 - 100°C lower) in argon as compared to oxygen or air. Also, the eutectic and peritectic temperatures had a strong positive dependence on the oxygen partial pressure.

A final phase study was presented by Wong-Ng et al.[11], which involved annealing several samples in air followed by rapid quenching. The phase transition from orthorhombic to tetragonal which occurred between 708 and 720°C was characterized as a second order, order-disorder type. This result was attributed to the strong connection between the Pmmm and the P4/mmm space groups of the two crystal systems. Several mechanisms for the removal and ordering of oxygen were discussed to explain the observed transition.

3. Physical and Mechanical Properties

Orthorhombic $YBa_2Cu_3O_x$ is a black, opaque material. In bulk form it is relatively hard, but extremely brittle. Thin films of $YBa_2Cu_3O_x$ are a black/grey color, and are generally very adherent to the substrate material. The thin films are very soft, however,

and can be easily removed by scraping with a razor blade. The anisotropic structure of the $YBa_2Cu_3O_x$ material dictates the usefulness of its promising electrical properties.

The superconducting transition temperature for the $YBa_2Cu_3O_x$ material is 95 K. Randomly oriented bulk samples have extremely low critical current densities (approximately 1000 A/cm^2), which is the amount of current that can pass through the material without destroying the superconducting properties (i.e., maintain zero resistance). The critical current criteria established by the Defense Advanced Research Projects Agency (DARPA) corresponds to the critical current attained at a field of 1 μV/cm. The critical current densities for thin films have been reported as high as 10^6 A/cm^2, even within a magnetic field[12]. These high values are attributed to the c-axis orientation obtained during thin film deposition.

A study by Dimos et al.[13] of orientation dependence of grain boundary critical currents using twinned single crystals of $YBa_2Cu_3O_x$ determined that even when the c-axis of individual grains are nearly parallel, with a textured microstructure, the critical currents achieved are only 10^4 A/cm^2. The misorientations in the basal planes between adjacent grains limits the property as compared with epitaxial thin films. Further research has shown that the electrical conductivity in the c direction is 100 times the conductivity in the a and b directions at 200 K, which contributes to the higher critical currents in the c-axis oriented thin film materials[7].

The mechanical properties of the $YBa_2Cu_3O_x$ material are also

extremely important for any potential application. This ceramic material is extremely brittle, so large bulk pieces cannot be easily machined or extruded into any form for uses requiring flexibility, such as wire or cable.

Mechanical properties were presented by Alford et al.[14] for pressed powder samples densified by viscous sintering. The final 0.2 to 4 mm rods ranged from 80 to 98 % dense. Table I summarizes the results, and includes values for Al_2O_3[15] and Ag[16] for comparison. Polycrystalline Al_2O_3 is a typical substrate material used in fiber tow work, and Ag is a proposed interlayer material between the substrate and $YBa_2Cu_3O_x$ coating.

Table I. Physical and mechanical properties of $YBa_2Cu_3O_x$, Al_2O_3, and Ag.

Property	$YBa_2Cu_3O_x$	Al_2O_3	Ag
Theoretical Density (g/cm^3)	6.8	3.97	10.5
Young's Modulus (GPa)	141.8	380	72.4
Fracture Toughness ($MPa/m^{1/2}$)	1.07	2.7-4.2	
Thermal Expansion ($x10^{-6}$ $^{\circ}C^{-1}$)	11.5-14.6	7.2-8.6	19
Thermal Conductivity (W/m*K)	2.0	27.2	427

A second study on the thermal expansion coefficients by Hashimoto et al.[18] compared the mean thermal expansion coefficient for $YBa_2Cu_3O_x$ and the expansion coefficients for various substrate materials. Table II summarizes the results, which indicate the superconducting material has a larger thermal expansion ($16.9 \times 10^{-6}\,°C^{-1}$) than any common substrate over the same temperature range (500-900°C). During cooling from 900°C, the film will be under tension, possibly causing cracks in the coating. Again, the anisotropy was discussed, with the thermal expansion coefficient in the c direction being greater than in the a or b directions.

Table II. Mean thermal expansion coefficients for $YBa_2Cu_3O_x$ and several substrate materials.*

Material	Mean Thermal Expansion Coefficient ($\times 10^{-6}\,°C^{-1}$)
$YBa_2Cu_3O_x$	16.9
$SrTiO_3$	11.1
YSZ	10.3
MgO	13.0
Al_2O_3	7.0

* All substrate materials are single crystal except for Al_2O_3

C. Deposition of $YBa_2Cu_3O_x$ by CVD

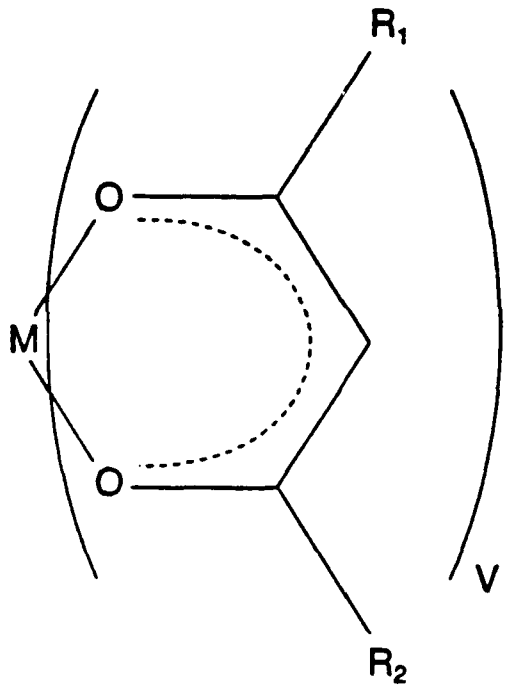
Chemical vapor deposition (CVD) is a common processing technique where chemical compounds are introduced into a furnace where they react or decompose in the vapor phase on or near a heated substrate resulting in the deposition of a solid coating. The chemical compounds, or reagent precursors, can be introduced as 1. gases, by 2. bubbling a carrier gas through a liquid, or by

3. heating a solid to vaporize the material and using a carrier gas to sweep the vapor into the furnace. Since the initial discovery of superconducting oxides, CVD has been used to deposit the $YBa_2Cu_3O_x$ compound[18-57], the bismuth based superconducting compounds[58-62], and the thallium based superconducting compounds[63-66].

1. Chemistry

In the chemical vapor deposition of the unique high temperature $YBa_2Cu_3O_x$ oxide superconductor, volatile compounds containing Y, Ba, and Cu are required. Unfortunately, relatively few volatile compounds exist for these metals; the most promising materials are the organometallic compounds.

Figure 2-4 represents a generic structure of a metalorganic compound in the β-diketonate family. The R positions can be substituted with a large number of organic groups, which distinguish the compound name and formula (See Figure 2-4). For example, $Y(ACAC)_3$ is a yttrium based metalorganic compound with three acetylacetonate ligands attached to the structure. The derivatives of the β-diketonate family, specifically the tetramethylheptanedionates (TMHD) compounds have been shown to have reasonable volatility for the Y and Cu complexes[24]. The barium TMHD compound is also commonly used, however, several researchers[23,33] have expressed concerns with the chemical stability of the material. The compound thermally degrades with time, resulting in changing volatility, and therefore a varying concentration is introduced by vaporization into the CVD furnace[23]. The fluorinated compounds (FOD's in Figure 2-4) have been used to provide better



NAME	R_1	R_2
TMHD	$-C(CH_3)_3$	$-C(CH_3)_3$
TOD	$-C(CH_3)_3$	$-CH_2CH(CH_3)_2$
FOD	$-C(CH_3)_3$	$-(CF_2)_2CF_3$
HFA	$-CF_3$	$-CF_3$
ACAC	$-CH_3$	$-CH_3$

Figure 2-4. Common organometallic compounds and the generic β-diketonate structure for materials used in $YBa_2Cu_3O_x$.

stability and higher vapor pressure, however, higher temperatures and the use of water vapor are required to eliminate fluorine from the film[33].

A second problem with the organometallic compounds used in CVD of $YBa_2Cu_3O_x$ are the extremely low vapor pressures even at temperatures close to the material decomposition temperature. Figure 2-5 presents a plot of log vapor pressure (torr) versus inverse temperature (K^{-1}) for compounds typically used in CVD vaporizers, and the organometallics used for $YBa_2Cu_3O_x$ deposition. As shown, the organometallics have vapor pressures below 0.1 torr, resulting in an extremely slow deposition rate when using the conventional vaporizer approach.

2. Conventional CVD of $YBa_2Cu_3O_x$

The first reported deposition of $YBa_2Cu_3O_x$ using CVD was completed independently by Barry et al.[18] and Yamane et al.[12] in 1988. Conventional CVD of the material was completed by separately heating three vaporizers with the Y, Ba, and Cu organometallic compounds, flowing a carrier gas (typically argon) at separate flow rates through each of the vaporizers to sweep the reactant vapor into the furnace, where the materials were mixed with oxygen and reacted to deposit the $YBa_2Cu_3O_x$ compound onto a substrate. A typical CVD furnace and conventional vaporizer arrangement is shown in Figure 2-6; both hot wall and cold wall furnaces have been used to deposit the $YBa_2Cu_3O_x$ material. Since the formation of the $YBa_2Cu_3O_x$ compound requires a high temperature treatment, two general types of conventional processing have been attempted. Films have

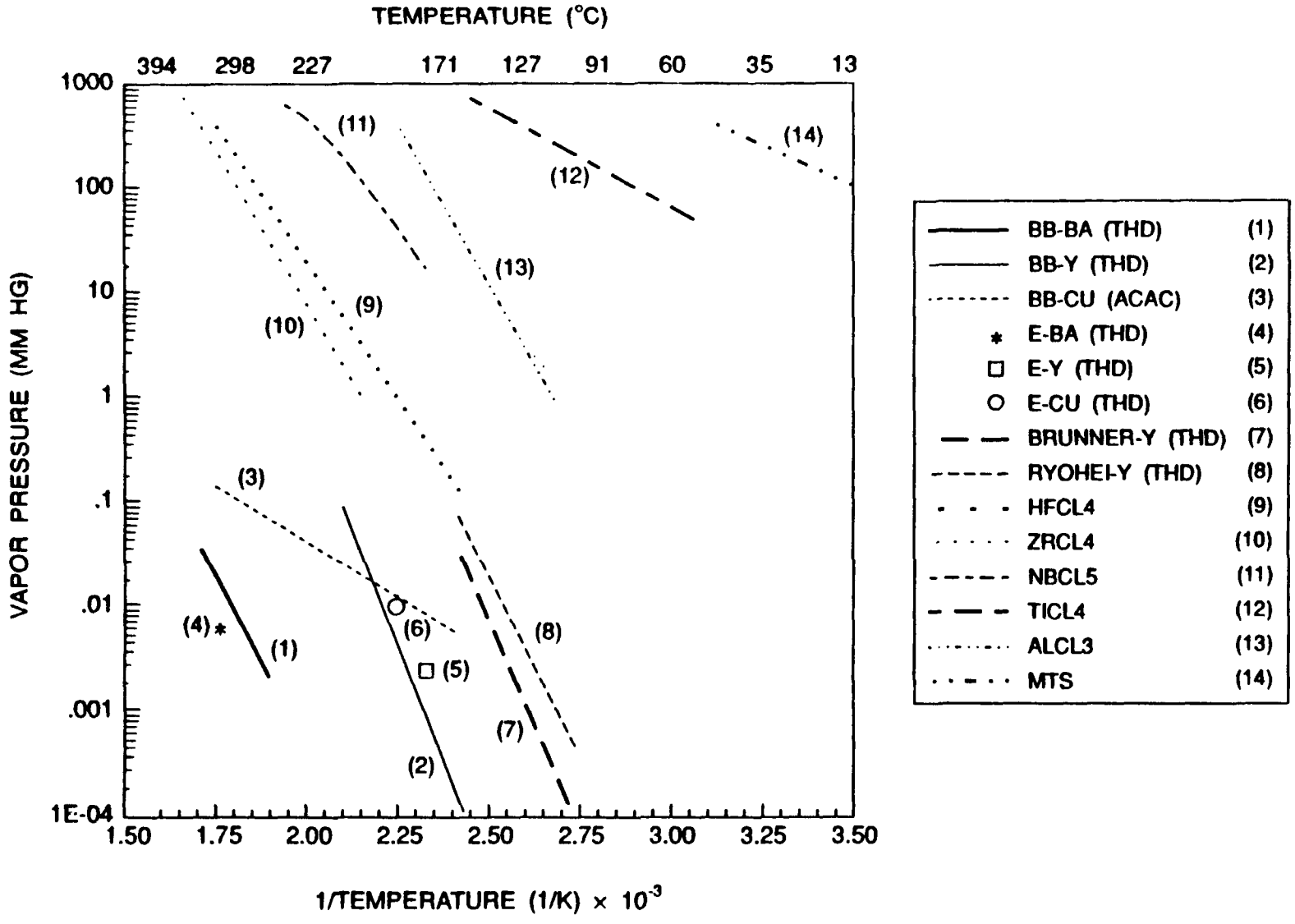


Figure 2-5. Vapor pressure versus 1/T for several commonly used CVD reagents. Lines 1-8 are for organometallics used in the deposition of $YBa_2Cu_3O_x$. Lines 9-14 are for compounds used for other common CVD systems.

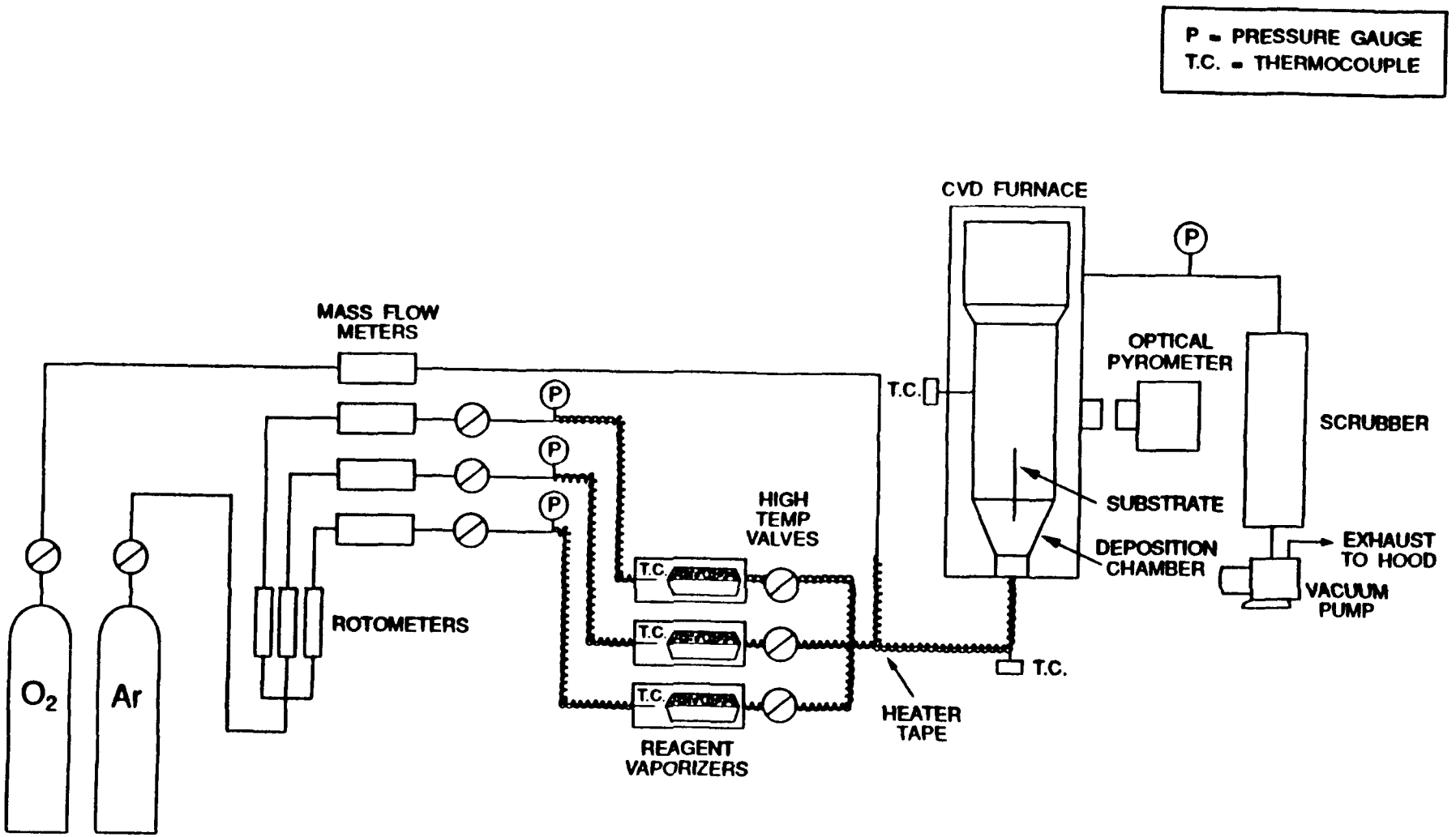


Figure 2-6. Schematic of a typical conventional vaporizer setup for CVD of $YBa_2Cu_3O_x$.

been deposited at low temperatures, followed by a second post-deposition annealing treatment at high temperatures (850-950°C) in oxygen. The second technique is to deposit and anneal the film in situ using a high temperature deposition process followed by a slow furnace cool in oxygen.

A summary of some of the earlier published work using the conventional three vaporizer approach is presented in Table III. Key points of this table include the low deposition rate for each effort (typically below 1 μm/h), and the wide range of critical temperatures reported on the variety of substrate materials. Strontium titanate single crystal substrates had the highest critical temperatures (92 K). Several researchers attribute this to the epitaxial growth observed when using this substrate[20,24]. Most of the groups used the TMHD organometallics reagents, however, some deposition work using metal chlorides can be noted. The work at Tohoku University by Yamane et al.[12,19,25,27,31,36] has been the source for the reported critical currents over 10^6 A/cm^2. This group presented their deposition conditions as:

	Vaporization Temperature(°C)	Carrier Gas Flowrate (sccm)
$Y(TMHD)_3$	132	150
$Ba(TMHD)_2$	250	150
$Cu(TMHD)_2$	130	150
Total Pressure	10 torr	
Deposition Temperature	850°C	
Deposition Time	1 hour	
Oxygen Flowrate	250 sccm	

A 1 μm thick film was deposited on $SrTiO_3$, and the film was cooled from 850°C to room temperature in 1 atmosphere of oxygen

Table III. Summary of prior CVD of $YBa_2Cu_3O_x$.

REF. #	GROUP	REAGENTS[a]	SUBSTRATE	TEMP(°C)	RATE (μm/hr)	T_c(ONSET)(K)	TC(R=0)(K)	ANNEAL CONDITIONS
1	NRL	Y,Ba-tmhd Cu-acac	MgO	400	0.48	80	20	890-920°C 20 min, 920°C- 10 min, 0.83°C / min in O_2
2	TOHOKU	tmhd	YSZ	900	0.8	43 83 90 89	– 33 62 80	10 torr O_2 400°C, O_2, 8 hr 30°C / min, 1 atm O_2 3°C / min, 1 atm O_2
3	BATTELLE	Cu-acac Y-tmhd Ba-hfa	YSZ	900				–
4	OKI	tmhd	MgO	600	2.4 - 6	76	60	950°C 30 min, 2°C / min in O_2
5	WESTING-HOUSE	Y,Ba-hfa Cu-acac	Sapphire $SrTiO_3$ Al_2O_3		5	90 90	65 70 65	H_2O/Ar 835°C 30 min, 900°C- 10 min Ar, 8.3°C / min to- 400°C, 400°C 30 min, - 8.3°C / min to 200°C
6	NISSAN	Y-tmhd Ba,Cu-hfa	$SrTiO_3$	600	0.33	83	65	850°C 2 hr, 2°C / min to - 500°C, 500°C 2 hr, - 2°C / min to RT (all in air)
7	NORTH-WESTERN	Y-tmhd Cu-acac Ba-fod	MgO	700	0.6 - 1.8	90	66.2	600°C 10 hr, 900°C 1.5 hr - 960°C 10 min in O_2
8	TOHOKU	tmhd	$SrTiO_3$	900	2	88	84	15°C / min, 1 atm O_2
9	GEORGIA TECH	tmhd	YSZ Sapphire	650	10	93 --	84 10	950°C 30 min, 4°C / min 895°C 15 min, 4°C / min in O_2
10	TOHOKU	tmhd	$SrTiO_3$	850	1	-- 89 92	91.5	90°C / min, 1 atm O_2
11	TOKYO IT/ MUSASHI	Cu-acac Y,Ba-hfa	Sapphire Si	500 700 500	0.3 - 0.9			
12	OKI	tmhd	$SrTiO_3$	800	0.9	88 75	83 60	100°C / min in atm O_2 quenched to RT in 5s
13	UPPSALA	YCl_3 BaI_2 CuCl	CSZ CSZ	870-910	3	80 80	40 70	– 475°C 48 hrs in O_2
14	TOHOKU	tmhd	YSZ	800	0.8 - 1	60 80 83	10 30 43	10 torr O_2 450°C, 1 atm O_2, 1 hr 1 atm O_2
15	ASEA BROWN BAVERIA	Cu-acac Y,Ba-tmhd	$SrTiO_3$ YSZ	900 950	0.6	94 94	88 86	20 min cool 1 atm O_2
16	STANFORD/ SRI/COLO	tmhd	$SrTiO_3$	800 900	0.1 - 1	90 87 70	70 77 64	550°C 2 hr
17	TOHOKU	tmhd	YSZ $SrTiO_3$	900 850 800	1 - 4	40 90	– 87 84 93 91	15°C / min, 10 torr O_2 15°C / min, 1 atm O_2
18	OKLAHOMA	tmhd	YSZ(BaF_2) YSZ	780	--	90 90	80 50	3.3°C / min Ar/O_2 to 400°C, - 2.5°C / min to 100°C
19	TOHOKU	tmhd	YSZ Al_2O_3 Sapphire	900	1 - 2			20°C / min, 1 atm O_2 - to 300°C, quench

[a] tmhd = tetramethylheptanedionate hfa = hexafluoroacetylacetonate
acac = acetylacetonate fod = heptafluorodimethyloctanedionate

which took 20 minutes. One of the films was reported to have a critical current J_c of $2x10^6$ A/cm^2 at 0 Tesla; the value decreased to $6.5x10^4$ A/cm^2 in a magnetic field of 27 Tesla. These results, along with values reported by Matsuno et al.[41], remain the most promising electrical data for $YBa_2Cu_3O_x$ thin films deposited by CVD.

The most comprehensive processing information for conventional CVD of $YBa_2Cu_3O_x$ was presented by Schmaderer and Wahl[32,57], which included data for log molar evaporation rates (moles/h) versus inverse evaporation temperature (K^{-1}) for Y and Ba TMHD and Cu(ACAC). Typical molar evaporation rates were $2-4x10^{-4}$ moles/h. A calculation based on this evaporation rate ($3x10^{-4}$ moles/h), assuming that Cu, which requires three moles delivered for every one mole of $YBa_2Cu_3O_x$ deposited, limits the process, and also assuming 100% deposition efficiency for 1 hour on a 1 cm^2 planar substrate, resulted in a hypothetical coating thickness of 98 μm. (See Appendix A) The major conclusions from this work indicated that the low observed deposition rate for the process is probably due to the small reactant concentrations in the gas phase limiting the diffusional transport across the stagnant boundary layer to the surface. Both TiO_2 "smoke" experiments and computer modeling were used to justify the conclusions. A plot of log deposition rate versus temperature showed a plateau at temperatures above 600°C, which is the region where mass transfer limits the process. At temperatures below 600°C, an activation energy of 73 kJ/mole was determined for this reaction limited region. Additional mass transport studies were presented by Erbil et al.[53], which concluded that the mass transport rates are insensitive to the carrier gas

flowrates for the Y and Ba TMHD compounds. The $Cu(TMHD)_2$ mass transport was reported to increase as the carrier gas flowrate was increased.

One problem which was mentioned and studied extensively by several researchers was the stability of the reagents, specifically the Ba reagent. Thermogravimetric analysis was commonly used to characterize the reagents and monitor the decomposition of the material[22]. To help stabilize the barium organometallic precursor, Dickinson et al.[33] added TMHD vapor to the barium vaporizer. The vapor pressure remained constant for nearly 2 hours. Other researchers used the fluorinated barium β-diketonate compound[24,35,38]. They found the presence of a BaF_2 layer at the interface between the substrate and $YBa_2Cu_3O_x$ coating, which is currently being investigated as an interlayer material. Finally, Matsunu et al.[41] used a mixture of TMHD vapor with argon as the carrier gas for the Y, Ba, and Cu DPM reagent precursors. The films produced were shown to have high critical currents as mentioned previously.

Two studies, by Ohnishi et al.[53] and Chew et al.[67] presented processing versus film property information for $YBa_2Cu_3O_x$ deposition. Ohnishi showed relationships between critical temperatures and composition of the film, with the highest T_c reported being 85 K on MgO substrates deposited at 750°C. Film composition was determined by ICP mass spectroscopy. Film compositions varying from $1/2<Y/Ba<2/2$ and $3/2<Cu/Ba<17/2$ were shown to be superconducting with critical temperatures varying from 25 to 85 K. Films which were slightly rich in copper were shown to have the highest T_c's,

and were favored over films with stoichiometry closer to Y:Ba:Cu = 1:2:3.

Chew et al. deposited films by evaporation on MgO and varied the copper content and the Y/Ba ratio in the film. Analysis was performed by both Rutherford Backscattering Spectroscopy and EDS. Strong correlations between surface morphology, composition, the (007) x-ray diffraction peak width, and the critical current were presented. As the copper content was increased at a fixed Y/Ba ratio, very little effect on the critical temperature was noted. The critical current and (007) peak width, however, were observed to decrease rapidly whenever the copper was altered from the optimal 1:2:3 cation ratio. The preferred morphology was determined to be smooth, with only a slight amount of pitting.

In the previous CVD work, the thin films were mainly characterized by x-ray diffraction, and resistance versus temperature curves. Most of the researchers report extensive c-axis orientation with the (00ℓ) reflections dominating the diffraction patterns. Epitaxial growth was claimed by Oda et al.[28], who deposited films on sapphire. Studies which completed the deposition at low temperatures (500-600°C) presented critical temperature measurements before and after annealing to 900°C. A high temperature anneal in oxygen or air followed by a slow cooling in oxygen was determined to be necessary to induce the tetragonal to orthorhombic phase transition at 720°C. This was also verified using x-ray diffraction and monitoring the relative intensities of the (101) and (013) unoriented $YBa_2Cu_3O_x$ peaks. Also, a decrease in c-axis orientation was observed for samples annealed at lower

temperatures. As deposition temperature was lowered from 900 to 700°C, an increase in the a- and b-axis orientation was observed.

The micrographs presented in previous CVD articles showed an extremely smooth and platey appearance for films deposited at high temperature (900°C). Films produced at lower temperatures appeared nodular. Hirai et al.[12] presented micrographs for the high critical current samples which appeared to be two phase. A $YBa_2Cu_3O_x$ matrix surrounded uniformly shaped and ordered CuO precipitates. Fracture surfaces of the films showed a very dense structure, with needle-like projections attributed to a- or b-axis oriented grains. Transmission electron microscopy of the material showed the c-axis oriented material surrounding various impurity phases, including Y_2BaCuO_5[21].

Another concern in the deposition of $YBa_2Cu_3O_x$ involved the post-deposition annealing of the films. Studies by Magro-Campero and Turner[68] and Feenstra et al.[69] have recently been published which emphasize low temperature, low oxygen partial pressure annealing treatments. Magro-Campero and Turner presented critical temperature measurements for films processed at ambient temperatures by co-evaporation followed by annealing in 29 Pa of oxygen at several temperatures. An optimal anneal temperature of 750°C resulted in a higher T_c on $LaAlO_3$ substrates of 89 K. Feenstra et al. also deposited films by co-evaporation and completed annealing studies using between 1 and 8.9×10^{-5} atm (10^5 and 9.0 Pa) of oxygen at 740-835°C. Their findings showed an enhancement in the electrical properties, with an increase in film epitaxy at low partial pressures of oxygen.

3. Georgia Tech Process

At the Georgia Tech Research Institute, following numerous failures with the conventional CVD approach to deposit $YBa_2Cu_3O_x$, a new reagent supply system was developed. This technique eliminated the use of vaporizers for the three organometallic compounds by simply mixing the three materials as fine powders into a specific ratio, and pneumatically feeding the powder directly into the furnace using a high gas carrier flow rate of argon (5 ℓ/min).

This idea is currently under patent consideration. Not only is the equipment operation simplified by the elimination of vaporizers, but the poor volatility of the reagent precursors is avoided. The reactant gas is essentially saturated with the vapor containing Y, Ba, and Cu when the mixture enters the heated (900°C) reaction zone. A schematic of a horizontal CVD system is shown in Figure 2-7. Along with the simplification of equipment operation, the other major advantage includes the ability to greatly enhance and control the deposition rate. Films were deposited at rates of 2 to 240 μm/h. The deposition efficiency remained comparable to the conventional technique, even at the fast feed rate (0.2-0.6% versus 1%). (See Appendix A) The potential for scale-up and commercialization of the process is also an advantage over conventional CVD techniques. Experimental results from this work will be presented in a later section of the report; however, several publications and presentations have already resulted[70,71].

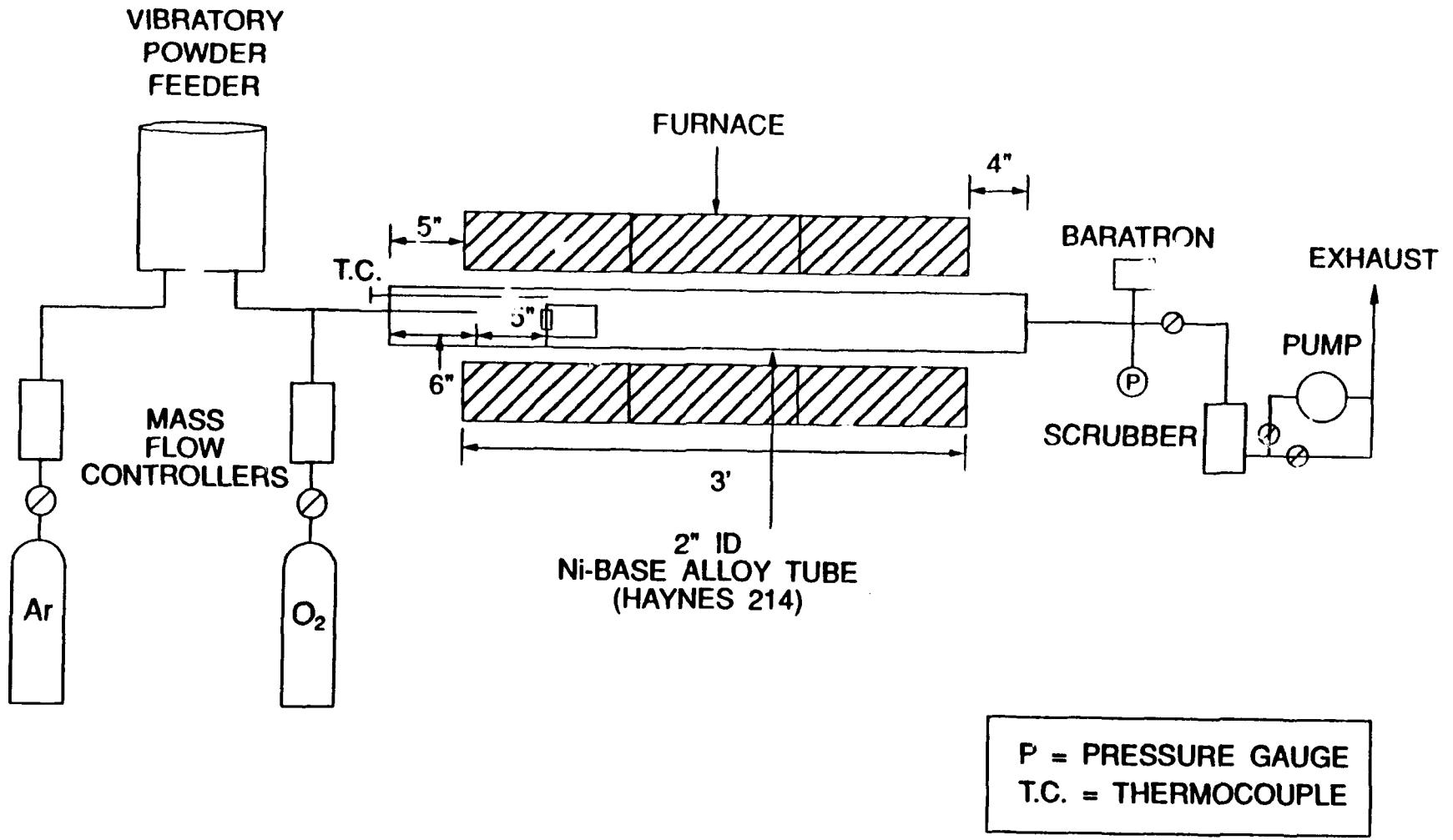


Figure 2-7. Organometallic powder feeding schematic for the horizontal CVD system used at Georgia Tech for $YBa_2Cu_3O_x$ deposition.

D. Fiber Coating

In parallel with experimental work using a batch horizontal CVD furnace at Georgia Tech, a continuous fiber coating furnace was designed and purchased. This equipment was designed based on a review of previous designs of fiber coating equipment. The importance of fiber coating technology extends into the field of high temperature, reinforced composite materials research. The ability to coat fibers used in these structures in order to tailor the fiber-matrix interface is currently being studied[72].

1. Design and Modification

A continuous fiber coating furnace design was presented by Higgs et al.[73], in 1964, for coating graphite yarn with pyrolytic graphite. The design consisted of a fiber feed spool, followed by several tension rollers, and graphite to graphite wheels on both sides of the furnace. The yarn was heated by placing a controllable electric current across the two graphite wheels. To prevent oxidation of the yarn, the furnace was continuously flushed with argon. The fiber pull speed was varied from 200 to 570 cm/min; no coating thicknesses were reported.

Studies by Brennfleck et al.[74-76], which deposited SiC on carbon fibers presented a quartz reactor with a motorized supply and take-up spool to control the fiber speed. The decomposition reaction was completed at 1 atmosphere, so vacuum equipment was unnecessary. Brennfleck mentioned the need for a vibration device, perpendicular to the fiber axis, which is required to prevent coating bridge formation between fiber monofilaments of a tow. Film thickness on

each fiber was determined to be about 100 nm; no correlation between fiber pull rate and thickness was presented.

Debolt et al.[77-78], studied deposition of SiC on tungsten and carbon monofilaments. The fiber coater was described as a 1.25 cm ID x 180 cm long glass chamber with the filament passing through a mercury pool at the inlet and outlet, which sealed the reaction chamber due to the high surface tension of the mercury, and allowed for an electrical contact to heat the fiber. An entrance zone, consisting of a heated hydrogen purge "cleaning stage" was shown on the system schematic.

Galasso et al.[79], presented another fiber coating furnace for studying high resistance inorganic coatings on graphite fiber. Fiber coating rates were typically 2.8 to 15 μm/min, which were determined by static coating experiments. A quartz chamber was used with an external RF coil heating the fiber. Coating thicknesses were varied from 0.14 to 0.4 μm. A unique feature to this design was an extension at the inlet and outlet of 15-30 cm, where an argon purge was used to contain the reagents in the chamber.

J. Cornie[80] studied deposition of HfC onto SiC filaments with fiber feed rates varying from 5 to 60 cm/min, and achieving coating thicknesses of 5-8 μm. A unique feature to the continuous coater was that the filaments were tension fed, using an adjustable magnetic brake on the spool axles, therefore eliminating motorized spool motion and electrical connections.

Aggour et al.[81], studied the deposition of several coatings onto various types of carbon fibers. Coating thicknesses of 0.2

to 0.8 μm were reported for residence times of 10 to 1800 seconds. This corresponds to fiber pull rates of 13 to 240 cm/min. The equipment was described as a 40 cm hot walled graphite reactor with an external RF induction coil used for heating. Both the supply and take-up spool were surrounded by a quartz glass shell, which permitted vacuum operation and containment of the reactants and exhaust gases.

A final fiber coating system was presented by Newkirk et al.[82], who studied tantalum coatings of carbon fibers for reinforced composites. Fiber pull rates of 3.3 to 83 cm/min were presented for a 40 cm hot walled reaction chamber. The fiber supply and take-up spool chambers were purged with a slow inert gas flow, typically argon or helium. The reaction chamber diameter was varied for the experiments, and two important findings were presented. First, as the diameter was decreased, the deposition rate increased. This was qualitatively explained as an increase in the mass transport due to the higher gas velocities at small diameters. Second, and in conflict to the first factor, is that when the reaction chamber diameter is reduced, the free and broken ends of the fiber bundle get trapped in the furnace, causing clogging of the path. When the reactor diameter was 1 cm and below, the fiber path became clogged shortly after starting the run.

2. Georgia Tech Fiber Coater

A schematic of the Georgia Tech fiber coating furnace is presented in Figure 2-8. The major features of this system consist

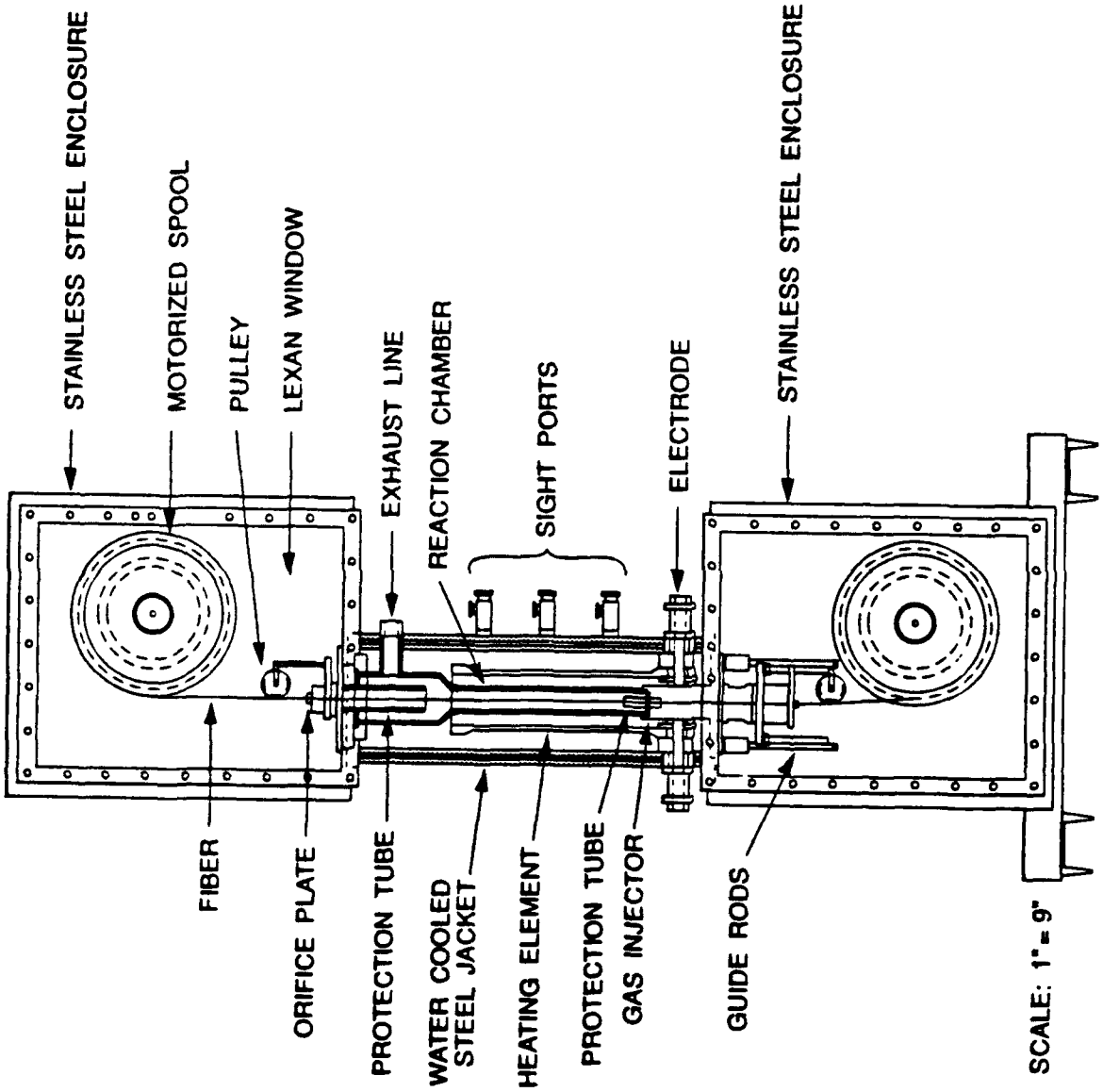


Figure 2-8. Continuous fiber coater used at Georgia Tech.

of a fiber supply spool located at the bottom of the furnace, a vertical, hot walled CVD furnace with a stainless steel water cooled jacket, and a take-up spool located at the top of the system. Each fiber spool is contained in a vacuum tight stainless steel box, which has removable 3.2 cm thick transparent lexan windows which allow monitoring of the fiber feed and take-up. The furnace design is hot-walled, which ensures that the entire fiber and chamber are heated uniformly, thereby eliminating Soret and wall effects. An argon purge is also provided in the top and bottom spool enclosures to prevent any contamination by back diffusion of reagents into the enclosures.

A thorough review of the design of the coater was presented by Lackey et al.[83]. Modifications to the equipment made to facilitate the deposition of the $YBa_2Cu_3O_x$ material will be presented in the experimental results.

3. Commercial Fiber Tows

The choice of fiber substrate for the $YBa_2Cu_3O_x$ coatings is limited by mainly by the availability of fiber tows, and the reactivity of the material with the coating. Table IV presents the fibers which were purchased for use at Georgia Tech. The material suppliers are listed below each fiber. Information on fiber diameter, Young's Modulus, composition, sizing, and number of filaments is included. Figure 2-9 lists common terminology used to describe commercial fiber tows.

The interaction of the $YBa_2Cu_3O_x$ material with several substrate materials has been studied by Cheung and Ruckenstein[84]. $YBa_2Cu_3O_x$

Table IV. List of continuous fiber tows used in experimental deposition of $YBa_2Cu_3O_x$.

Fiber Type (Vendor)	# Fibers per Tow	Size Diameter (μm)	Youngs Modulus (GPa)	Thermal Expansion ($x10^{-6}/°C$)	Sizing Information	Composition/ Comments
PRD-166 (DuPont)	200	21±5	380		organic burns off @ 350-400°C	ZrO_2 + Al_2O_3 strong but brittle, easiest fiber to handle
FP Al_2O_3 (DuPont)	200	21±5	345-379	7.2		> 99% Al_2O_3, very brittle, ran several stagnant
Nextel 480 (3M)	420	10-12	220	4.38 to 4.99	organic carbowax + H_2O	70% Al_2O_3 2% B_2O_3 28% SiO_2
Nextel 440 (3M)	420	10-12	207-241	4.38 to 4.99		
Nextel 211 (3M)	420	10-12	--			32% SiO_2, 68% ZrO_2 nearly impossible to use
Sumitomo (Textron)	1000	15-20	210	6	organic PVA burns off @ ~ 400-500°C	85% Al_2O_3, 15% SiO_2 extensive fraying
Kanthol Ribbon	--	--				4 stagnant runs 1 continuous
3M	400	9	350	7.8	PVA sizing	>99% Al_2O_3
Carbon (Several Vendors)	1000 - 12000	5-7	276		sized or unsized	smallest diameter, needes oxidation protective coating

Continuous Strand	A stand in which individual filament lengths approach the strand length.
End	A single strand, roving or yarn, incorporated in a product.
Fiber	Thread-like structure having a length at least 100 times its diameter; can be either definite short lengths or continuous.
Filament	Variety of fiber having extreme length.
Fill	Ends that run crossways in a fabric.
Finish	Material applied to fiber products to improve fiber-resin bonding, improve lubricity and high temperature abrasion, or stabilize a weave. Can contain a coupling agent.
Griege Goods	Loom state fabric; fabric that may contain sizing but no finish.
Heat Cleaning	Batch or continuous process to remove sizing.
Plied Yarn	An assembly of two or more previously twisted yarns.
Roving	A loose assemblage of fibers in single strand, without twists.
Serving	Wrapping a yarn such as rayon around a roving or yarn for protection.
Singles	A yarn made from one or more strands twisted together but not plied. Examples: 1/0, 2/0, 4/0. Single strand construction is singles yarn made from one strand (1/0).
Sizing	Starch, oil, wax, or other suitable organic ingredient applied to a fiber strand to protect and aid handling. A sizing contains ingredients to provide lubricity and binding action. Unlike a finish, a sizing is usually removed before final product use.
Sliver	Overlapping, parallel staple fibers that have been gathered into a continuous bundle .
Staple	Natural fibers or cut lengths from filaments [less than 17 inches (43.18cm)] to be gathered into slivers.
Strand	An untwisted primary filament bundle, can be either continuous filament or staple fiber (sliver).
Strand Count	Measure of linear strand density expressed in suitable units. Example: denier - number of grams per 9000 meters tex - number of grams per 1000 meters
Throwing	Twisting and/or plying of strands or singles.
Warp	Ends that run lengthwise in a fabric.
Yarn	An assembly of one or more strands twisted together. Examples: 1/0, 4/0, 1/2, 2/2, 4/5.

Figure 2-9. List of common terminology used in fiber technology.

powder was mixed with several materials and sintered at temperatures above 900°C. X-ray diffraction was used to show the interaction products and "rank" the reactivity of the $YBa_2Cu_3O_x$/substrate mixture. A summary list of these results is shown below:

$$Nb>Si>ZrO_2>Al_2O_3>SrTiO_3>MgO>Cu>Ag\ .$$

Silicon reacted to form Ba_2SiO_4; alumina reacted to form $BaAl_2O_4$. The MgO/123 material had a slight copper enrichment of the MgO phase, and a glassy Ba-Cu phase was also noted.

As can be seen from Table IV and the above list, it appears that no commercial fiber can be obtained which can act as a favorable substrate material with the $YBa_2Cu_3O_x$. The 3M fibers (Nextel) contain SiO_2, as does the Textron Sumitomo fiber. The most promising fibers include the pure polycrystalline Al_2O_3 fibers (FP DuPont fiber, and 3M), and the 85% Al_2O_3 + 15% ZrO_2 fiber (DuPont PRD-166). Even though a $BaAl_2O_3$ layer is expected to form, previous and ongoing work developing barrier layer coatings may provide the protection necessary to deposit a superconducting $YBa_2Cu_3O_x$ film. One barrier layer of particular interest is silver. The chemical vapor deposition of this material has been the focus of research by M. J. Shapiro at Georgia Tech.[84] The interaction of Ag with the 123 material is minimal, as presented by Cheung and Ruckenstein[84]. Other proposed barrier coatings mentioned in the literature are BaF_2 and ZrO_2[36,85-86].

Another factor in fiber selection is the size (diameter) of the fibers commercially available. In Figure 1, a 4 μm fiber with a 2 μm coating is shown; as presented in Table IV, the smallest

diameter for a commercially available oxide fiber is approximately 10 μm. In order to have a 75 volume % superconducting material on a 10 μm fiber, a coating thickness of 5 μm is required. Carbon fibers, which are available down to 5 μm diameters, have not been investigated as a substitute material. The high temperature oxygen rich environment would combust the material. Possible oxidation resistant coatings could be used prior to $YBa_2Cu_3O_x$ deposition on carbon fibers, but no work in this area has been pursued. The lack of a suitable commercial fiber substrate remains a major limitation to this technology. A zirconia fiber would be of interest but none is currently commercially available.

EXPERIMENTAL PROCEDURE

A. Deposition

The experimental procedure for the deposition of $YBa_2Cu_3O_x$ involves the handling of reagents, the deposition of the films, sample handling, and finally characterization of the samples. Each facet of this work was developed over a three year period.

1. Reagent Handling

Organometallic Y, Ba, and Cu reagents were used in the CVD of $YBa_2Cu_3O_x$. Both commercial (purchased from Strem Chemicals) and in-house reagents were used in the deposition work. The cost of the TMHD and ACAC organometallic reagents is presented in Table V. The values presented in Table V represent the commercial costs of small quantities of the reagents; in-house fabrication of the same reagents was significantly cheaper. Since most of the deposition runs were completed using the TMHD reagents, the cost/run was extremely significant--nearly $100 in chemical costs/run.

Table V. Comparison costs for the organometallic reagents used in the CVD of $YBa_2Cu_3O_x$.

	Cost ($/g)	
Reagent Type	Acetylacetonates	TMHD's
Yttrium	0.80	16.00
Barium	0.35	12.00
Copper	0.18	12.00

Thermogravimetric measurements (TGA) studying weight loss versus time or temperature were made on the reagents in order to understand the thermal performance of the compounds and to pinpoint the optimal vaporization temperature. TGA was also used to determine if water adsorption on the compounds changed their volatility. Typically, a 100 mg sample was placed in a platinum boat and heated at a rate of 10°C/min in a DuPont Thermogravimetric Analyzer. Temperature-time profiles were used to study the decomposition rate of the material.

Initially, the reagents were kept in a desiccator, with Drierite used to eliminate the water in the environment. Due to concerns over reagent degradation, the organometallics were later kept in a vacuum desiccator to prevent water adsorption and material contamination.

2. Equipment

Thin films of $YBa_2Cu_3O_x$ were deposited using three CVD furnaces. A horizontal system (shown in Figure 2-7) and a vertical furnace were used for batch processing runs on flat and fiber substrates. A vertical, continuous fiber coating furnace (shown in Figure 2-8) was used to deposit films on longer lengths of both fibers and flexible tapes. System descriptions are presented below.

The $YBa_2Cu_3O_x$ deposition was performed using the horizontal CVD reactor shown in Figure 2-7. The clam shell furnace (Lindberg Model #59744A) had three independently controllable temperature zones, and surrounded a 115 cm long, 5 cm diameter Haynes 214 alloy

reaction tube with water cooled stainless steel endcaps. Temperature was monitored using an Inconel sheathed Type K thermocouple. Ultra high purity argon and oxygen gases were metered using MKS mass flow controllers (Type 259B), and controlled using an MKS control box (Type 247C). The gases were introduced into the reaction tube through 0.635 cm Cajon ultra torr fittings in the endcap. The system was maintained at a reduced pressure using a Leybold-Heraeus D8AC vacuum pump which was charged with a fluorocarbon oil (Fomblin) for oxygen use. The pressure was monitored using an MKS Baratron (Type 122A) gauge with a digital readout (Type PRD-1).

The vertical furnace consisted of a stainless steel water cooled shell, with a resistively heated graphite element. Several materials were used for the reaction chamber, including quartz, SiC, and Inconel. The 76 cm long, 2.54 cm diameter chamber isolated the reactant gases from the heating element. Similar gas handling and pumping setups were used in the studies; the reactants were introduced into the chamber through a water cooled gas injector, and flowed upwards through the system.

The continuous fiber coating furnace (Figure 2-8) is similar to the vertical furnace, with the addition of two 61 cm x 91 cm stainless steel enclosures at the top and bottom of the furnace. These enclosures house the fiber spool assembly -- a 30 cm diameter motorized fiber supply spool is located in the bottom enclosure; plastic guides align the fiber up through the 2.54 cm diameter reaction chamber and into the top enclosure, where it is wound onto a motorized takeup spool. Transparent lexan plates are used to

view the fiber movement during the experiment. A small purge of argon (500 cm^3/min) into the spool enclosures was used to prevent reagent back diffusion into the fiber supply and handling equipment. Gas handling and pumping equipment is similar to the previous description.

3. Deposition Procedure

Following some initial experimentation using a vaporizer approach to the deposition of $YBa_2Cu_3O_x$, the powder feeding of reagents was developed. The following general experimental procedure was followed for the powder feeding approach, which involved over 750 of the 900 batch processing runs, and all of the fiber coating experiments.

The three powder organometallic compounds ($Y(tmhd)_3$, $Ba(tmhd)_2$, and $Cu(tmhd)_2$) were combined in a weight ratio (typically 1:1.38:1.51), and mixed in air using a Spex Mixer/Mill (Model #800) for one minute. The fine powder was then loaded into the slots of a Georgia Tech designed controllable powder feeder shown in Figure 3-1. The feeder was kept under vacuum until the deposition started. Approximately 2 grams of mixed reagents were loaded into the powder feeder; a typical run lasted 20 minutes.

For the horizontal furnace, the 10 cm long fiber and 1 cm^2 flat samples were loaded into an Inconel wire cage, centered in the reaction tube, and placed 28 cm from the front of the reaction tube. Following the furnace heatup, the argon gas was turned on and the powder feeder was started at a speed setting of 25%. The argon (usually 5 ℓ/min) pneumatically transported the finely ground

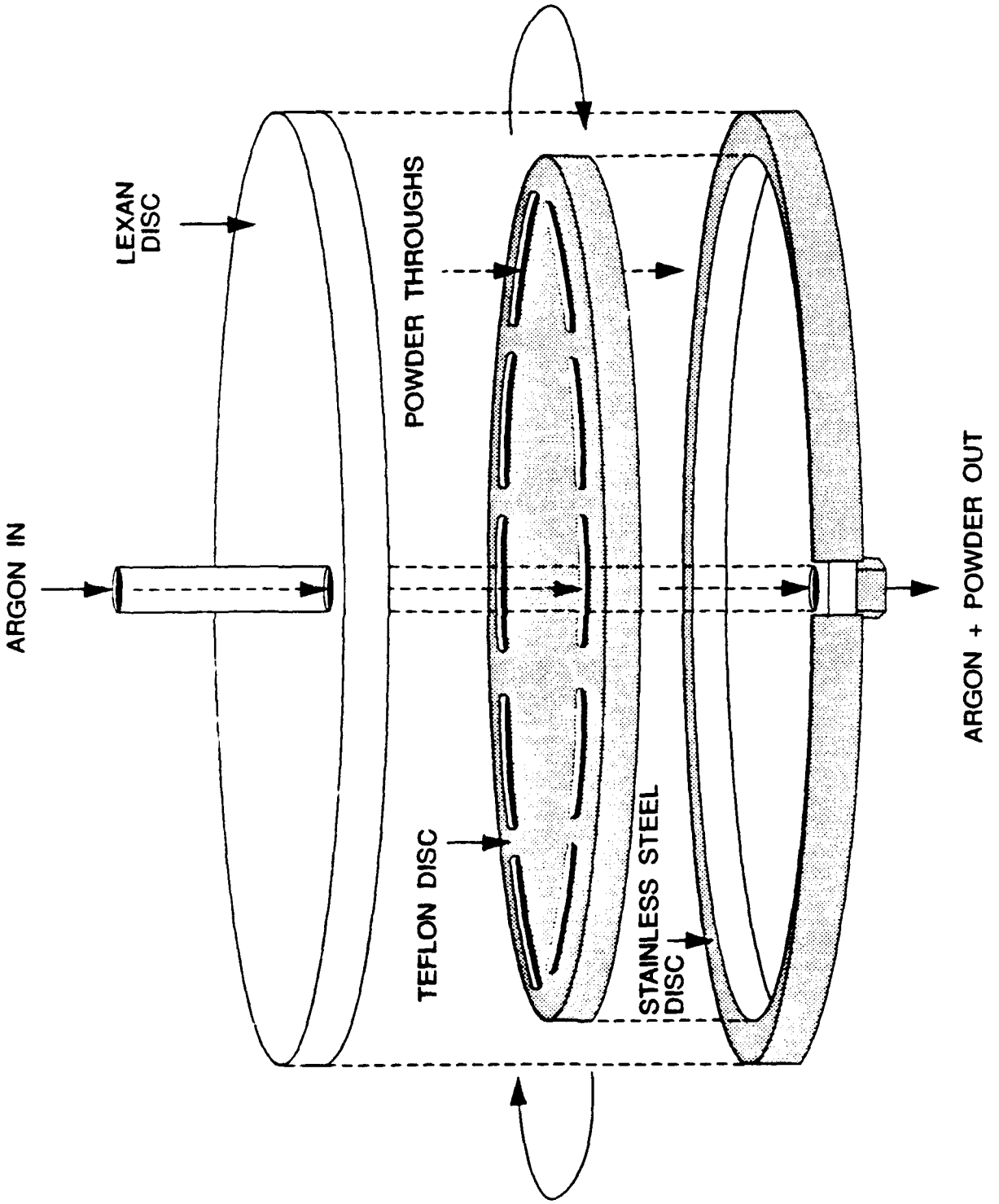


Figure 3-1. Exploded view of the powder feeder design.

powder into the furnace through a 0.635 cm diameter Inconel tube, where it was introduced into the reaction tube 12.7 cm from the samples. Oxygen (1 ℓ/min) was mixed with the argon and powder before entering the furnace.

Following the deposition, the argon was turned off, and oxygen was used to backfill the system to atmospheric pressure. During cooldown, the oxygen was left on at 1 ℓ/min, and was passed through a 500°C CuO furnace, Ascarite, and Drierite in order to eliminate any organics, water, or carbon dioxide impurities.

4. Sample Handling

Following deposition, samples were weighed on a microbalance, observed using an optical microscope, and measured for room temperature resistance using a 2 point Fluke 8026B True RMS Multimeter. The samples were kept in a desiccator with Ascerite and Drierite until they were subsequently characterized. Each sample was given an M number for identification. Whenever the sample was moved from one laboratory to another, a plastic bag filled with Ascerite and Drierite was used. This was necessary to prevent any surface contamination, and to prolong the integrity of the coating.

5. Sample descriptions

Several sample types were coated with the $YBa_2Cu_3O_x$ material. Both polycrystalline and single crystal flat plate and fiber tow and monofilament samples were used. A list of these materials and brief descriptions are presented in Table VI.

Table VI. Substrates and vendors used for depositing the $YBa_2Cu_3O_x$ compound.

	Substrate	Crystallinity	Vendor	Comments
Flats	MgO	single crystal	Canadian Sub. Supply	Rough cut, several cleavage steps
	MgO	single crystal	Estes	Polished on one side
	PSZ	single crystal	Ceres Corp.	Polished 1cm^2
	Ag	polycrystalline		several sources suspected large oxygen content
	$LaAlO_3$	single crystal	Commercial Crystal	Brittle, easily cleaved
	$LaGaO_3$	single crystal	Commercial Crystal	Brittle, easily cleaved
	$SrLaAlO_3$	single crystal	Dr. Chai (U. of Cent. Fla.)	Brittle, easily cleaved
	Al_2O_3	single crystal	Saphikon, Kyocera	
	Al_2O_3	polycrystalline	Coors	
	$SrTiO_3$	polycrystalline	TRAK Microwave Corp.	5mm x 5mm
	PSZ	polycrystalline	Kyocera	2 cm diameter disk
Fibers	Al_2O_3	single crystal	Saphikon	250, 150, 75 μm diameter
	Al_2O_3	polycrystalline	DuPont	FP Fiber (15-20 μm diameter)
	Al_2O_3	polycrystalline	3M	9 μm diameter
	Al_2O_3- ZrO_2	polycrystalline	DuPont	PRD - 166
	Si_3N_4	polycrystalline	Dow Corning	HPZ
	Al_2O_3 - SiO_2	polycrystalline	Textron	Sumitomo
	Ag	coated Al_2O_3 fiber tow	Georgia Tech	DuPont & Georgia Tech
Tape	Ag	polycrystalline		5mm wide, 0.5mm thick

B. Characterization

Coated samples were initially analyzed by optical microscopy, scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and x-ray diffraction (XRD). If the coating appeared promising, i.e., based on composition and the extent of c-axis orientation, it was characterized electrically. A flowchart of the characterization protocol is shown in Figure 3-2.

1. Microscopy

The microscopic evaluation of the samples was used to obtain surface morphology information and semiquantitative chemical analysis. A standard of $YBa_2Cu_3O_x$ was prepared by powder compaction and firing of $YBa_2Cu_3O_x$ powder obtained from Grace for use with the energy dispersive spectrometer. A list of the equipment and brief description and specifications follows.

Several optical microscopes were used to view the coating appearance and uniformity. Typically, a Cambridge Instruments Stereozoom 5, with a range of 0.8 to 40 X magnification was used.

Chemical and microstructural analysis of the films were completed using either a Cwikscan Model 100-4 scanning electron microscope (SEM) with a Kevex Model 7500 energy dispersive analyzer (EDS) or a Hitachi S-800 (SEM) equipped with a Kevex (EDS). An accelerating voltage of 15 kV was used. The samples were mounted using a small dot of graphite paint to eliminate any charging effects. The quantitative analysis were performed using a "standardless" technique[87].

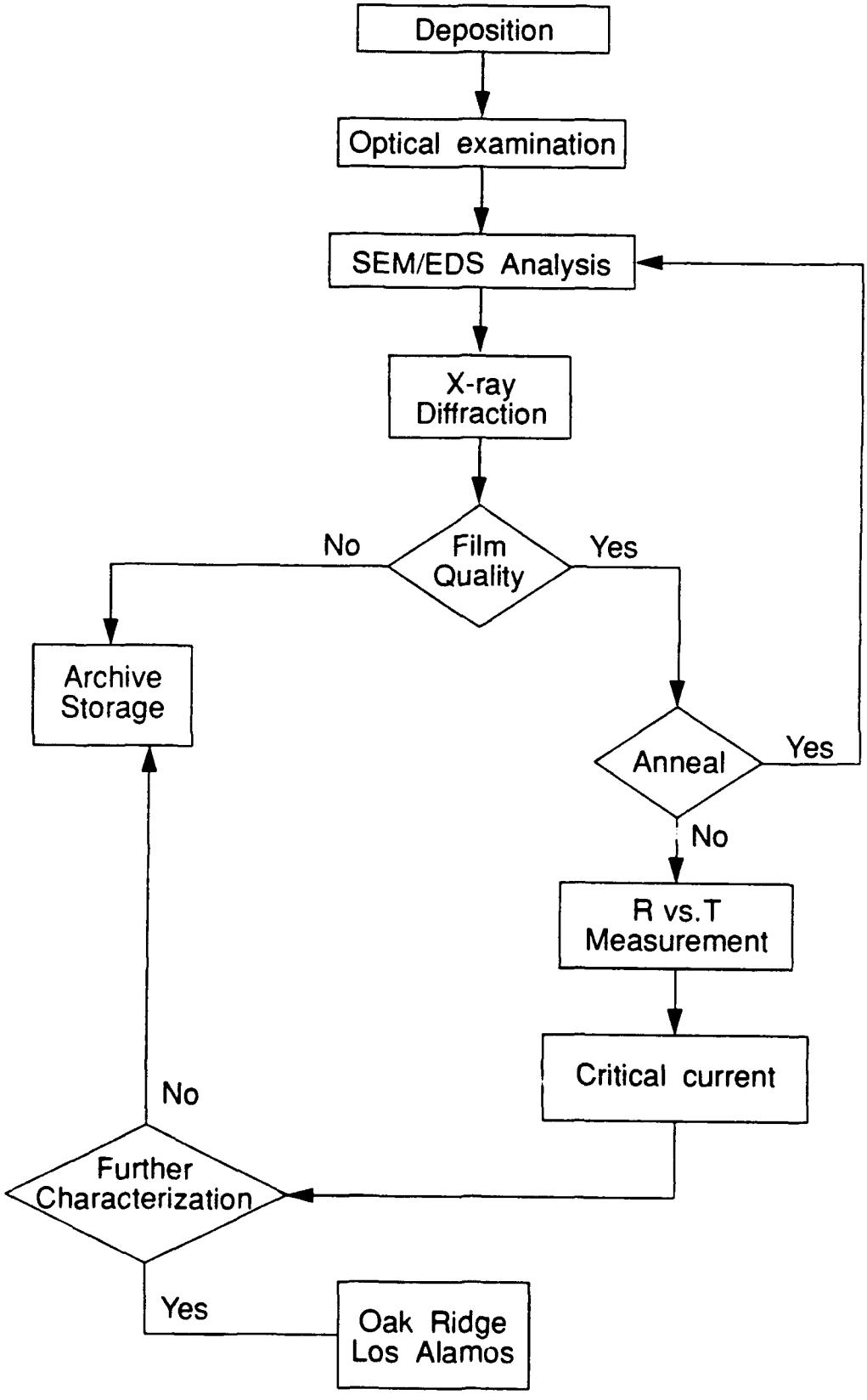


Figure 3-2. Characterization protocol for $YBa_2Cu_3O_x$ deposits.

Transmission electron microscopy (TEM) was completed in cooperation with the Oak Ridge National Laboratory Superconducting Pilot Center through an agreement between American Magnetics, Inc. and the Oak Ridge National Laboratory. Cross sections of the samples were prepared by gluing two coatings face-to-face and subsequently cutting slices from this "sandwich". Specimens were mechanically thinned to 75 μm from the substrate side and then dimpled and back-thinned in the ion mill to perforation. The specimens were ion milled at 77 K to minimize damage to the superconducting film. Samples were analyzed with both a JEOL 2000FX analytical electron microscope operated at 200 kV and a high resolution JEOL 4000EX electron microscope operated at 400 kV. All photographs were taken near Scherzer defocus (47 nm) and a convergence angle of 1 mrad.

2. X-ray Diffraction

One of the most useful characterization tools was X-ray diffraction. The presence of strong c-axis oriented peaks and the analysis of impurity phases present in the films was routinely completed.

The equipment used was a Philips PW-1800 Automated Powder Diffractometer operated with Cu K_α radiation, 40 kV/30 mA, scanned from 20-60° 2θ, which encompassed the major peaks of interest. The step size was 0.010° 2θ, and the sampling time was 0.8 seconds/step.

Pole figure analysis was completed using an apparatus constructed at Georgia Tech using the Schulz reflection geometry. Copper K_α radiation, operated at 40 kV and 20 mA was filtered and

introduced at a fixed beam size and shape. The specimens were carefully mounted to align the beam, and tilted at the horizontal axis while rotating at an axis normal to its surface[8].

3. Electrical Characterization

Promising samples were further characterized using mainly resistance versus temperature techniques. Several post-deposition annealing procedures were completed prior to electrical testing. A schematic of the annealing furnace is shown in Figure 3-3. Gases were passed through a furnace containing CuO at 500°C and subsequently through Drierite and Ascerite in order to eliminate any organics, water vapor, or carbon dioxide respectively. The annealing was performed in a Marshall Model 227 tube furnace surrounding a 91 cm long, 5 cm diameter quartz reaction chamber. Temperature was controlled using an Omega Model CN-2010 programmable furnace controller and monitored using a Type K thermocouple. The annealing gases, which included argon, oxygen, and specified Ar/O_2 mixtures, were verified by an Ametek Thermox Model TM-1B Analyzer, which used a zirconia electrochemical cell as an oxygen sensor.

Resistance versus temperature measurements were completed using the Van der Pauw Four-Point test[9]. A schematic of the test configuration is shown in Figure 3-4. Samples were prepared for electrical testing by depositing silver contacts, two each for current and voltage. The contacts were either silver or silver/epoxy paint. Following a 500°C anneal in oxygen for the samples with silver paint, thin copper wires were attached using a conductive epoxy and mounted on the cold finger.

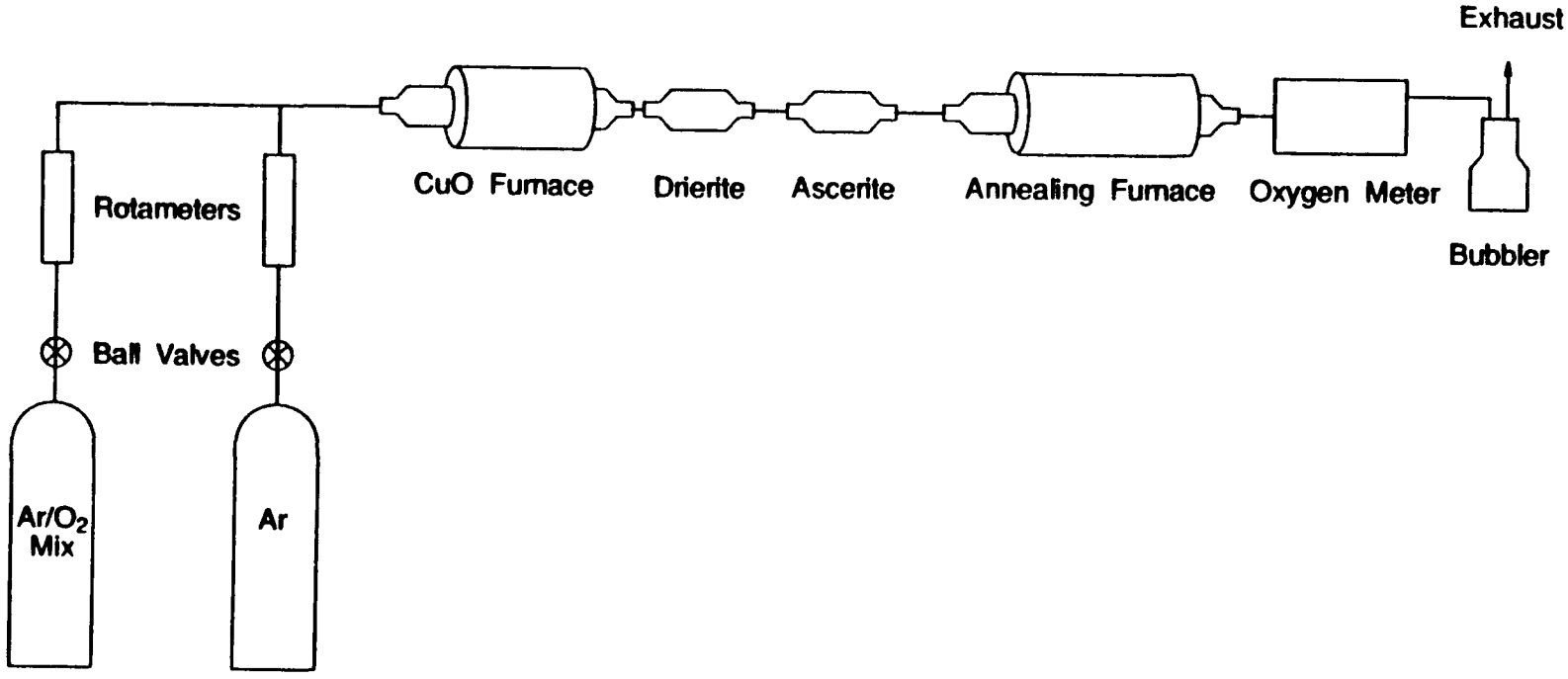


Figure 3-3. Furnace system used for post-deposition annealing of $YBa_2Cu_3O_x$.

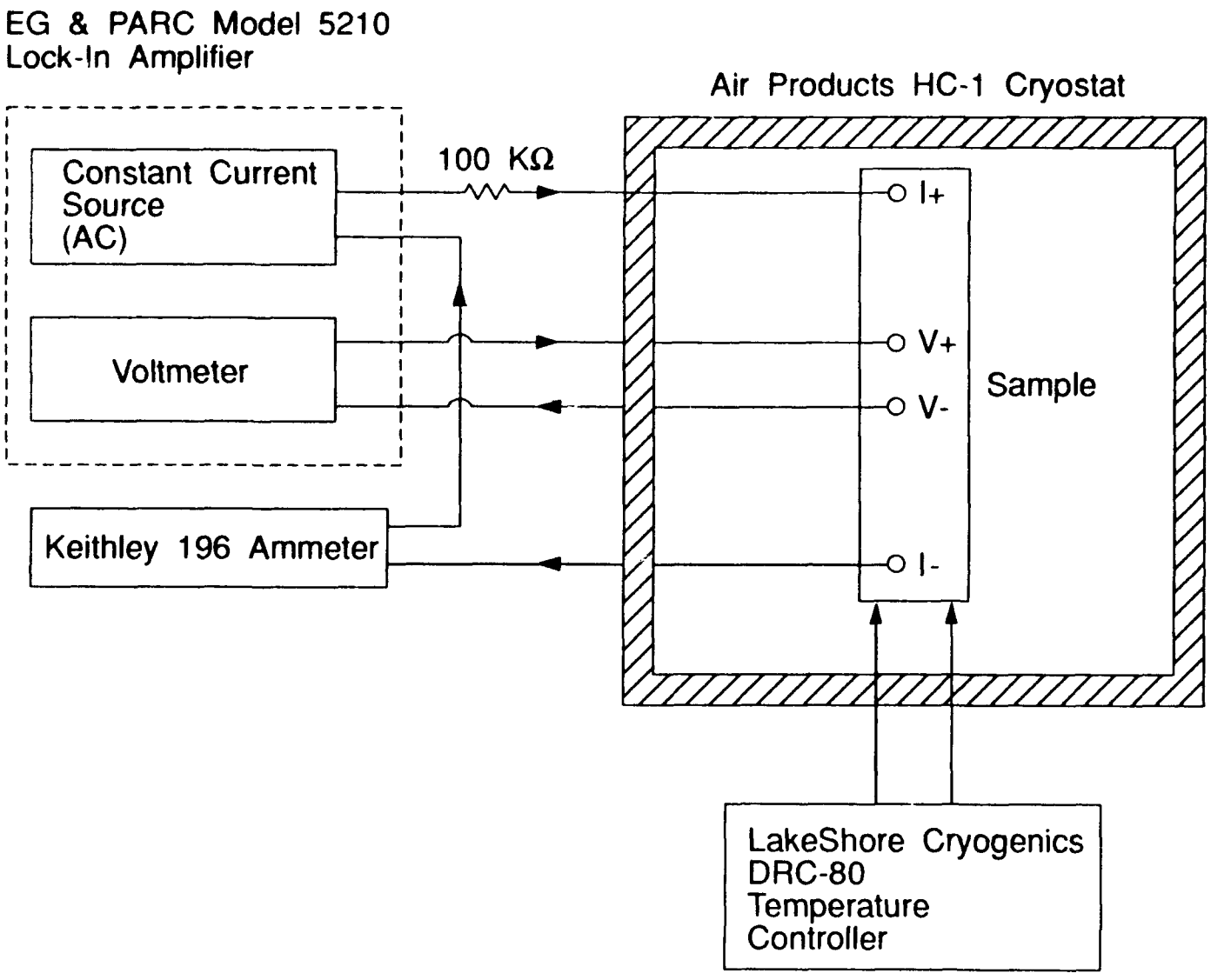


Figure 3-4. Configuration of the resistance versus temperature measurement technique.

AC current was supplied across the two outer contacts and the corresponding voltage drop was measured across the two inner contacts using lock-in techniques as the sample was cooled. The use of AC current eliminated thermal EMF's resulting from the Seebeck Effect. The resistance of the sample was then calculated using Ohm's law. The sample was considered to be superconducting when the voltage dropped below the detectable limit (in this work it was determined to be less than 100 nV).

An EG&G Princeton Applied Research Model 5210 lock-in amplified supplied an AC output ranging from 1.020-1.999 V at 113 Hz. This voltage was passed through a 100 kOhm resistor, resulting in an AC current of approximately 10 μA. Current was monitored using a Keithley 196 System Digital Multimeter. The sample voltage drop was then measured by the lock-in amplifier. An Air Products HC-2 closed cycle refrigerator was used to obtain the temperatures required. The temperature was measured by a Lakeshore Cryotronics DT 471-SD silicon diode and monitored with a Lakeshore Cryotronics Model DRC 80C Temperature Controller.

Critical current measurements were completed by first etching (using a razor blade) a narrow path (5 mm long and 3 mm thick) between the voltage contacts. (See Figure 3-5) The sample was then mounted on an aluminum disk and submerged into liquid nitrogen. The voltage was monitored as the current was raised from 10 μA to 100 mA, or until the sample was no longer considered superconducting. The critical current was defined as the current required to produce a field of 1 μV/cm across the constriction. The critical current density (A/cm^2) was calculated using the film

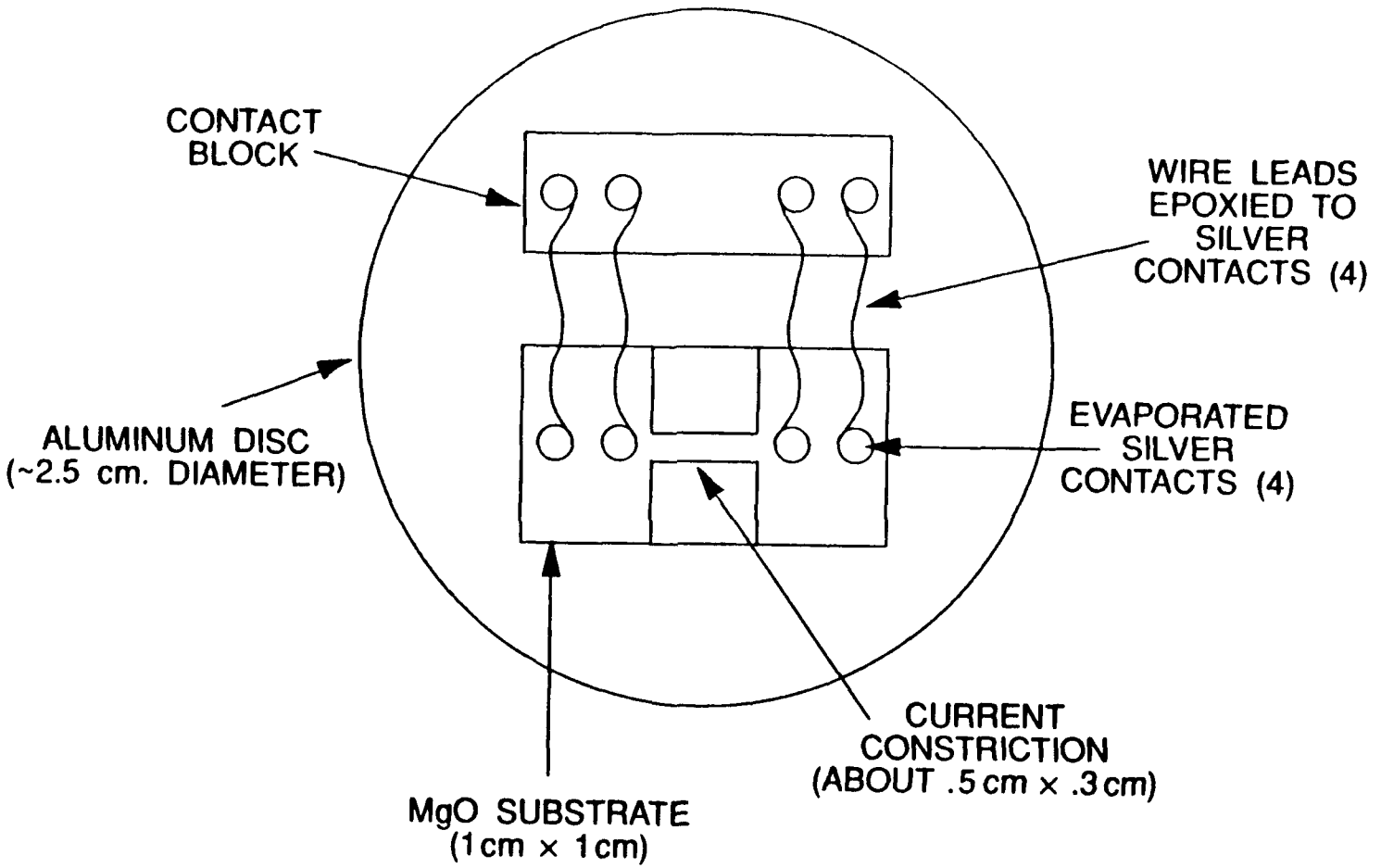


Figure 3-5. Schematic of the critical current configuration used for flat substrates.

thickness determined from profilometry and the width of the current constriction.

4. Fiber Characterization

The handling and characterization techniques were developed and continuously modified during the project in order to obtain improved and routine procedures. Following deposition, the fiber tows were optically characterized and evaluated for any further characterization. Since most of the uncoated fiber tows were white, the preliminary determination of coating uniformity and infiltration was made visually. Short sections (10-15 cm) of the fibers were divided and sent for microscopic, x-ray, and electrical characterization. Scanning electron microscopy was used to determine coating thickness on the filaments, and to verify and quantify the optical evaluation of infiltration and uniformity. Typically, the fibers were mounted by tying a thin silver wires at 2 points approximately 5 mm apart around the tows, and using a razor blade to slice between the ties. The cross section was then coated with carbon and glued to a hex bolt for analysis. Several samples were prepared for examination of the coating uniformity along the length of the filaments. Mounting and polishing of the fibers was not a viable technique since the fibers were harder than the coatings; i.e., the coating was polished away leaving a bare fiber.

Energy dispersive spectroscopy was also completed to determine relative percentages of the Y, Ba, and Cu present in the film. This analysis was complicated by the curvature of the fiber, and

the point to point variation of the position of the detector with respect to the sample. The data obtained were considered as only qualitative information on the film composition.

X-ray diffraction of the fiber tows was completed by cutting and stacking the coated tows onto a glass slide, and using tape or glue to anchor the filaments. The pattern intensity was typically low, with a large background. In order to avoid this problem, a large quantity of fibers were used to make up a "mat" of material.

Electrical testing of the filaments was the most difficult and trying procedure. Contacting each filament of the coated tow with silver was difficult and probably not accomplished. The fibers were typically brittle, accumulated static charge, and were oftentimes unmanageable. Techniques used included wrapping a silver wire around the tow and dabbing silver paint (Ernest F. Fullam 14810 silver paint) at the wire. This approach was complicated by the necking of the silver/methanol solution down the length of the fiber. Another technique was to evaporate silver on the fibers which were spread at the ends to maximize the filaments coated. This also proved to be extremely difficult since the sample had to be rotated to obtain the desired contact. A final technique was to utilize capillary tubes to help contain the fibers for further handling. Once the contacts were made, a semiconducting grease was used to anchor the test section to an aluminum holder. Four contacts were made for resistance versus temperature and critical current density measurements. Attempts at critical current density measurements were performed by submerging the sample in liquid nitrogen.

IV. RESULTS

A. Experimental Work

In order to understand and develop the CVD coating process in an efficient manner, deposition summaries were maintained throughout the project. The summaries are presented in Appendix B, and give a quick reference to the variations and parameters studied in each experiment.

1. Initial Deposition

Initial runs were completed in the horizontal and vertical CVD systems using the conventional vaporizer approach. The initial run conditions were based on work completed by the Japanese and NRL researchers[18,19]. Three vaporizers were loaded with approximately 2 grams of the Y and Ba TMHD, and Cu ACAC compounds respectively, and heated to 110-120°C for the Y, 250°C for the Ba, and 150-170°C for the Cu material. Argon was used as the carrier gas at 50 sccm for the Y and Cu, and 100 sccm for the Ba reagent. Oxygen was added in one of two places; either before the CVD reactor endcap (mixing with the reagents) or inside the CVD reactor in a separate line at a flowrate of 100 sccm. The total run time was typically 1 hour; temperature and pressure were 900°C and 0.02 atm, respectively.

Substrates including MgO and polycrystalline Al_2O_3 were initially placed inside an alumina boat; however, due to a lack of coating, an Inconel sample holder was designed to force the reagents to contact the substrate placed perpendicular to the flow

in the center of the reaction tube. In different experiments, the vaporizers were also placed both inside and outside of the CVD furnace in an attempt to achieve better reagent delivery. Initial results indicated that the vaporizer weight loss varied significantly from run to run; and the coating uniformity was extremely poor.

In order to understand the variation of reagent delivery from the vaporizers, a statistically designed study was completed which varied the carrier gas flowrates for each reagent. This "box" experiment and the resulting film compositions are shown in Figure 4-1. The argon carrier flowrate for each reagent is presented along the x, y, and z axes, the resulting film composition is indicated as the molar ratio Y:Ba:Cu at each corner, as determined by EDS. The center point, which was duplicated in the experiment, was identical to the earlier Japanese work. The extreme variability in the resulting compositions indicated poor reagent control during the initial experiments.

The Ba reagent had the greatest variation; which was a problem mentioned by other researchers. The Ba tended to undergo thermal degradation and therefore depletion in concentration during the run. This was studied using TGA; weight loss versus time at 250°C for the Ba TMHD reagent is presented in Figure 4-2. As shown, a non-constant slope was observed, indicating the vaporization behavior of the reagent varied with time.

Furnace and vaporizer profiles were also completed in order to determine temperature gradients present. When using internal vaporizers, a +/- 50°C differential was observed across the

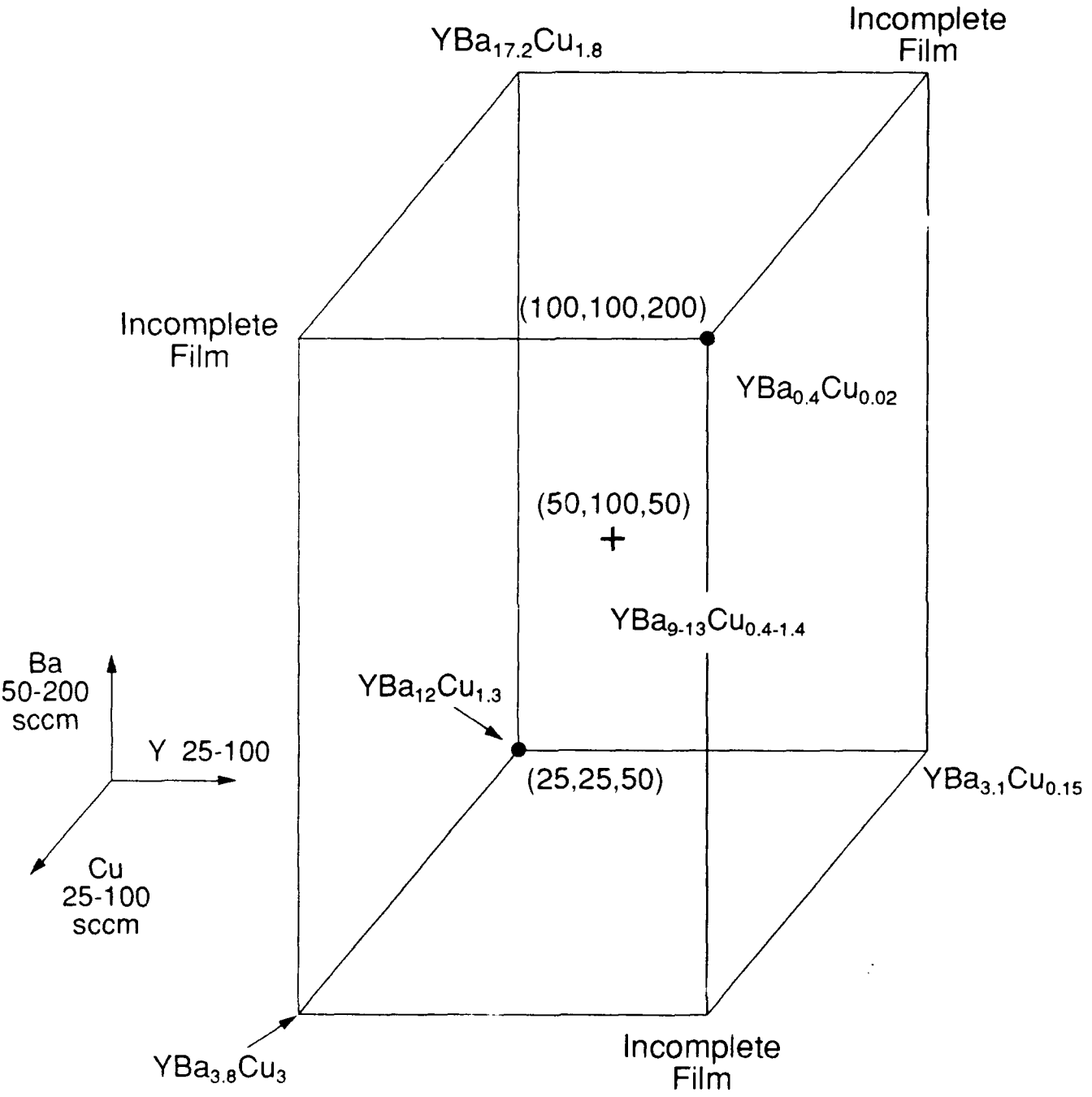


Figure 4-1. Statistical box design varying carrier gas flowrates. The film composition for each flow variation is indicated at the box corners.

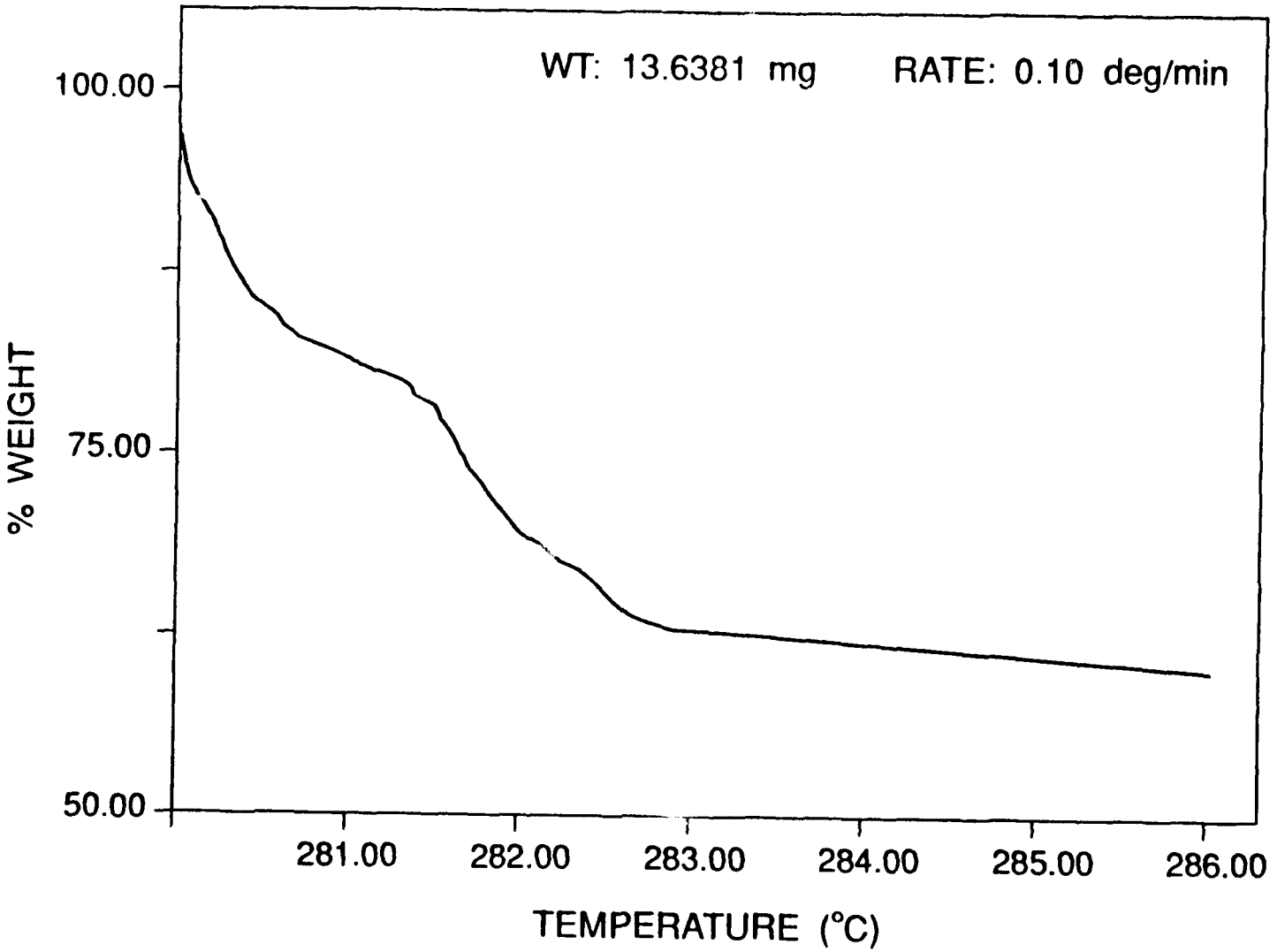


Figure 4-2. Thermogravimetric analysis of the $Ba(tmhd)_2$ reagent at 280°C. A non constant slope indicates a variable vaporization rate.

vaporizers. The furnace profile showed a 15 cm uniform hot zone. At this point in the research, several coating runs were completed using only one of the reagents, or reagent pairs, in order to separately study the vaporization/coating process. This work was abandoned, however, as the powder feeding approach was developed.

2. Powder Feeding

Concurrent with the vaporizer studies, the development of the powder feeding technology was pursued. Based on some previous experience in this field[90,91], a method for directly introducing a mixture of the powder reagents into the furnace was developed. The initial work utilized a commercial Syntron vibratory feeder, which was designed to feed large parts for a mechanized process. Modifications to the Syntron feeder were made to enhance feeding of the powder mixture and its entrainment into the process gas stream. These included:

1. Attaching a funnel at the feeder outlet,
2. Reducing the surface area of the "ramps" which vibrate the powder towards the funnel,
3. Attaching a powder/argon exit line to the system, and
4. Enclosing the feeder in a vacuum tight (stainless steel) vessel.

A cutaway schematic of the modified Syntron powder feeder is shown in Figure 4-3. A typical run used 5-10 grams of the premixed powders, and lasted from 5 to 30 minutes; the powder feed rate depended on the vibration control. The amount of powder fed also depended on the initial charge of powder in the bowl; therefore, the feed rate varied during the length of the run. The deposition rate resulting from this approach was much greater than any

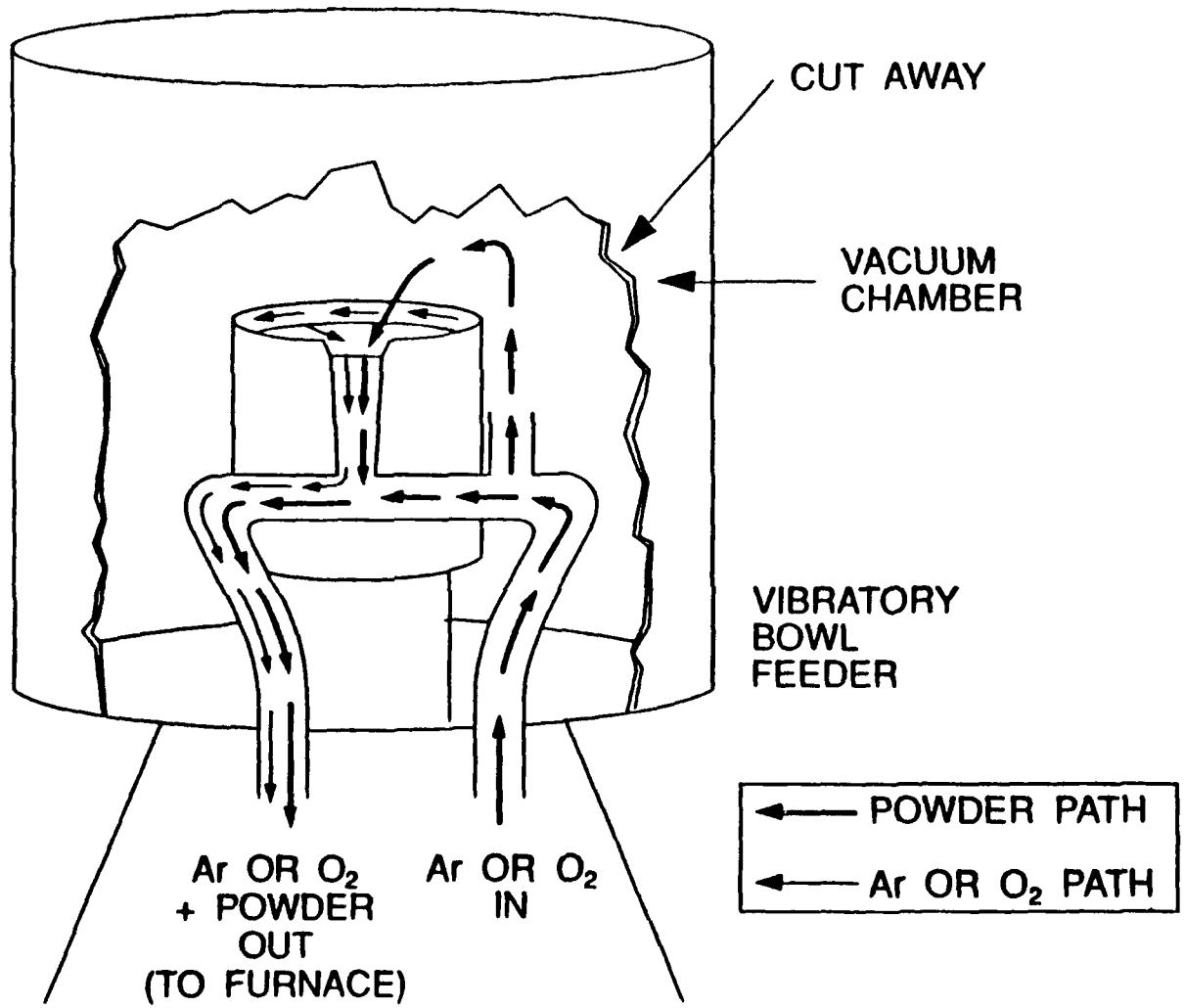


Figure 4-3. Schematic of the vibratory feeder design initially used to powder feed the reagents.

previous studies on the CVD of $YBa_2Cu_3O_x$. Prior to this work, the fastest rate reported was 10 μm/h. All the other reported rates were around 1 μm/h. Films deposited using the powder feeding approach ranged from 2 to 240 μm/h, determined using SEM examination of fractured cross sections for thickness.

a. Initial runs/ composition study

Initial runs using the powder feeder resulted in improved coatings as compared to the vaporizer approach. Optically, the films appeared black and uniform, with a grainy texture. Typical morphology, x-ray diffraction results, and energy dispersive spectra are shown in Figures 4-4 to 4-6. The morphology had a plate-like appearance (See Figure 4-4); indicating the presence of c-axis oriented grains. X-ray diffraction verified the presence of preferred orientation as shown in Figure 4-5. The (00ℓ) peaks are indexed, along with the impurities (including CuO, Y_2BaCuO_x [211]) and the unoriented $YBa_2Cu_3O_x$ peaks. Finally, the EDS analysis shown in Figure 4-6 indicated a molar Y, Ba, and Cu film composition of 17:31:49, close to the ideal 16.7:33.3:50.

Two point room temperature resistance measurements of the films typically ranged from 25 to 500 Ohms. Weight measurements were inconclusive; in some instances the sample showed a weight loss. This was attributed to the desorption of water from the MgO sample surface at the high deposition temperatures. Resistance versus temperature measurements for the films are presented in Figure 4-7. The solid black lines indicate the electrical behavior of the samples; which had critical temperatures between 69 and 78 K. The slope of the curve prior to the sharp superconducting

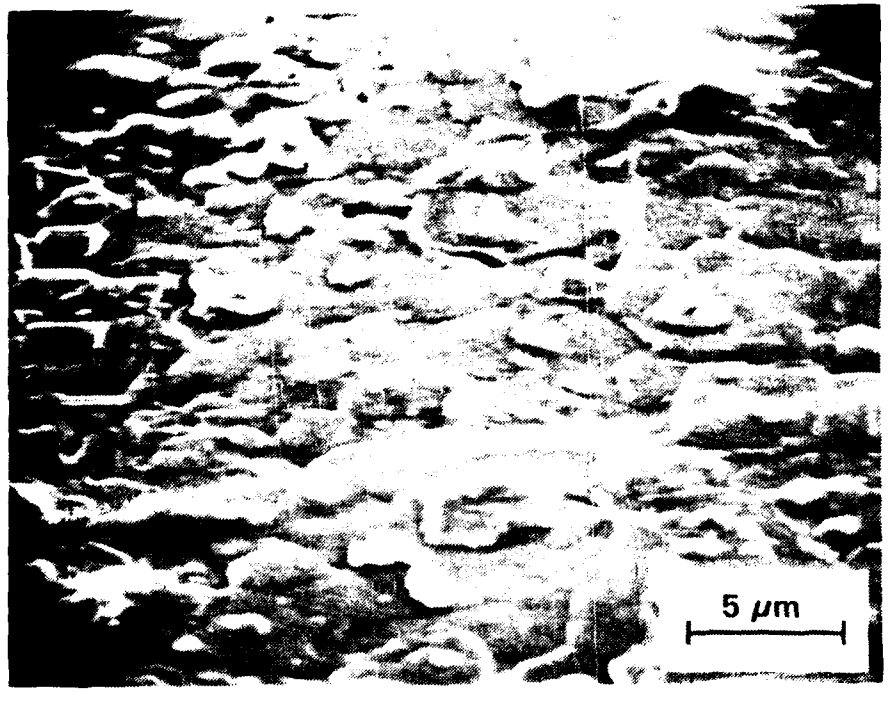


Figure 4-4. SEM micrographs show the plate-like features indicative of c-axis oriented material.

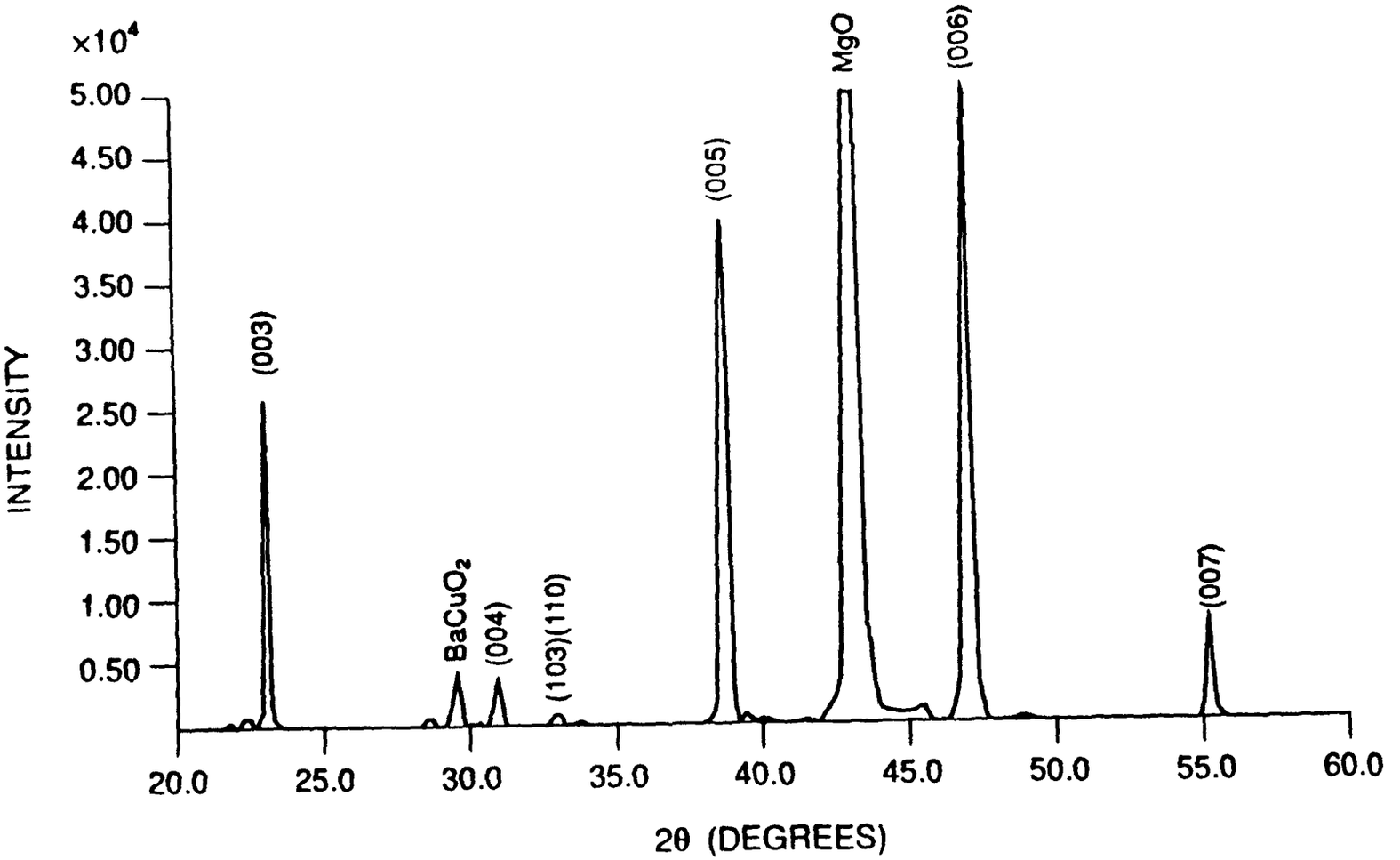


Figure 4-5. XRD pattern for $YBa_2Cu_3O_x$ deposited on MgO.

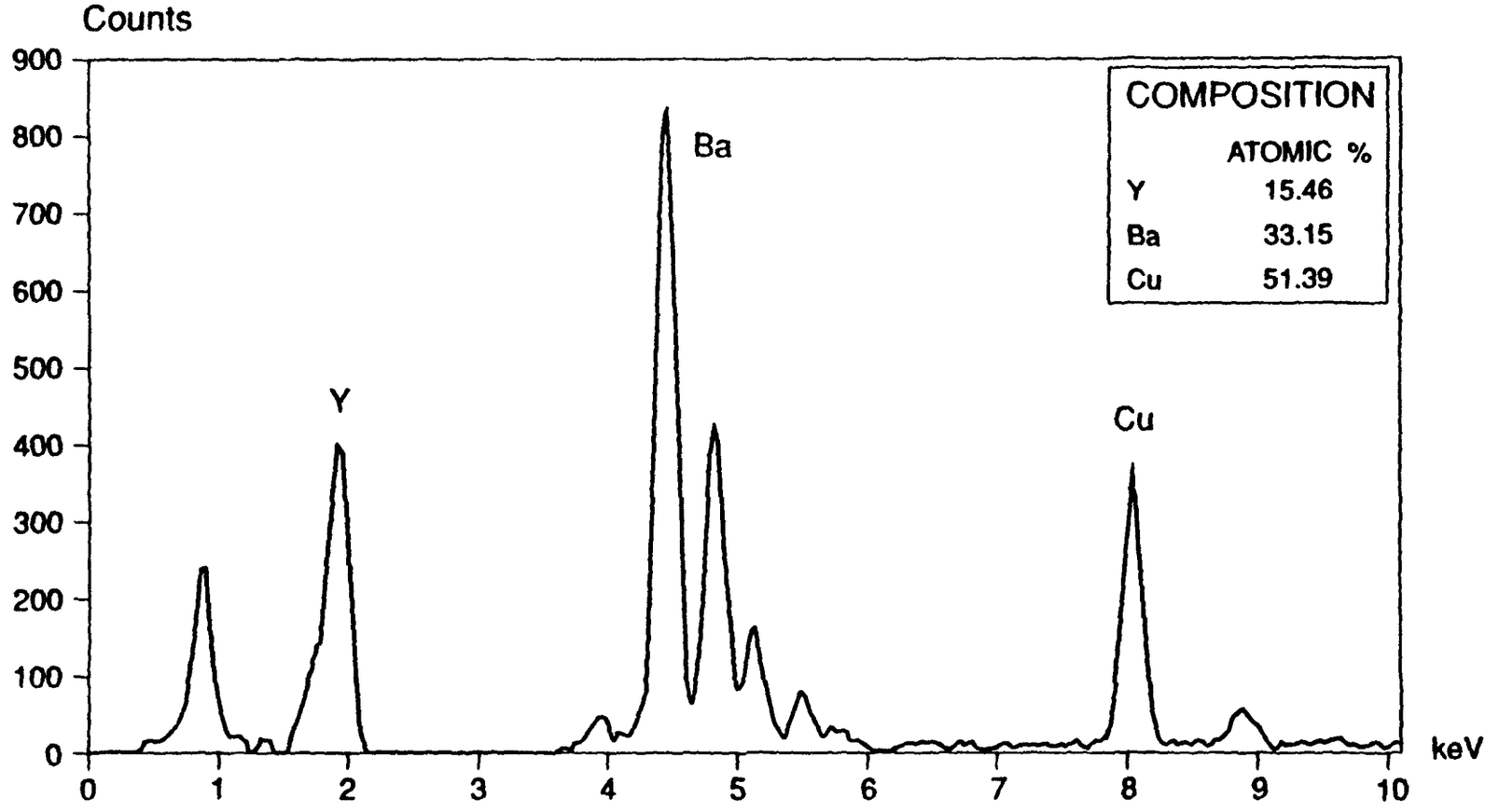


Figure 4-6. EDS spectra for $YBa_2Cu_3O_x$ deposited by MOCVD. The composition was determined using a standard of bulk $YBa_2Cu_3O_x$.

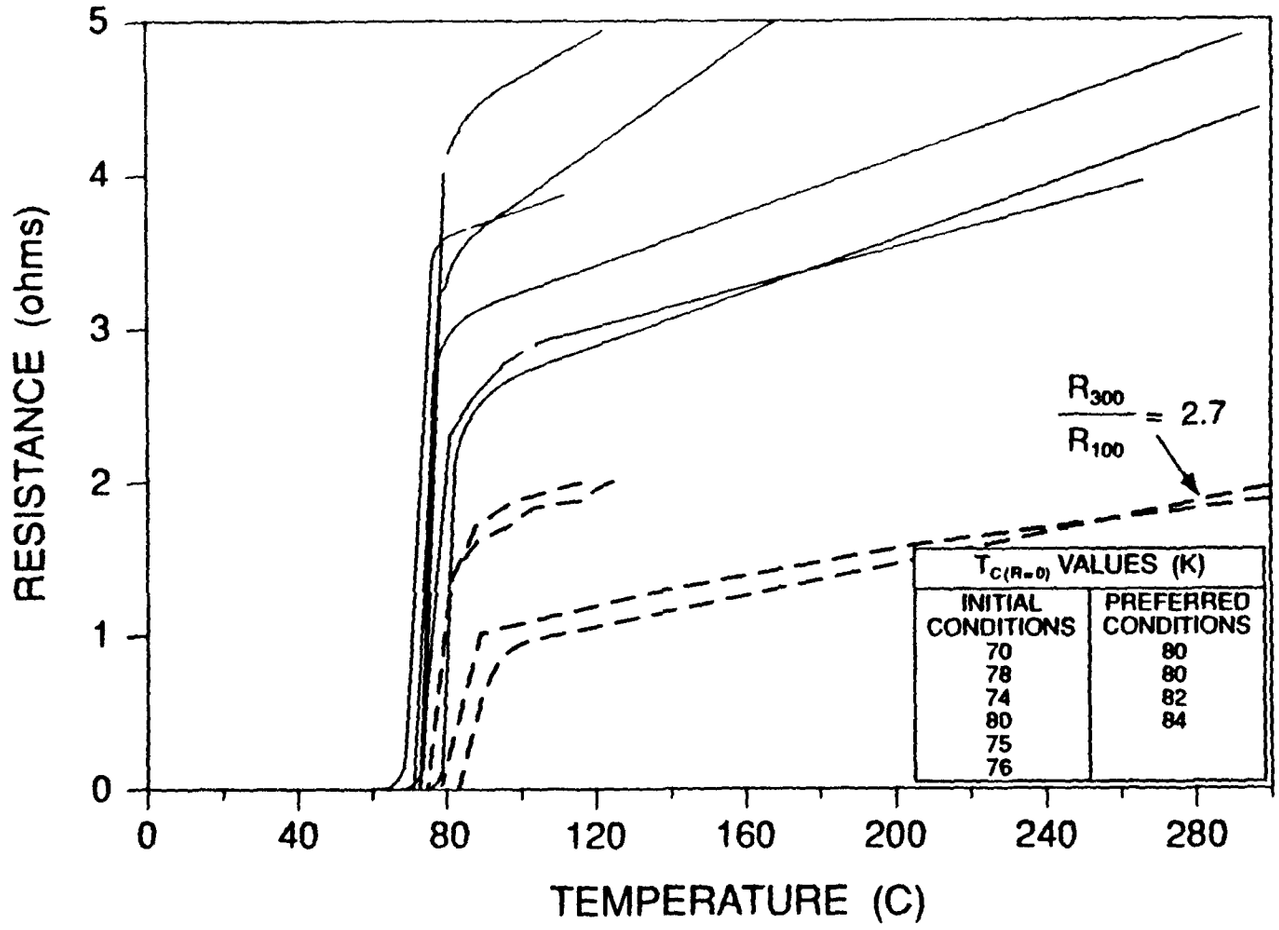


Figure 4-7. Resistance versus temperature curves for films deposited using the vibratory feeder. The solid lines represent the initial coatings, the dashed lines are for films deposited at the preferred conditions.

transition, commonly referred to as the R(300)/R(100) ratio (the ratio of the resistance at 300 K compared to the resistance at 100 K), ranged from 1.2 to 2.1.

In order to study the variation in final film composition as a function of input powder feed composition, a second statistically designed study was completed. Several of the early powder feeding runs were determined to be yttrium poor by EDS (13.0 vs. the desired 16.7 mole percent), prompting a design which specifically varied the yttrium input. Figure 4-8 shows a ternary composition diagram; the axes represent the molar content of the Y, Ba, and Cu TMHD compound present in the feed. Also indicated on the diagram is the ideal Y:Ba:Cu ratio for $YBa_2Cu_3O_x$, and the 10 feed compositions chosen for study. Two of the runs were replicated in order to indicate process repeatability. Figure 4-9 presents the initial to final molar composition of Y:Ba:Cu; the final film composition is indicated by the arrow tip, the ten feed compositions are at the tail of the arrows. As shown, there was a general trend resulting in a coating with a higher content of Cu, and a lower content of Y and Ba than in the input composition. Table VII gives a summary of the results. The improved films consisted of a plate-like morphology, indicating a strong c-axis orientation. The films also had low two point resistance values, and were slightly rich in Ba and Cu.

Several other processing parameters were varied in order to optimize the film quality, including temperature, pressure, and deposition rate. A summary of the parametric variations is given in Table VIII. Descriptions of these results are included in the

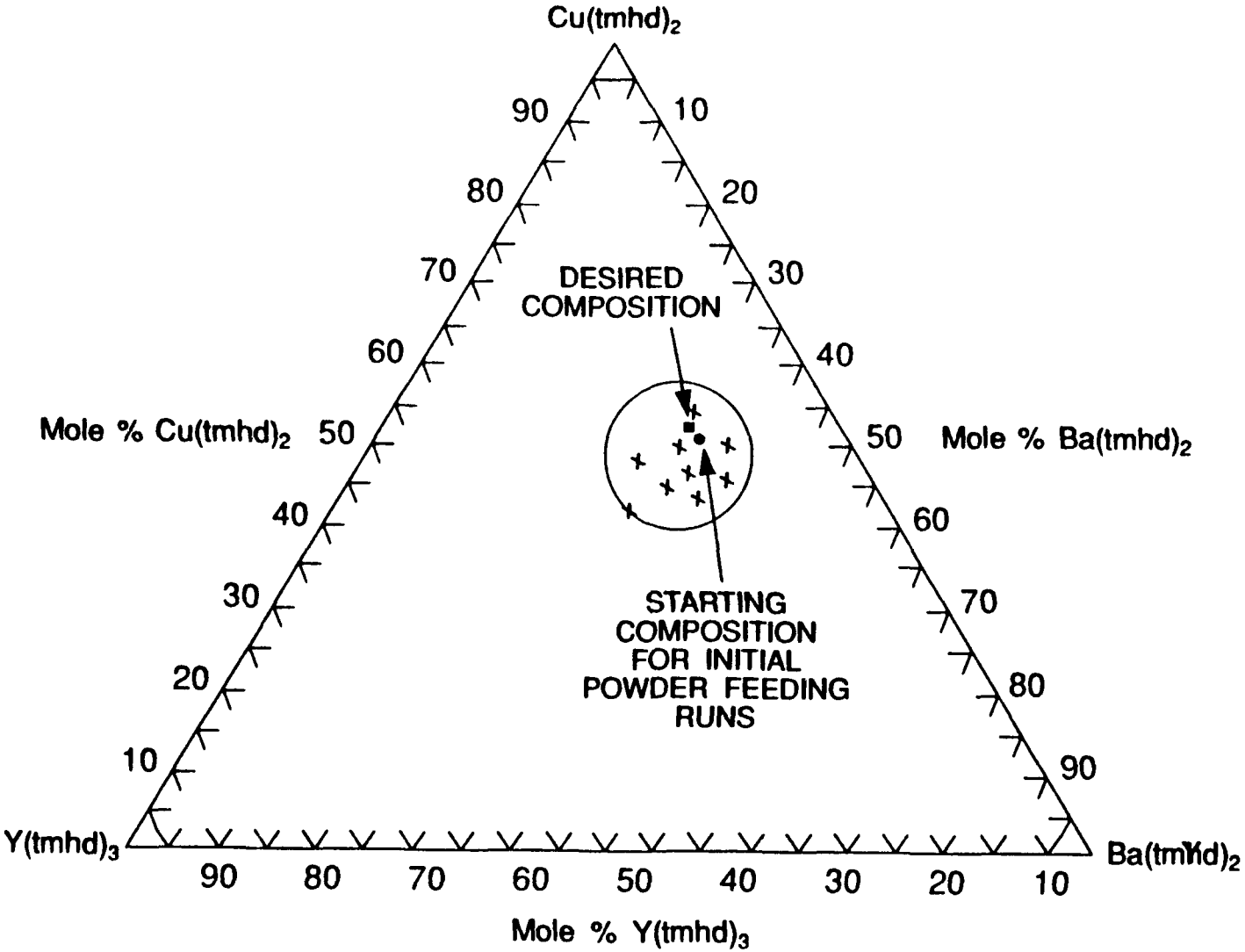


Figure 4-8. Reagent composition diagram showing experimental design for improving film quality. The runs (denoted by X) were analyzed using EDS.

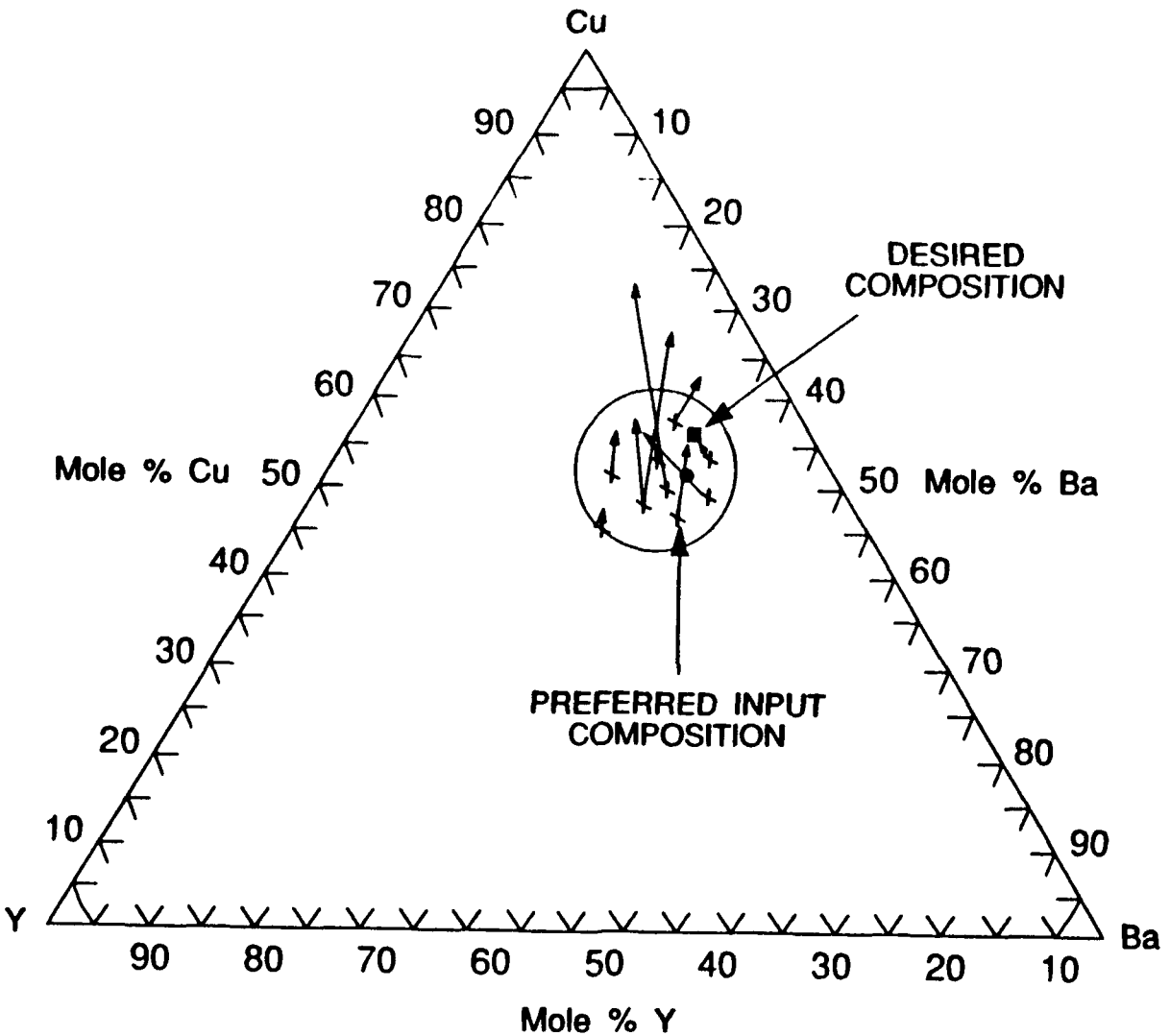


Figure 4-9. Output compositions from input (X) to final (tip of arrow) for the composition study. The desired composition was obtained at the preferred input condition.

Table VII. Summary of the 13 run composition study using the vibratory powder feeder.

Run (Data Point)	Input Mole%	Output Mole %	Resistance (Ω)	MgO wt gain (g)	Powder in/out (g)	Powder Lost (%)	Output Y/Ba	Output Ba/Cu	Output Y/Cu	EDS & X-ray Comments
ST-235(1)	15:2:33.8:51	18:25:57 16:25:58	600-1000				1:1.6	1:2.3	1:3.6	several phases 011, CuO not oriented, very bumpy
ST-236(1)	15:2:33.8:51	11:27:61	450	0.0021	3.62/3.06	84	1:2.4	1:2.2	1:5.5	flat microstructure oriented 123 011 7 CuO also
ST-244(1)	15:2:33.8:51	11:34:54 10:34:55	15-50		4.82/3.79	78.6	1:3.4	1:1.6	1:5.5	junky microstructure - some small plates oriented 123
ST-238(2)	20:32:48	8:17:74	1000 to 1 MX	0.0016	5.07/3.93	77.5	1:2.1	1:4.35	1:9.2	definitely 2 phases unoriented 123 & CuO & 011 & 101 & 211 yicky microstructure
ST-239(3)	25:30:45	7:26:66	300 KX varied	0.0012	5.19/4.80	92	1:3.7	1:2.5	1:9.4	very grainy unoriented 123 (maybe) 011 & 211 & CuO
ST-246(3)	25:30:45	17:25:58 13:26:60		0.0085	5.02/4.92	98	1:2	1:2.3	1:4.6	flat surface - round grains on top variable composition oriented 123 & 211 & CuO
ST-240(4)	15:33:52	16:32:52	80-160	0.0024	6.47/6.21	96	1:2	1:1.6	1:3.25	small grains possibly 2q sample 123 unoriented & 011 & 101
ST-241(5)	20:35:45	11:34:55 10:34:55	45-90	0.0155	5.53/5.15	93.1	1:3.4	1:1.6	1:5.5	very sintered 11 flat 123 oriented
ST-242(6)	20:30:50	11:30:59	40-100	0.0110	5.93/5.03	85	1:2.7	1:1.97	1:5.4	good sintered plate microstructure oriented 123 & CuO
ST-243(7)	15:30:55	10:29:61 9:26:62	40-120	0.0062	8.06/6.66	75.2	1:2.9	1:2.4	1:6.9	some b axis, but plately oriented 123 & CuO
ST-249(8)	15:40:45	15:25:60	80-140	0.0073	5.54/4.43	80	1:1.7	1:2.4	1:4	good sintered microstructure 123 oriented & 211 & CuO
ST-251(9)	25:25:50	variable 22:24:53	70-120	0.0105	6.54/6.49	99.2	1:1.1	1:2.2	1:2.4	very variable composition, sulphur impurity sintered plates, no x-ray
ST-252(10)	30:27:43	41:7:52	5 MX	0.0007	3.78/3.71	98	1:0.17	1:7.4	1:1.26	pebbly microstructure 211 & 101

Table VIII. Processing variables investigated for the $YBa_2Cu_3O_x$ deposition.

	RANGE INVESTIGATED	PREFERRED CONDITIONS
TOTAL PRESSURE (atm)	0.01-1	0.03
DEPOSITION TEMPERATURE (°C)	500-970	900
ARGON FLOW RATE (ℓ/min)	0-5	5
OXYGEN FLOW RATE (ℓ/min)	1-5	1
DEPOSITION TIME (min)	5-55	30
INPUT REAGENT MOLE RATIO $Y(tmhd)_3$:$Ba(tmhd)_2$: $Cu(tmhd)_2$	1:(1.58-2.55): (2.24-3.80)	1:1.75:2.25
TOTAL REAGENT MASS (g)	1-10	1.75

following sections. The conditions determined to give the "best" coating, based on conditions presented in Table VIII, were then duplicated several times. The dashed lines in Figure 4-7 show the resistance versus temperature behavior for the runs completed at the preferred input conditions. The critical temperatures were improved to between 77 and 81 K.

b. Temperature/Pressure

Several studies were completed using this setup in order to understand and develop the coating process. A nine run temperature-pressure study was completed to determine if lower processing temperatures could be used. Three temperature levels (900, 700, 500°C) and three pressure levels (0.04 atm, 0.5 atm, 1 atm) were studied. The rough, nodular microstructure observed for the high pressure and low temperature runs are presented in Figure 4-10. Coatings prepared using these conditions had very high room temperature electrical resistances. Only the high temperature, low pressure runs appeared promising for oriented, smooth deposits. Subsequent annealing of the low temperature films did improve the quality of the deposit, as shown in Figure 4-11. X-ray diffraction patterns obtained before and after annealing the film in argon at 900°C and cooling in oxygen show that annealing increased the degree of c-axis orientation.

c. Deposition/Post Deposition Flowrates and Anneals

During the first few runs using the powder feeder, the post deposition procedure consisted of flowing the oxygen for 30 minutes under vacuum followed by a cooldown with 1 ℓ/min oxygen flowing through the furnace. During this development period, several other

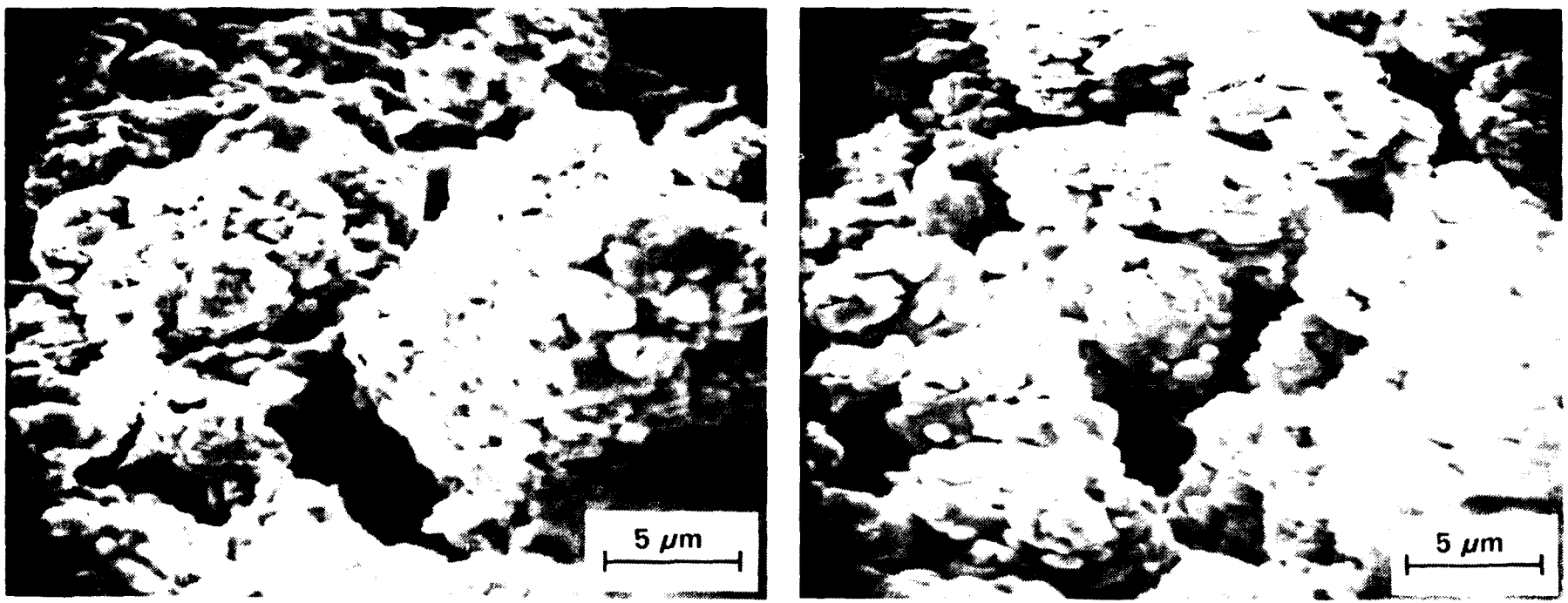


Figure 4-10. Rough surface morphology was observed for low temperature, high pressure runs.

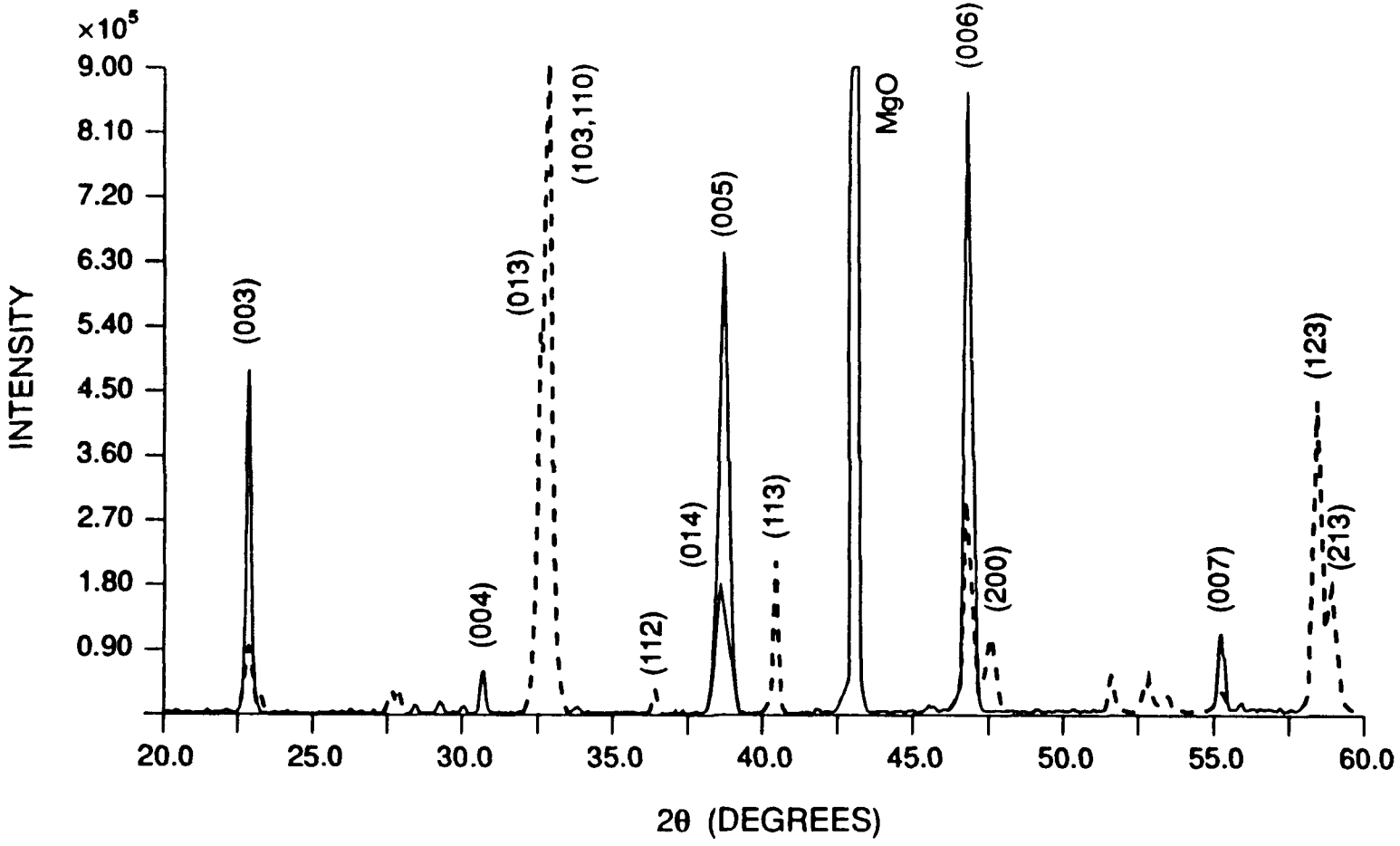


Figure 4-11. X-ray diffraction pattern before (dashed) and after (solid) a 900°C anneal of a low temperature, low pressure deposition experiment. The anneal was performed in argon followed by a slow cool in oxygen.

researchers presented evidence that the annealing step was crucial in the final film properties[60]. Oxygen was required to induce the tetragonal to orthorhombic phase transition which can occur during cooling. The orthorhombic structure is required to attain the ultimate superconducting properties of the material. A study which varied the post deposition procedure for the process was completed. This involved backfilling with either argon or oxygen followed by cooling in oxygen. The 30 minute high temperature anneal was determined to be unnecessary; no change in film properties were noticed with or without the anneal.

The use of argon in the backfill was used to try to melt the film prior to cooling. Argon was reported to reduce the melting point of the $YBa_2Cu_3O_x$ compound to below 900°C[10], which was close to the processing temperature. The analysis was completed using SEM and XRD; no noticeable melting was observed to occur repeatably in the samples; however, the electrical properties of the films with oxygen backfilling were improved over those prepared with the argon backfill. Studies completed using a second annealing furnace where samples were characterized prior to and following the 900°C argon anneal were successfully in verified the melting of the $YBa_2Cu_3O_x$ film.

The amount of reactant gases, typically 5 ℓ/min argon and 1 ℓ/min oxygen were varied to a ratio of 500 sccm argon and 500 sccm oxygen in order to reduce the system pressure and determine the effect of gas composition on coating quality. The reduction of gas flowrates (mainly argon) did lower the pressure of the system to 0.02 atm, but did not repeatably improve the film quality. The

films deposited using the lower gas flow rates had less coating as expected, but were not uniform in appearance across the 1 cm^2 surface area. The EDS analysis indicated a large variation of composition. The powder feeding relied on an efficient feed of the powder to the furnace; reducing the flowrates permitted a large quantity of the reagent to settle in the lines.

d. Additional experiments

The use of several substrates and powder feed rates were investigated. Single crystal PSZ, $SrTiO_3$, and various types of fibers were included in the batch furnace runs. The clearest evidence indicating the CVD nature of the process was determined during these runs. First, the fibers appeared to be uniformly infiltrated (See Figure 4-12); a powder spraying process would only have line-of-sight deposition. Also, five MgO substrates were placed in 2.54 cm intervals along the length of the furnace hot zone. Each sample was uniformly coated and all five were superconducting. This was further evidence that the $YBa_2Cu_3O_x$ coatings were being deposited by a CVD process as opposed to powdered reagents simply impinging onto the substrate.

The deposition rate was also varied by controlling the powder feed rate. A slower feed rate was found to deposit smoother, higher quality films. Both c-axis orientation and critical temperature were enhanced using a slower feed rate. Finally, the use of other, less expensive reagents was investigated. Acetylacetonates, acetates, and liquid feeding were attempted in order to develop an inexpensive deposition process. (See Table V) An x-ray diffraction pattern of a $YBa_2Cu_3O_x$ film deposited using

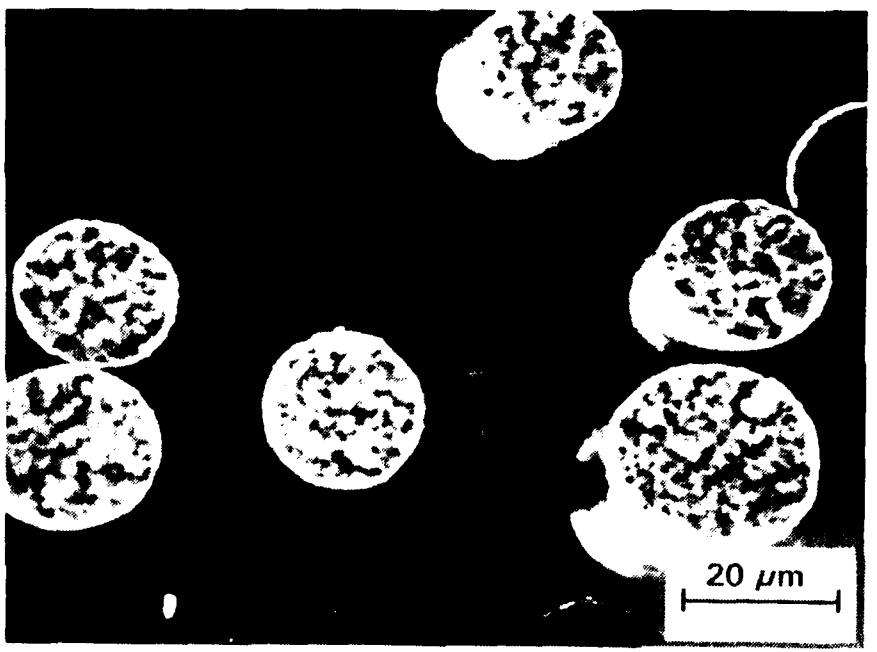


Figure 4-12. DuPont PRD-166 (85% Al_2O_3 + 15% ZrO_2) fiber tow coated uniformly with the $YBa_2Cu_3O_x$ compound.

ACAC reagents is presented in Figure 4-13. The (00ℓ) peaks indicative of c-axis orientation are clearly seen; however a large quantity of impurity peaks are also present. Only a minimal amount of effort was made using these reagents; the TMHD reagents appeared to be more promising for developing high quality coatings in the time available.

Several samples were analyzed using electron microprobe to determine the spatial composition through the coating thickness. Figure 4-14 shows a polarized micrograph cross section of a superconducting $YBa_2Cu_3O_x$ film deposited on MgO. As shown, the surface morphology appeared extremely rough, with several grain orientations and impurity phases present in the film. Microprobe of the film is shown in Figure 4-15 with 9 points described by their Y:Ba:Cu ratios. The film was mainly $YBa_2Cu_3O_x$, with minor 211 and CuO impurities--this result was verified by x-ray diffraction. A TEM micrograph shown in Figure 4-16 also indicated the presence of impurity phases within the $YBa_2Cu_3O_x$ matrix.

e. Pro lems/ solutions

One initial drawback to the powder feeding approach was the pyrophoric nature of the reagent powder. Early runs were completed using only oxygen as a carrier gas; this resulted in some violent explosions both in the powder feeding chamber and in the powder/carrier gas lines. In order to avoid this problem, argon was substituted for the carrier gas and oxygen was added downstream of the feeder. Operating procedures were developed to prevent any possibility for oxygen backflow into the powder feeder and

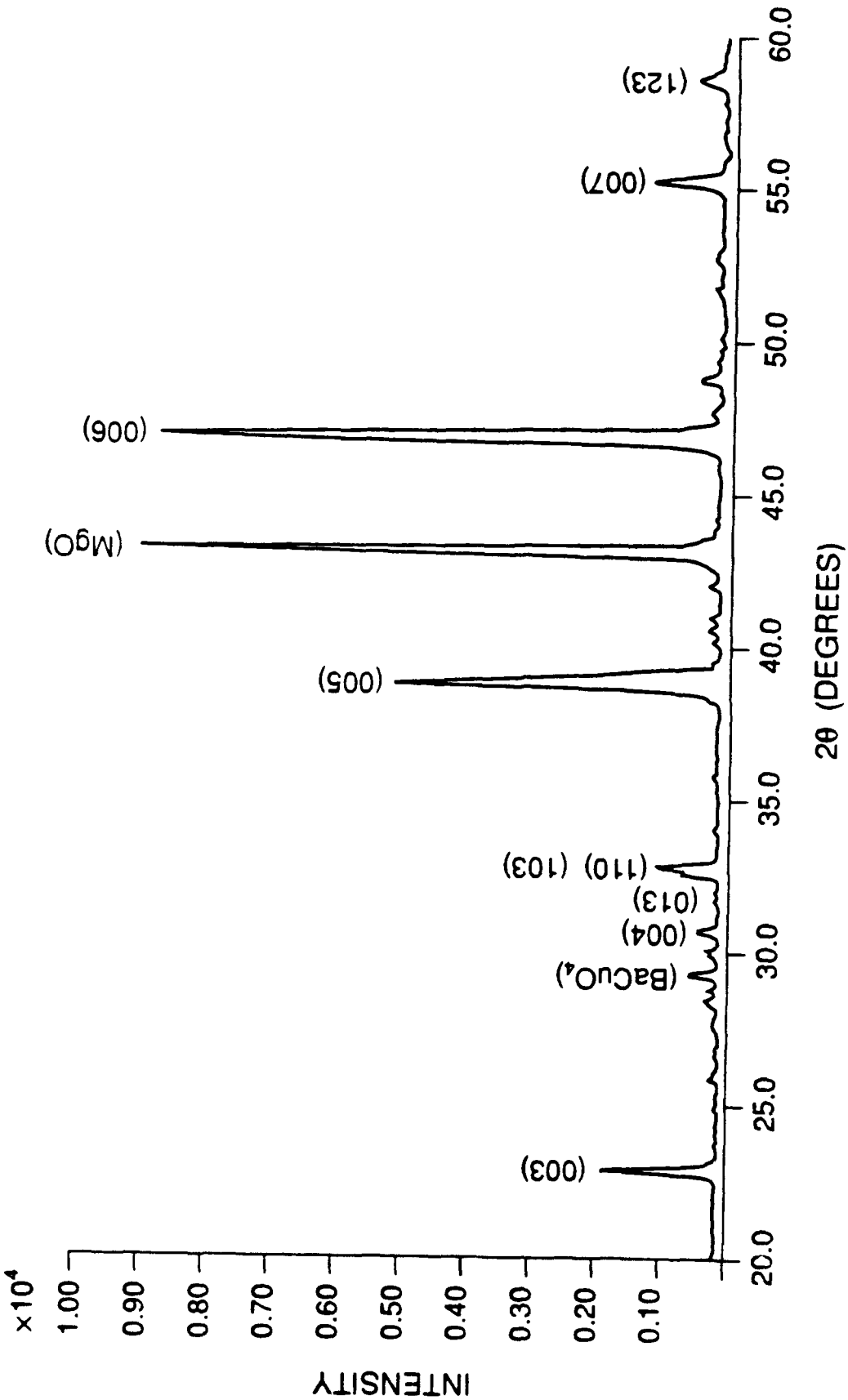


Figure 4-13. X-ray diffraction pattern obtained by depositing a $YBa_2Cu_3O_x$ film with ACAC organometallics.

Figure 4-14. Optical micrograph of the $YBa_2Cu_3O_x$ film cross section indicated a large variation in grain size and orientation.

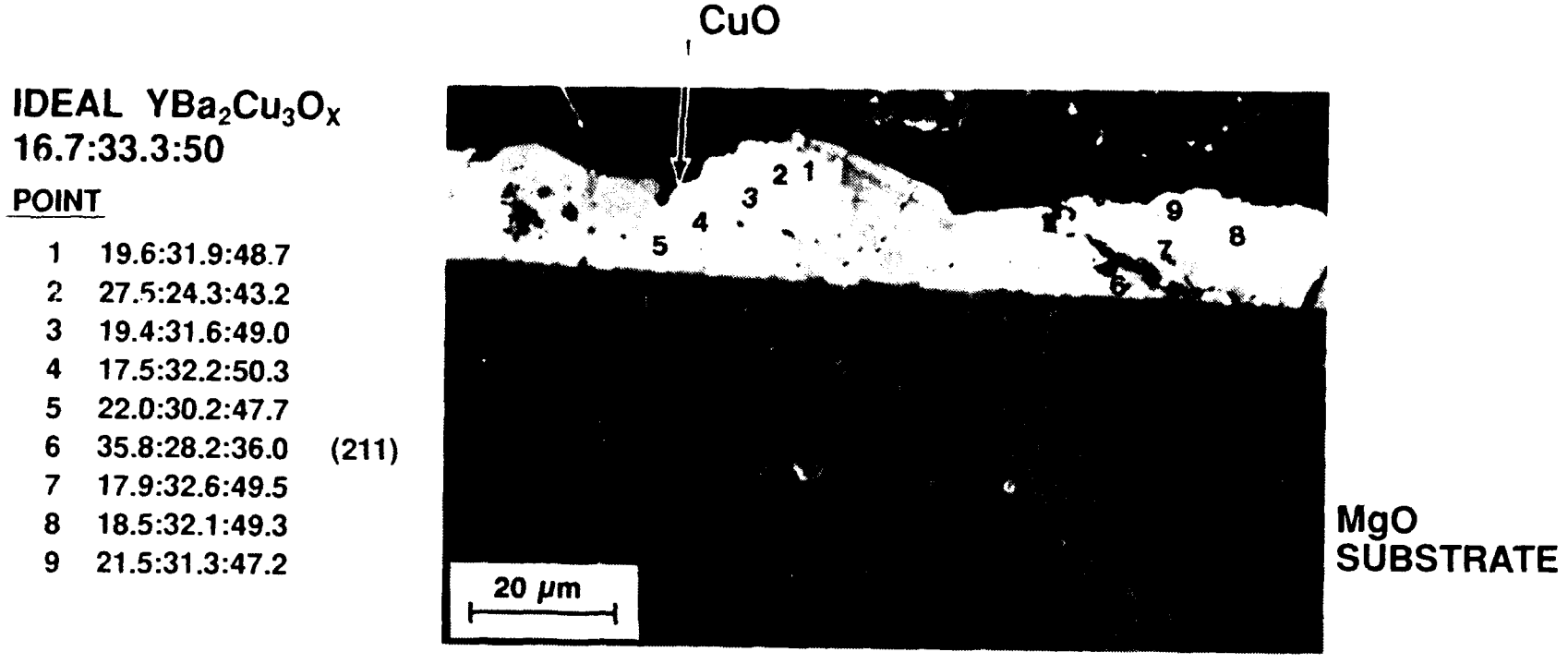


Figure 4-15. Electron microprobe analysis of several points along a $YBa_2Cu_3O_x$ film cross section. The Y:Ba:Cu ratios indicate a fairly uniform composition with some 211 and CuO impurities.

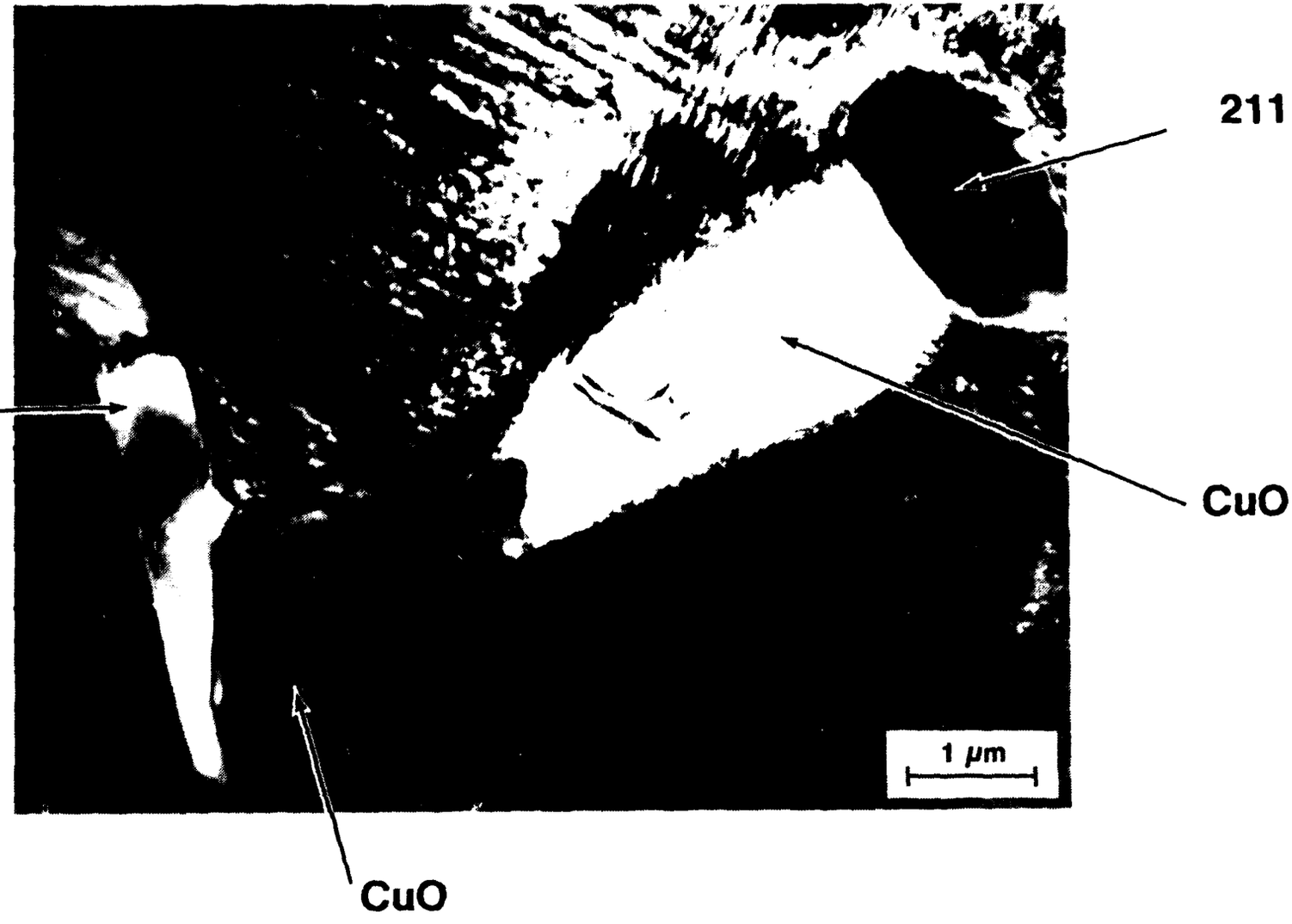


Figure 4-16. TEM analysis was used to identify impurity phases in the $YBa_2Cu_3O_x$ film. CuO and 211 impurities were most commonly identified by x-ray diffraction and TEM.

electrical grounding lines were connected to the stainless steel fixtures in order to eliminate any static charge buildup.

Also, the early runs used a powder/carrier dispersion chamber which was placed between the feeder and the furnace. The chamber was envisioned to permit the trapping of the larger reagent particles, providing a more uniform feed into the reactor. This was removed from the process following an analysis of the input powder feed, the powder caught in the dispersion chamber, and the powder entering the furnace. The powder was determined to segregate (the concentration of the Cu reagent appeared to be reduced entering the furnace). Atomic absorption results indicating the composition variations are presented in Table IX.

Table IX. Atomic absorption analysis (mole %) of input feed powder. The powder was collected at several points in the process.

	Powder in Feeder	Powder in Dispersion Chamber	Powder to Furnace
Y:	3.58-3.68	3.78-3.99	3.72-3.76
Ba:	8.85-10.77	6.01-9.86	9.93-11.25
Cu:	5.89-5.91	6.43-7.09	5.36-5.46

The final problem, and one mentioned previously, was that the powder feed rate was not uniform during the entire run, and depended on the amount of powder in the feeder at a given time. Several potential solutions were suggested, including development of a feed auger, use of multiple powder feeders, or a cyclone dispersion chamber; however, the final decision involved designing

a powder feeding system which would uniformly introduce powder into the system. This system is shown in Figure 3-1; it provided a controllable, uniform delivery of powder into the furnace.

3. Improved Powder Feeder

The feeder shown in Figure 3-1 consists of a teflon plate with powder slots, which slowly rotates over an argon jet. The argon forces the powder through the slots and entrains the material under vacuum conditions in the feed line which enters the furnace. Initially, one large row of powder feed slots were used; however, in order to prevent any unfilled areas caused by friction and drag, a second teflon tray was designed which had two rows of smaller slots, which reduced the drag and kept a continuous powder supply moving into the system. The feeder operated at a slower feed rate as compared to the vibratory feeder, and used typically 2-3 grams of feed. A schematic of the improved powder feeding system design is shown in Figure 4-17. Typical operating conditions were 875°C, 0.04 atm; the films were slow cooled from the deposition temperature in oxygen to 300°C, which normally lasted 2 hours.

Several of the studies completed using the vibratory feeder were duplicated using the improved powder feeder. The powder delivery rate was considered extremely uniform compared to the initial method, and the new feeder also allowed for better control of the total quantity of powder used per run. The first noticeable difference in the films was the thickness; the films were determined to be much thinner using optical and scanning electron

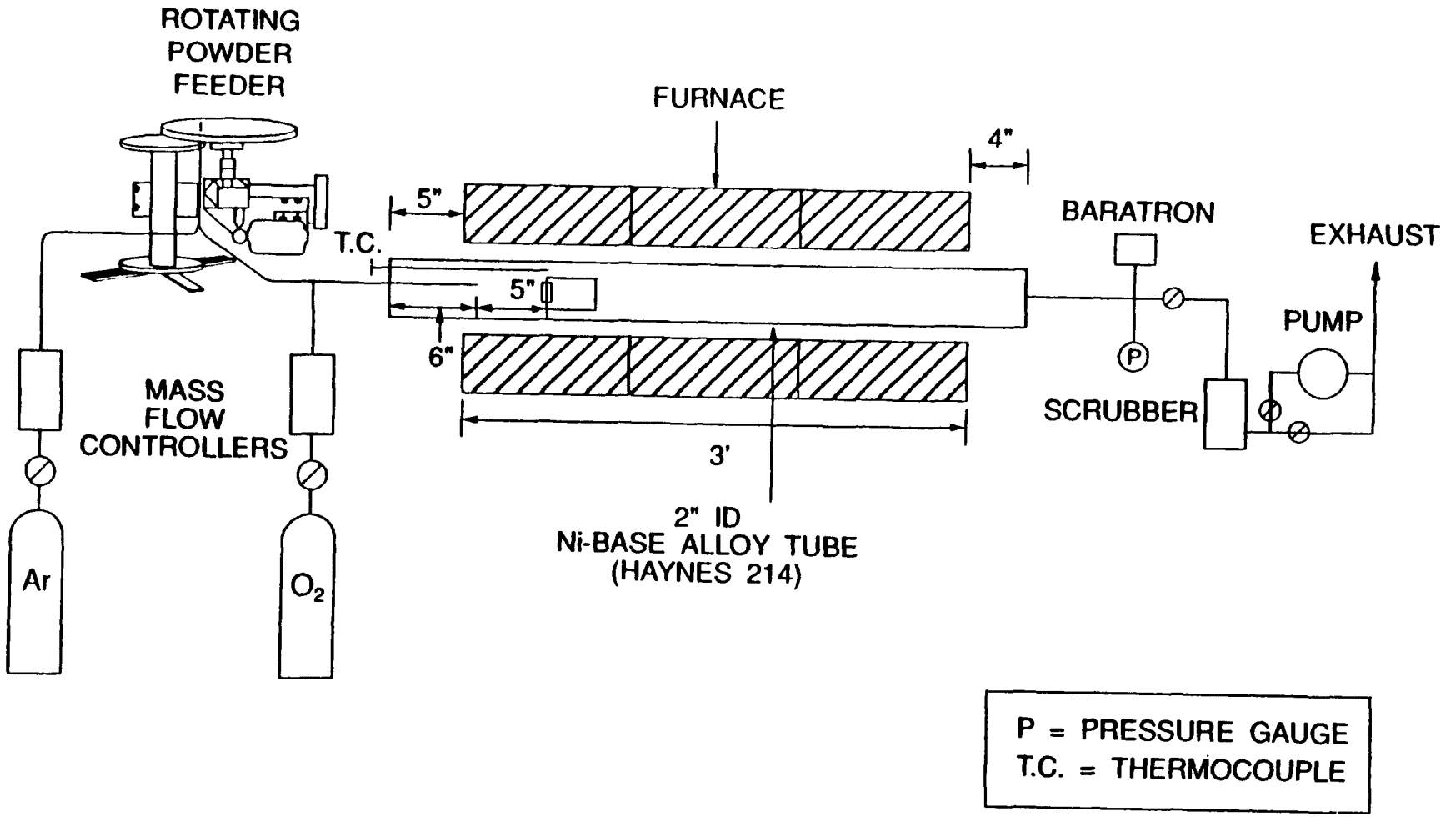


Figure 4-17. Improved powder feeder which resulted in a more uniform powder feed rate. Multiple troughs of powder are impinged upon by an argon jet.

microscopy. The coating appearance was black and gray, and was uniform across the 1 cm^2 area of the substrate.

a. Improving the quality of the $YBa_2Cu_3O_x$

1. Composition Studies

Several composition studies were completed in order to pinpoint the exact starting composition with the improved powder feeder. By depositing a film closer to the ideal $YBa_2Cu_3O_x$ composition, one might expect to reduce or eliminate the CuO and 211 impurities determined by x-ray diffraction and SEM/TEM analyses. The initial compositions were chosen over a narrower range than the earlier vibratory feeder work, and were centered around the preferred conditions for the vibratory feeder shown in Table VIII. Figure 4-18 summarizes one of the studies in which all the films were tested for their critical temperatures. The two dimensional plot is generated by comparing the EDS ratios of Y to Ba, and Cu to Ba, and is analogous to the ternary diagrams presented previously. An ideal composition of $YBa_2Cu_3O_x$ would be at (3,1) on the graph. The electrical properties and/or critical temperatures ranged from semiconducting behavior to 76 K. These studies resulted in changing the initial reagent composition from 1:1.75:2.25 for the previous preferred conditions for the vibratory feeder to 1:1.38:1.51 for the rotating powder feeder. The yttrium content was increased relative to the Ba and Cu content. Subsequent composition studies, which narrowed the input range further, did not reproducably improve the electrical properties of the films. A large variation in the measured critical temperature, from 81 to 40 K, was observed throughout the studies.

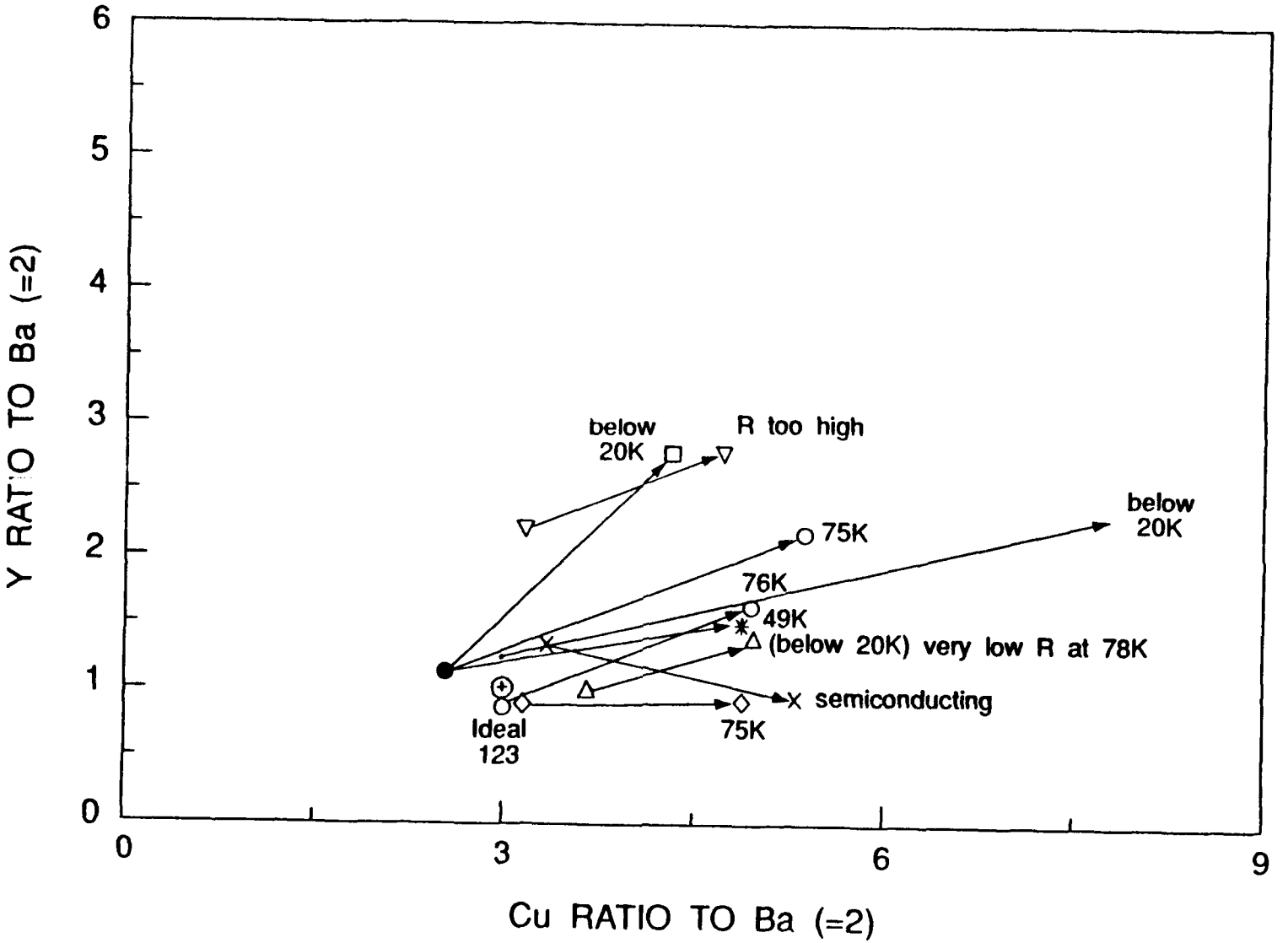


Figure 4-18. Summary (initial to final compsition) for runs completed using the improved powder feeder. The critical temperatures electrical behavior for each film are overlayed on the figure.

2. Parametric Studies

A second attempt to improve film quality was initiated through a 26 run processing study, which encompassed several processing variables including temperature, pressure, annealing schedule, and substrates. The temperature variation was over a narrow range, between 800 and 900°C. The extent of c-axis orientation in the film was shown to decrease as temperature was decreased. Extensive a-axis orientation was observed for films deposited at 800°C as shown in Figure 4-19. The best films, determined by critical current measurements, were obtained at 875°C.

Results for films deposited at lower flowrates were inconclusive. The total gas flowrates were varied from 5 ℓ/min of argon and 1 ℓ/min of oxygen to 1 ℓ/min of argon and 0.5 ℓ/min of oxygen resulting in a pressure variation of 0.04 to 0.01 atm. Two of the five films deposited at the lower pressures had excellent electrical properties; however, the repeatibility and coating uniformity was poor in subsequent runs. The use of a slower feed rate (0.05 g/min) as compared to 0.1 g/min resulted in smoother films, with a grayish/black shiny coating. The results from both x-ray diffraction and critical current measurements were enhanced by slowing down the deposition rate. Fewer impurity peaks (including CuO, 211, and 011) were observed in the films.

A final part of the parametric study was the deposition on different substrate materials. Films deposited on polished and acid washed MgO substrates were not improved over the typically used unpolished MgO material. Limited work with $SrTiO_3$, reported as an excellent substrate material for $YBa_2Cu_3O_x$ deposition, also

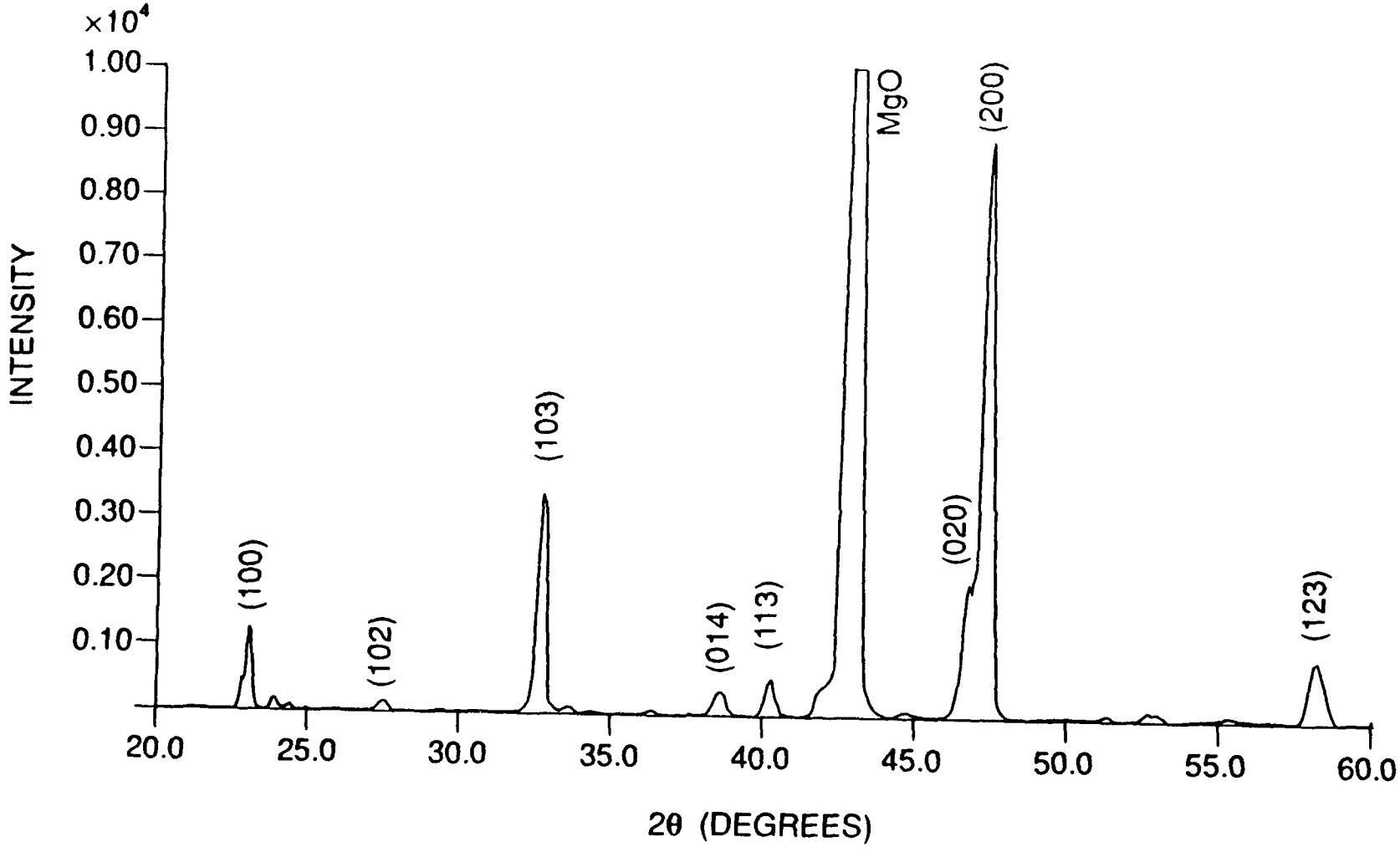


Figure 4 19. The $YBa_2Cu_3O_x$ film deposited at 800°C exhibited extensive a-axis orientation.

did not result in a high quality, oriented film. The best sample deposited during the study was on single crystal partially stabilized zirconia (PSZ). The x-ray intensity was extremely high ($>10^5$) as compared to our normal pattern on MgO (See Figure 4-5), and there were few impurity peaks, as shown in Figure 4-20.

3. Additional Substrates

In order to study and further improve the $YBa_2Cu_3O_x$ film, depositions were completed on unique, newly developed single crystal substrates including $LaAlO_3$ and $LaGaO_3$. The highest critical temperature achieved in this study occurred on a $LaAlO_3$ substrate. Measurements which verificated the 91 K transition temperature (Figure 4-21) were performed at the Oak Ridge National Laboratory Superconducting Pilot Center. Surface resistance measurements completed at the Los Alamos National Laboratory were considered encouraging, and prompted further studies.

The film was highly textured as shown in Figure 4-22. Optical micrographs of the surface were used to identify surface impurities and grain orientations. Figure 4-23 shows a transition region between textured and non-textured $YBa_2Cu_3O_x$ growth on the $LaAlO_3$ sample. Results with $LaGaO_3$ and $SrLaAlO_3$ substrates were less promising, due to the large thermal expansion mismatch between the film and substrate. Cracking of the coating was observed in several of the deposited films; this defect might be avoided with thin coatings. Thinner coatings might possibly be uncracked.

b. Annealing/ Interaction Studies

1. Film/Substrate Interaction

Several runs were completed in order to prepare samples

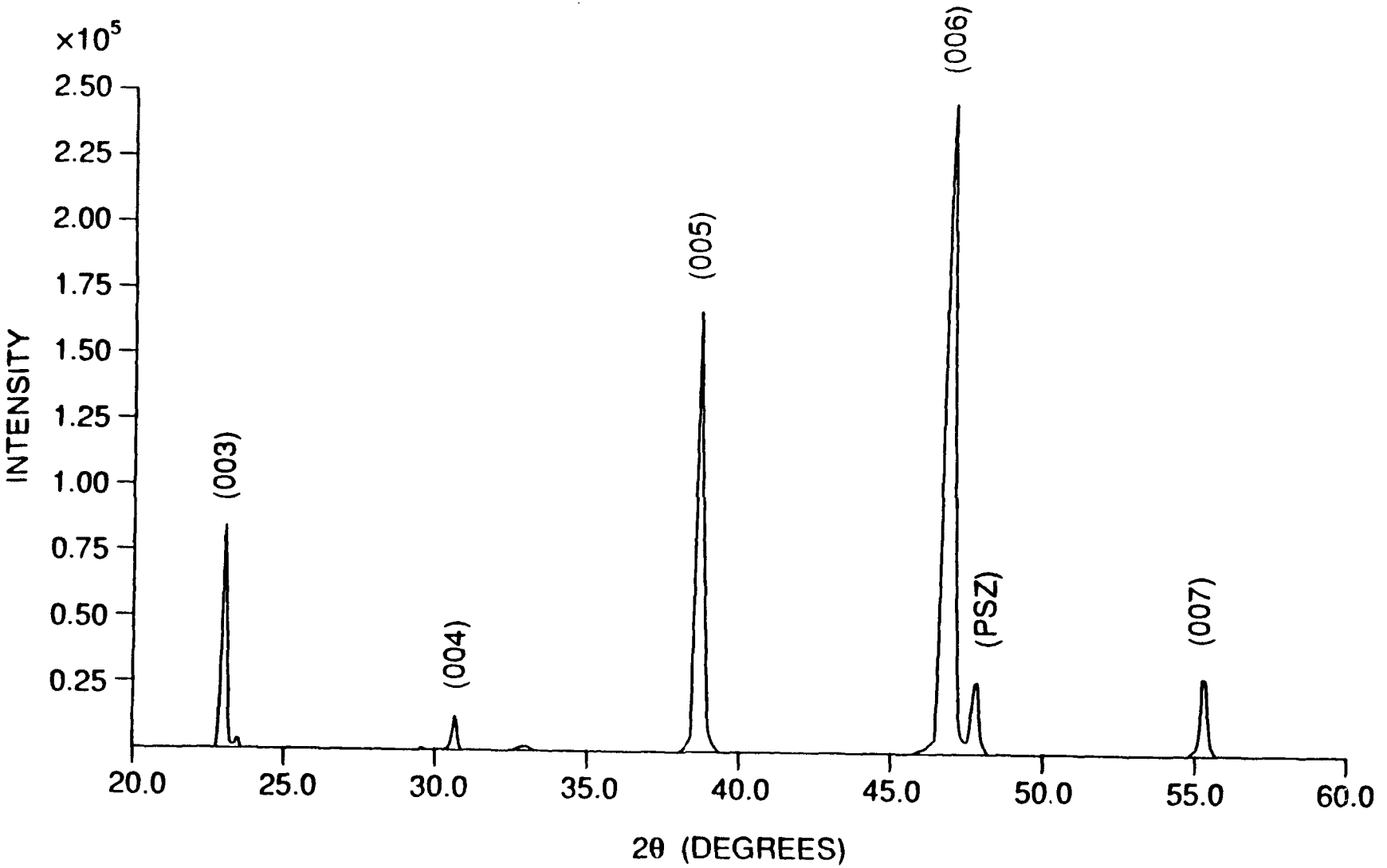


Figure 4-20. X-ray diffraction pattern for a $YBa_2Cu_3O_x$ film on partially stabilized zirconia.

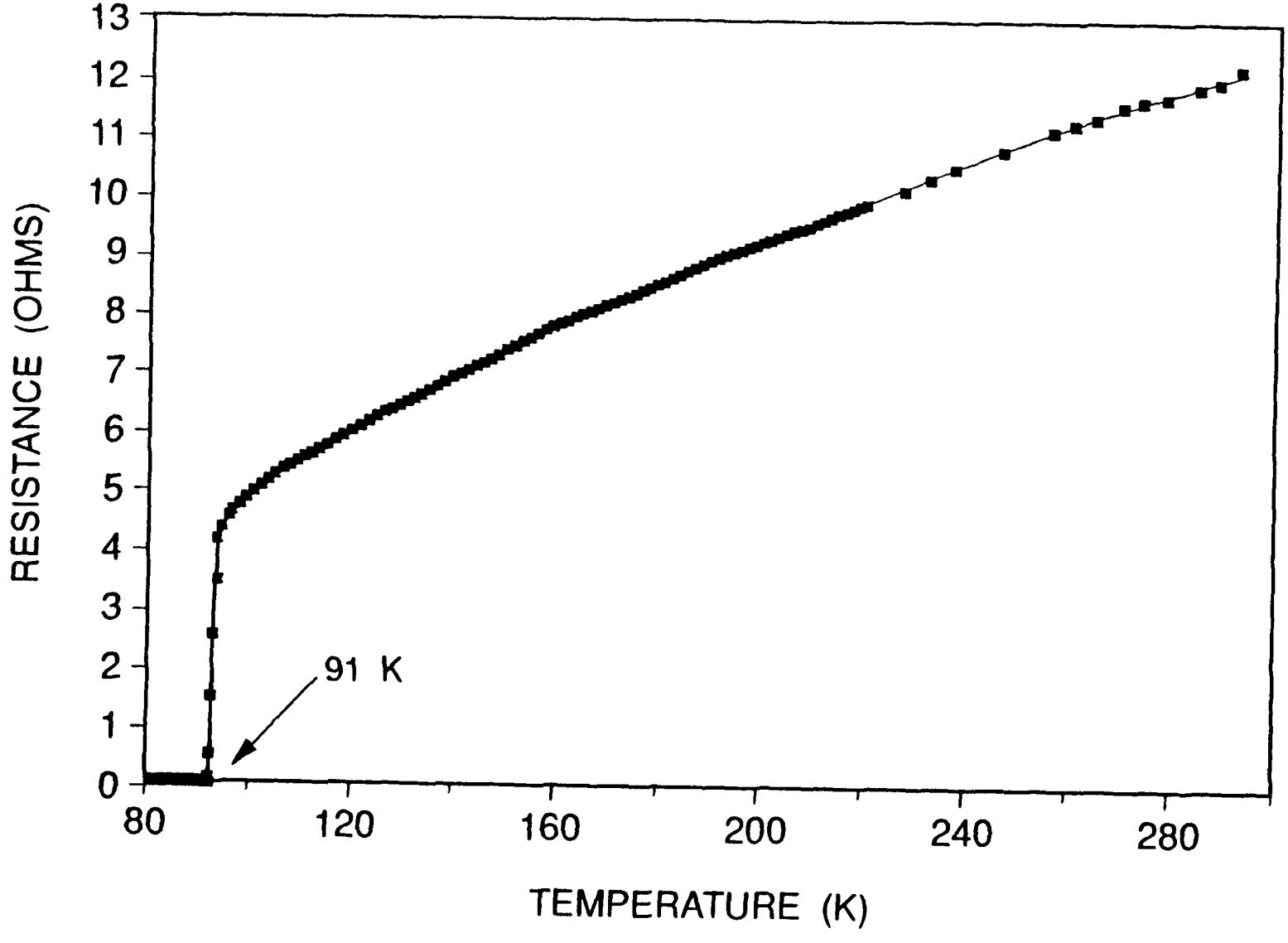


Figure 4-21. Resistance versus temperature curve for a $YBa_2Cu_3O_x$ film deposited on single crystal $LaAlO_3$. This film displayed the highest critical temperature ($T_c = 91K$) observed during this program.

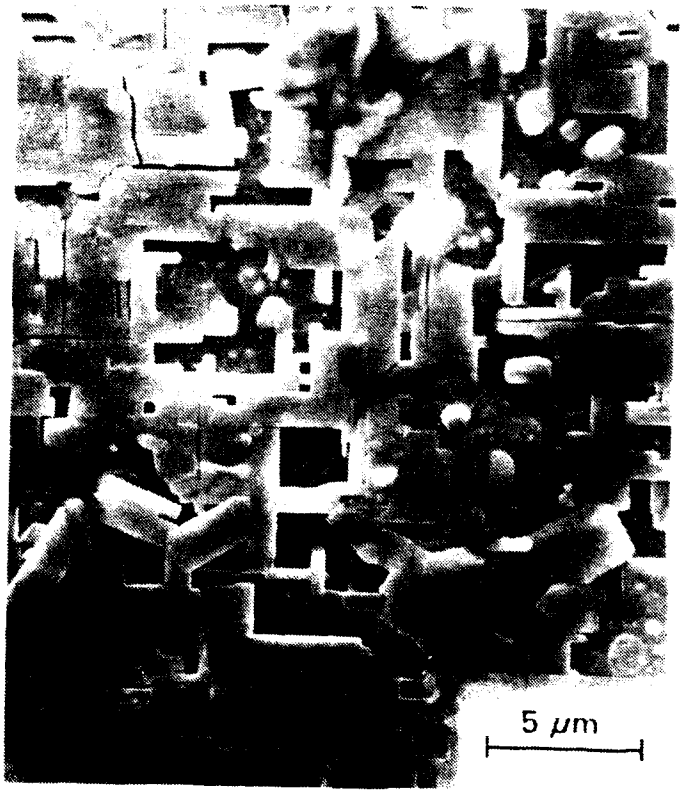


Figure 4-22. A highly textured $YBa_2Cu_3O_x$ film morphology was observed for deposits on $LaAlO_3$.

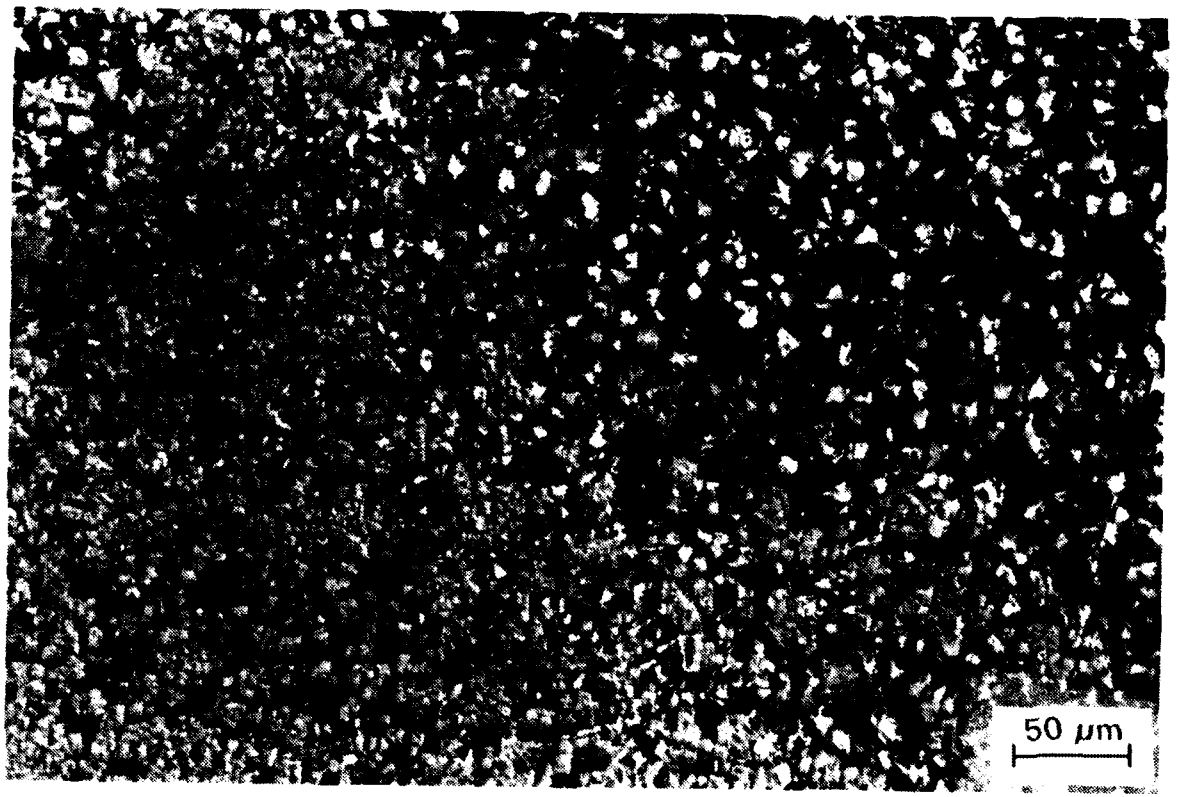


Figure 4-23. Optical microscopy of the $YBa_2Cu_3O_x$ film surface showed regions of textured and nontextured film growth. The more uniform contrast of the left portion of the micrograph indicates greater texturing.

for film/substrate interaction studies. Films were deposited onto MgO, partially stabilized zirconia, and alumina in order to determine the extent of substrate interaction in order to help guide fiber selection. As shown in Table IV, nearly all the commercially available fibers contain alumina or silica, which are known to react adversely with the $YBa_2Cu_3O_x$ compound. X-ray diffraction, EDS, and TEM were used to characterize the films. Figure 4-24 shows a diffraction pattern for a film deposited on polycrystalline alumina. The formation of $BaAl_2O_4$ is evident, indicating the reaction between the substrate and coating. A TEM micrograph is shown in Figure 4-25. Again, a large $BaAl_2O_4$ reaction layer was observed and verified by EDS; also, large CuO and 211 grains were present in the film along with $YBa_2Cu_3O_x$.

TEM micrographs of $YBa_2Cu_3O_x$ coatings on MgO single crystal substrates did not shown any interaction layer. A polycrystalline $YBa_2Cu_3O_x$ coating was observed near the substrate surface followed by c-axis oriented growth as shown in Figure 4-26. An electron diffractogram (inset) indicated the extent of c-axis orientation.

Finally the $YBa_2Cu_3O_x$ film deposited on partially stabilized zirconia resulted in a unique interaction layer[65]. TEM micrographs showed the formation of a $BaZrO_3$ layer at the substrate/coating interface. This 10 nm layer (shown in Figure 4-27) was fairly rough at the substrate side, and extremely smooth on the coating side. This sharp interface indicated that barium was diffusing from the coating into the substrate, as opposed to zirconium diffusing into the coating; however, EDS showed the presence of Zr throughout the film. Following the 10 nm layer, a c-axis oriented

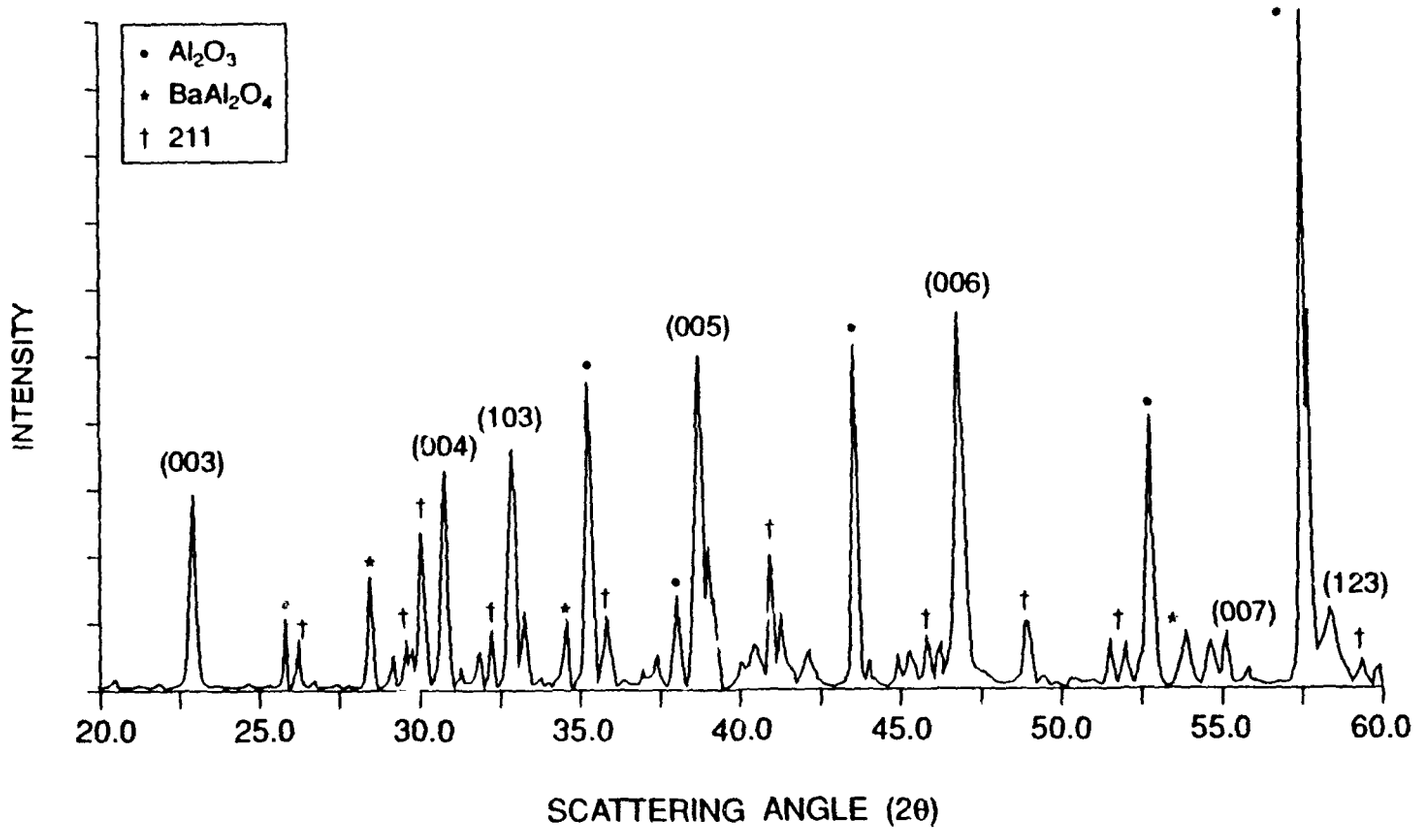


Figure 4-24. X-ray diffraction scan of as-deposited $YBa_2Cu_3O_x$ film on a polycrystalline alumina substrate. A large amount of $BaAl_2O_4$ was observed in the film.

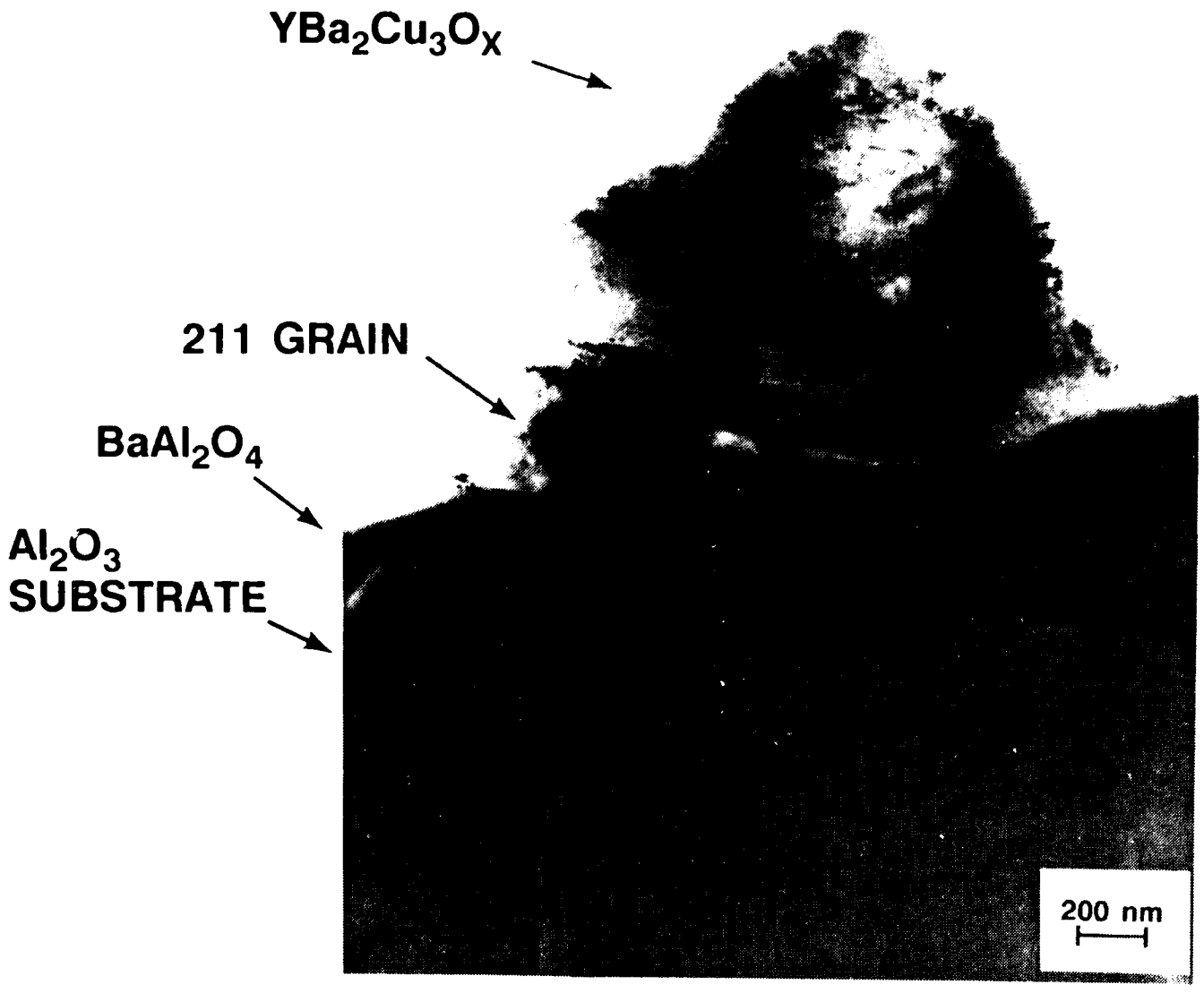


Figure 4--25. Micrograph showing the $BaAl_2O_4$ and 211 impurities in a $YBa_2Cu_3O_x$ deposited on polycrystalline alumina.

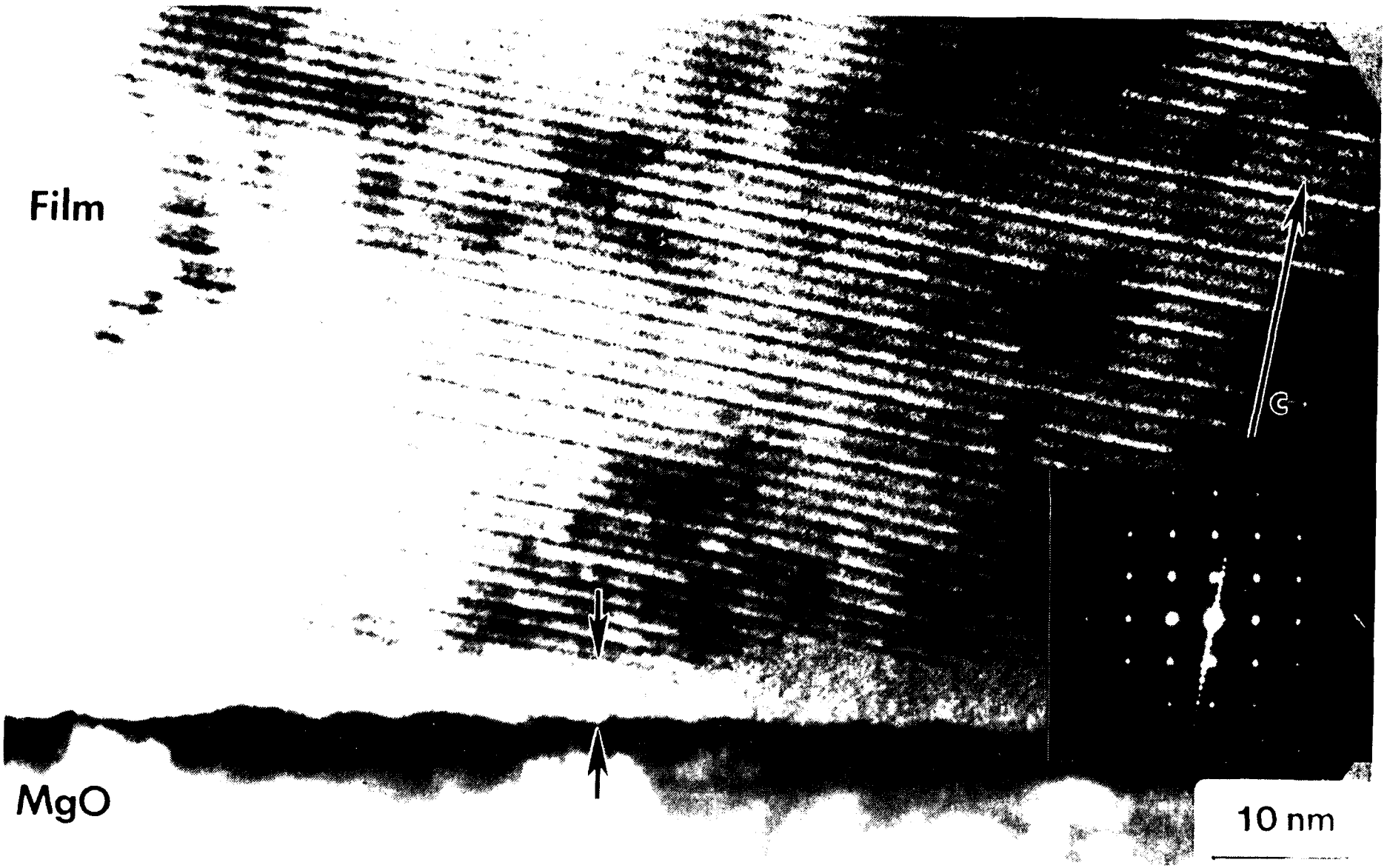


Figure 4-26. TEM analyses indicated a 10 nm polycrystalline layer of $YBa_2Cu_3O_x$ on MgO followed by c-axis oriented growth. The electron diffractogram (inset) shows the c-axis orientation.

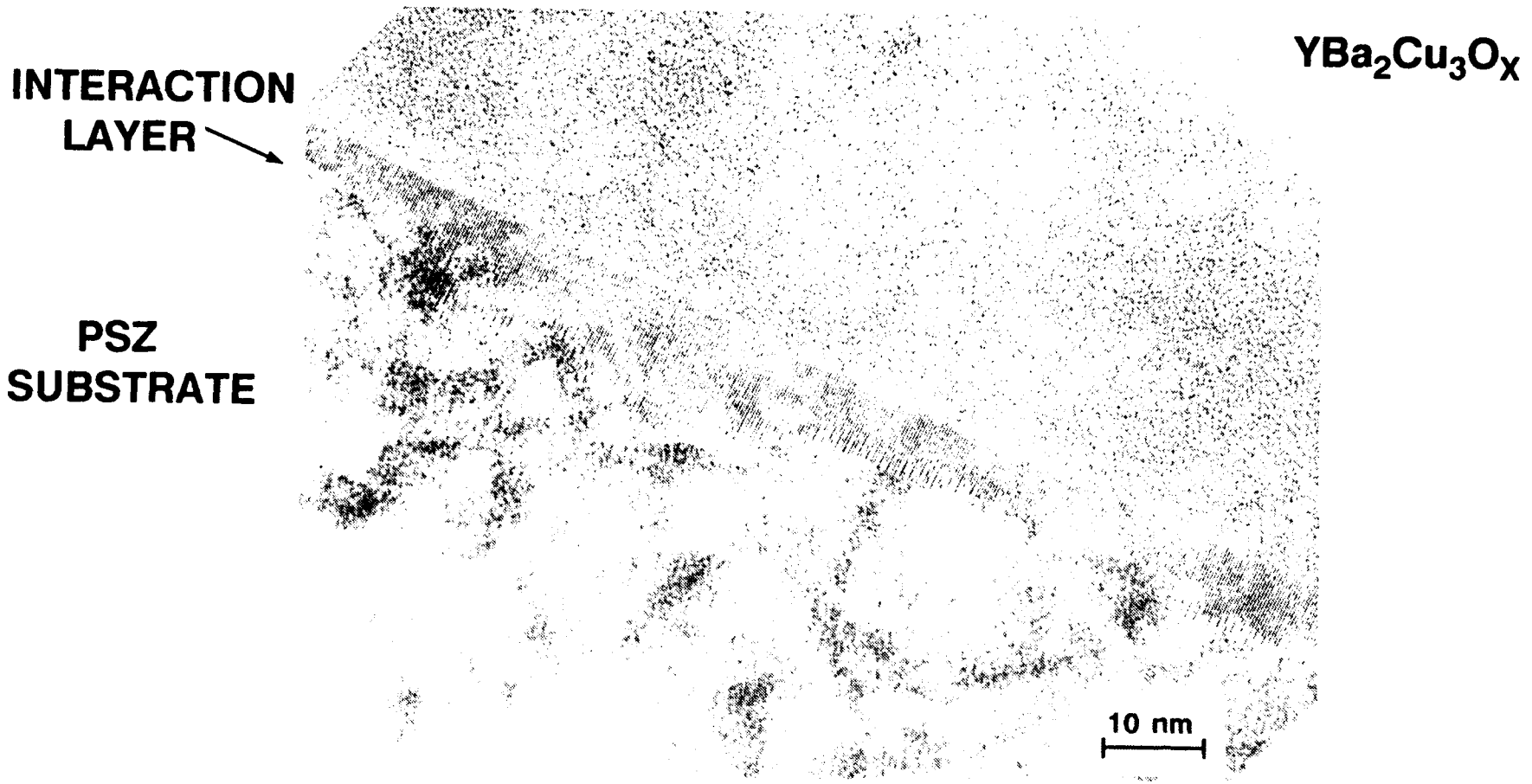


Figure 4-27. A $BaZrO_3$ reaction layer was observed on a $YBa_2Cu_3O_x$ film deposited on single crystal partially stabilized zirconia. The film side appears very smooth whereas the substrate side is rough, indicating Ba diffusion into the substrate.

deposit was observed and verified by x-ray diffraction. Optical diffractograms (inset in Figure 4-28) verified that the $BaZrO_3$ interlayer was epitaxial to the single crystal PSZ substrate. The film had large columnar grains as shown in Figure 4-29, and electrical characterization indicated a critical temperature of 86 K as shown in Figure 4-30. Critical current measurements in liquid nitrogen showed a critical current density of 3.6×10^4 A/cm^2 as presented in Figure 4-31. Both the raw data, and an empirical curve fit are presented for the PSZ sample.

2. Pole Figure Analyses

A second technique used to study the substrate/film interaction and to determine the preferred orientation was to analyze pole figures for several single and polycrystalline samples[88]. Figure 4-32 shows an (006) pole figure for an as-deposited $YBa_2Cu_3O_x$ film on MgO. As shown by the intensity versus Chi plot, the majority of the c-axis of the film is oriented within 5° of the perpendicular of the substrate surface. The affects of annealing were also studied; Figure 4-33 indicates the change in the (006) pole figure by annealing a coated sapphire substrate at 900°C in argon. X-ray diffraction indicated that $BaAl_2O_4$ was forming during the annealing process. Less orientation was evident for films deposited on the polycrystalline substrates; 211 and $BaAl_2O_4$ impurities were observed in as-deposited films on polycrystalline alumina.

3. Silver deposition

In order to circumvent the problem associated with fiber/substrate interactions, silver films deposited by

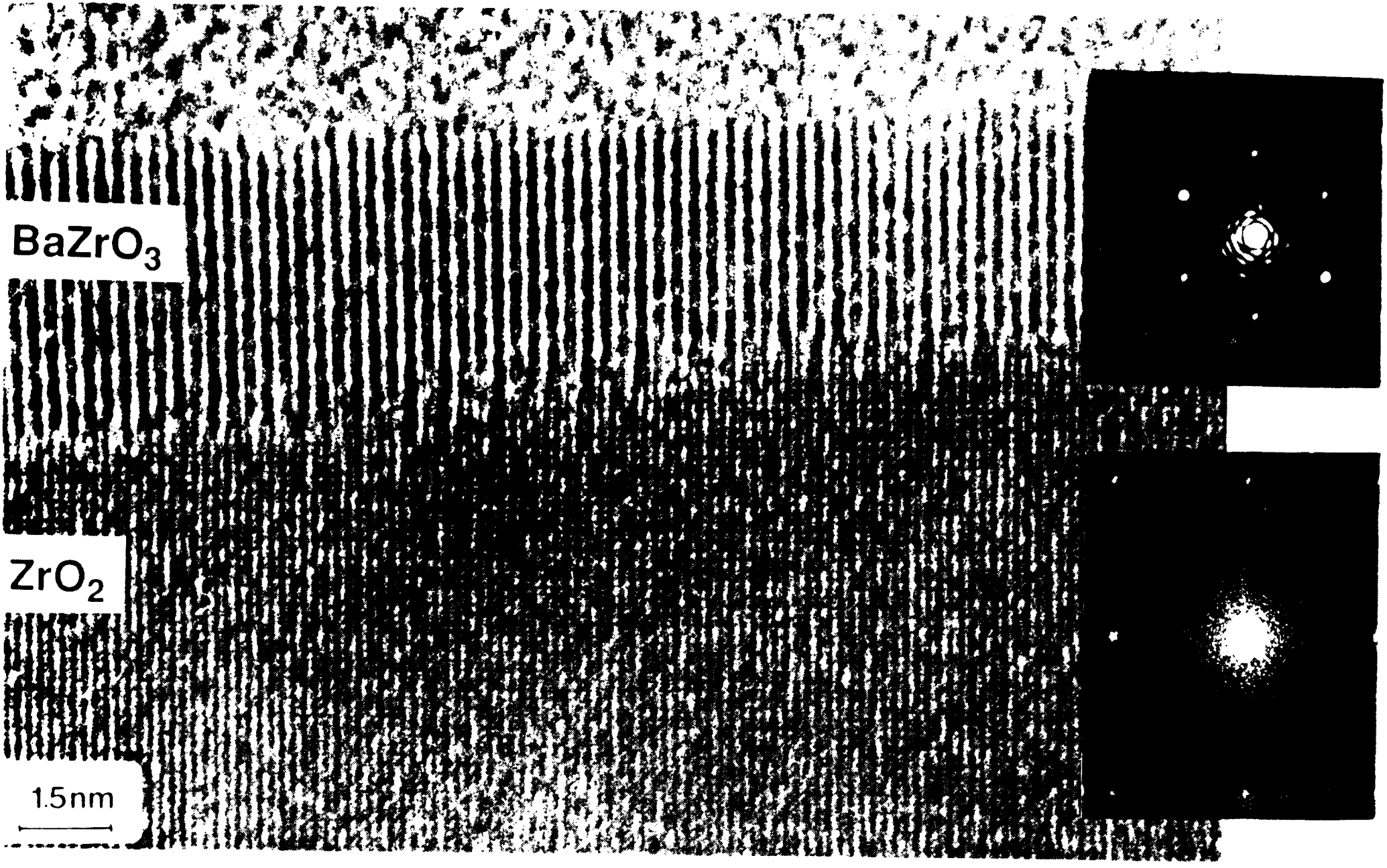


Figure 4-28. Electron diffractograms (inset) were used to verify the phases, including the $BaZrO_3$ interlayer.

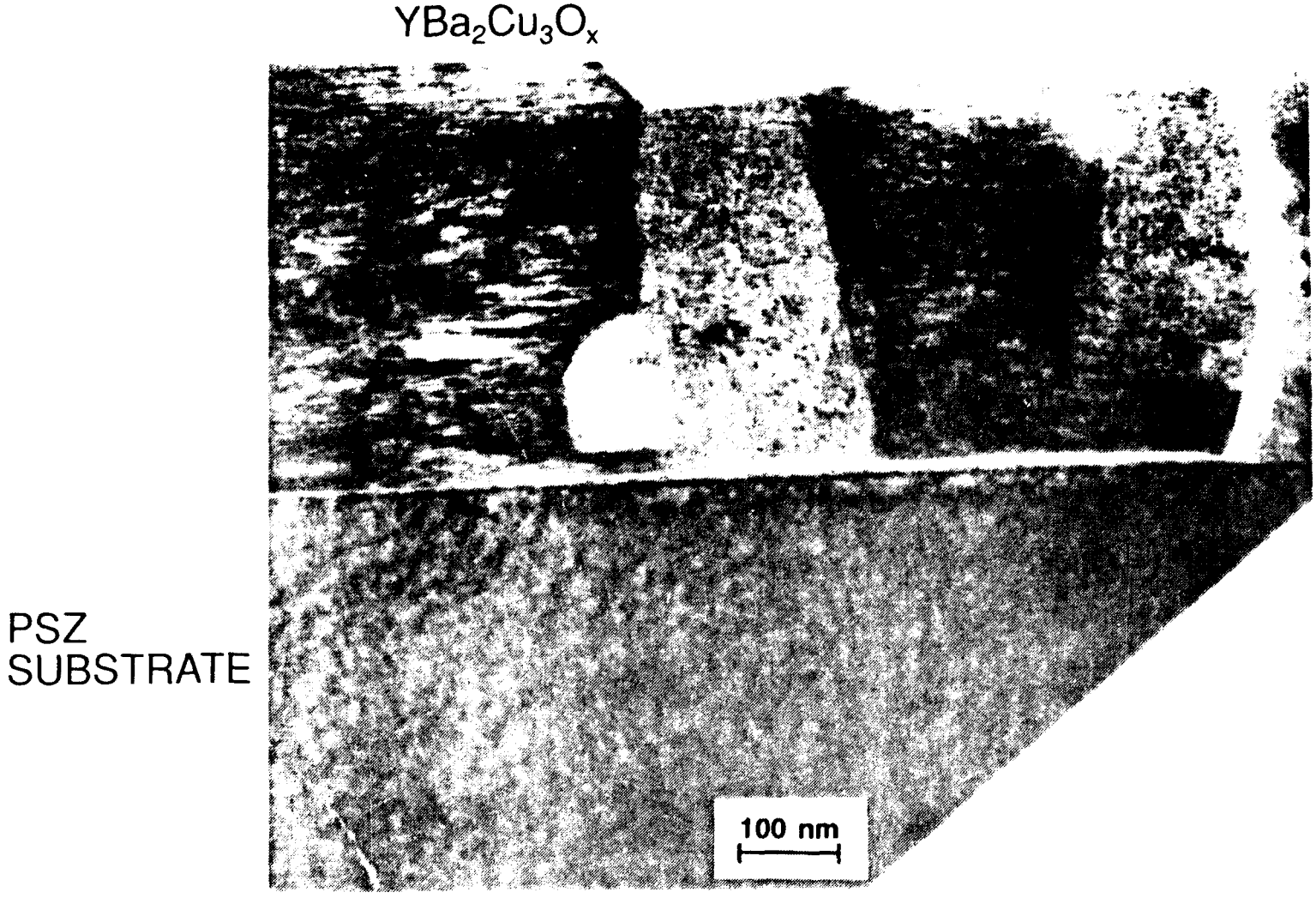


Figure 4-29. $YBa_2Cu_3O_x$ films prepared on single crystal partially stabilized zirconia had large columnar grains.

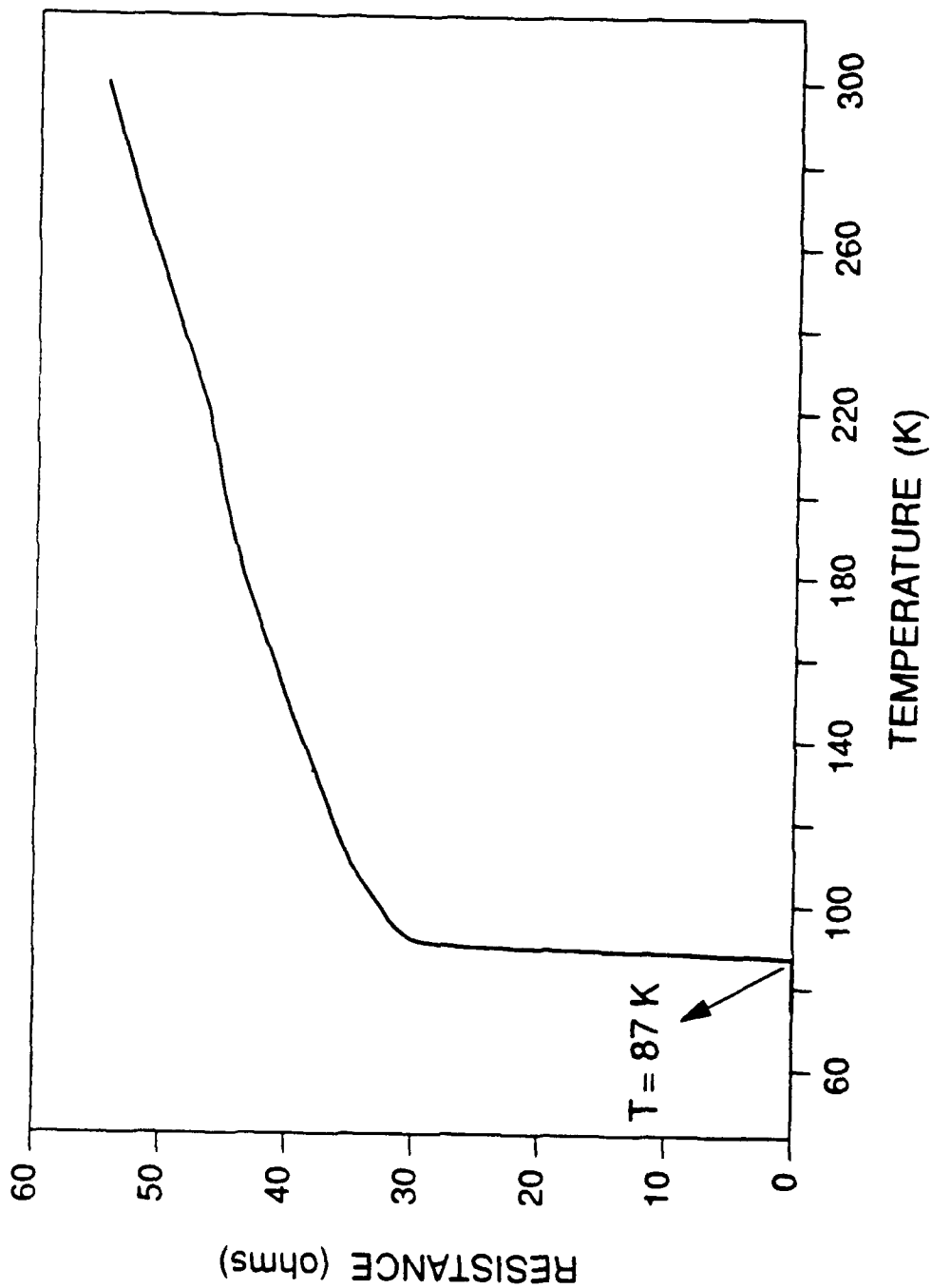


Figure 4-30. Resistance versus temperature measurement for a $YBa_2Cu_3O_x$ coating on single crystal partially stabilized shows a critical temperature of 87 K.

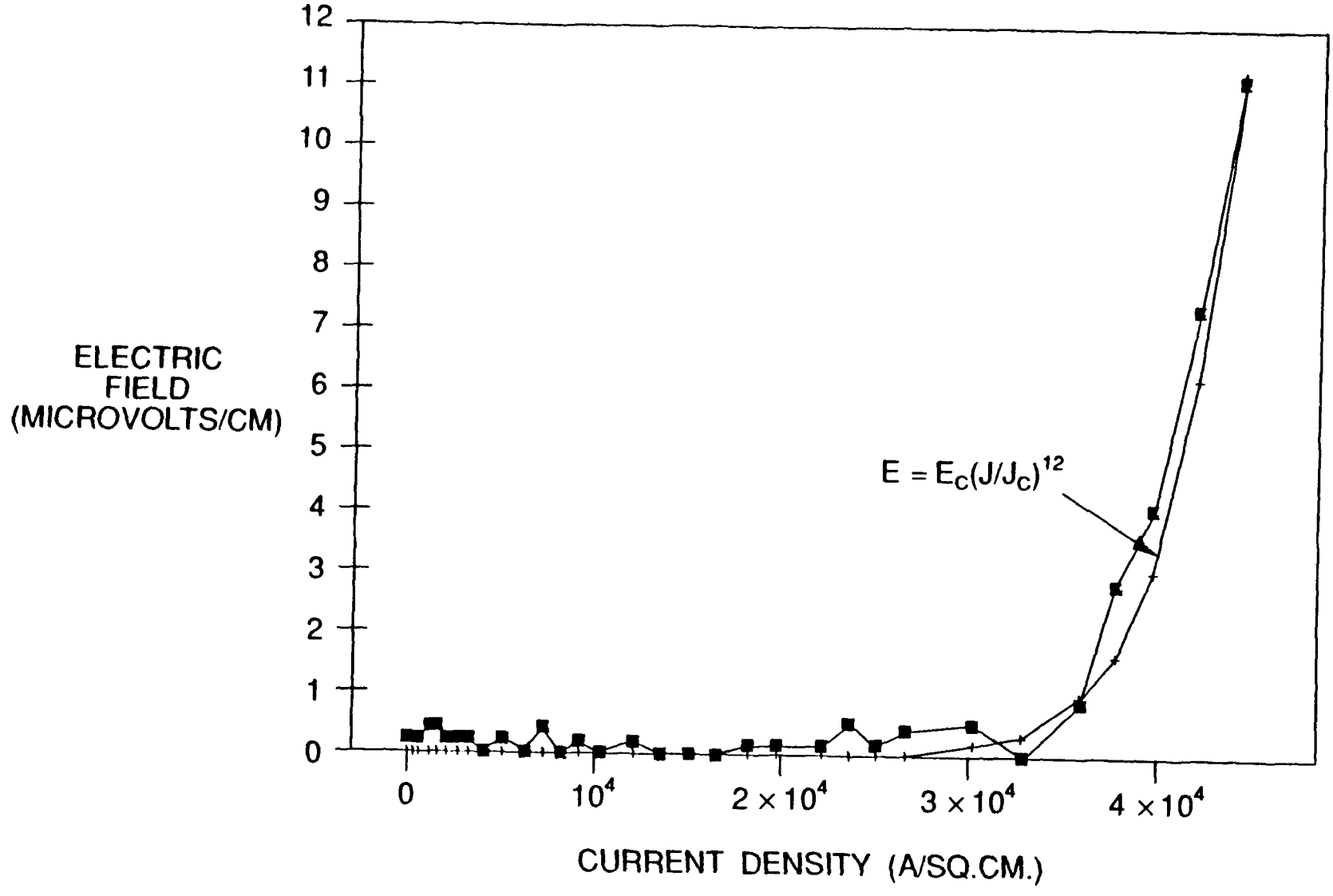


Figure 4-31. Electric field (E) versus current density (J) of a $YBa_2Cu_3O_x$ film on single crystal stabilized zirconia. The crosses indicate a curve fit to the data where $E_c = 1\ \mu V/cm$ and $J_c = 3.6 \times 10^4\ A/cm^2$. The squares represent the actual data.

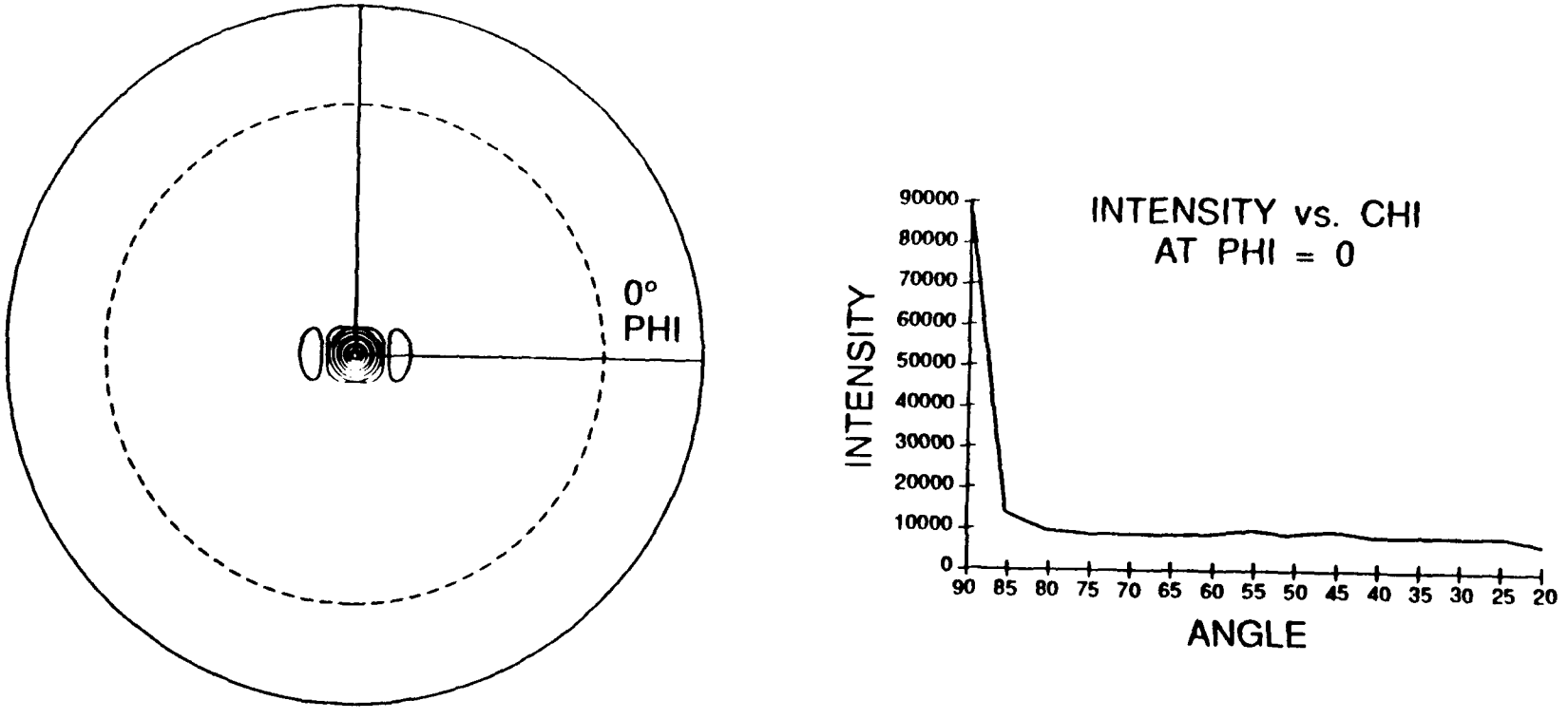


Figure 4-32. An (006) pole figure for $YBa_2Cu_3O_x$ coated MgO shows that most of the c-axis planes are within 5° of the substrate surface.

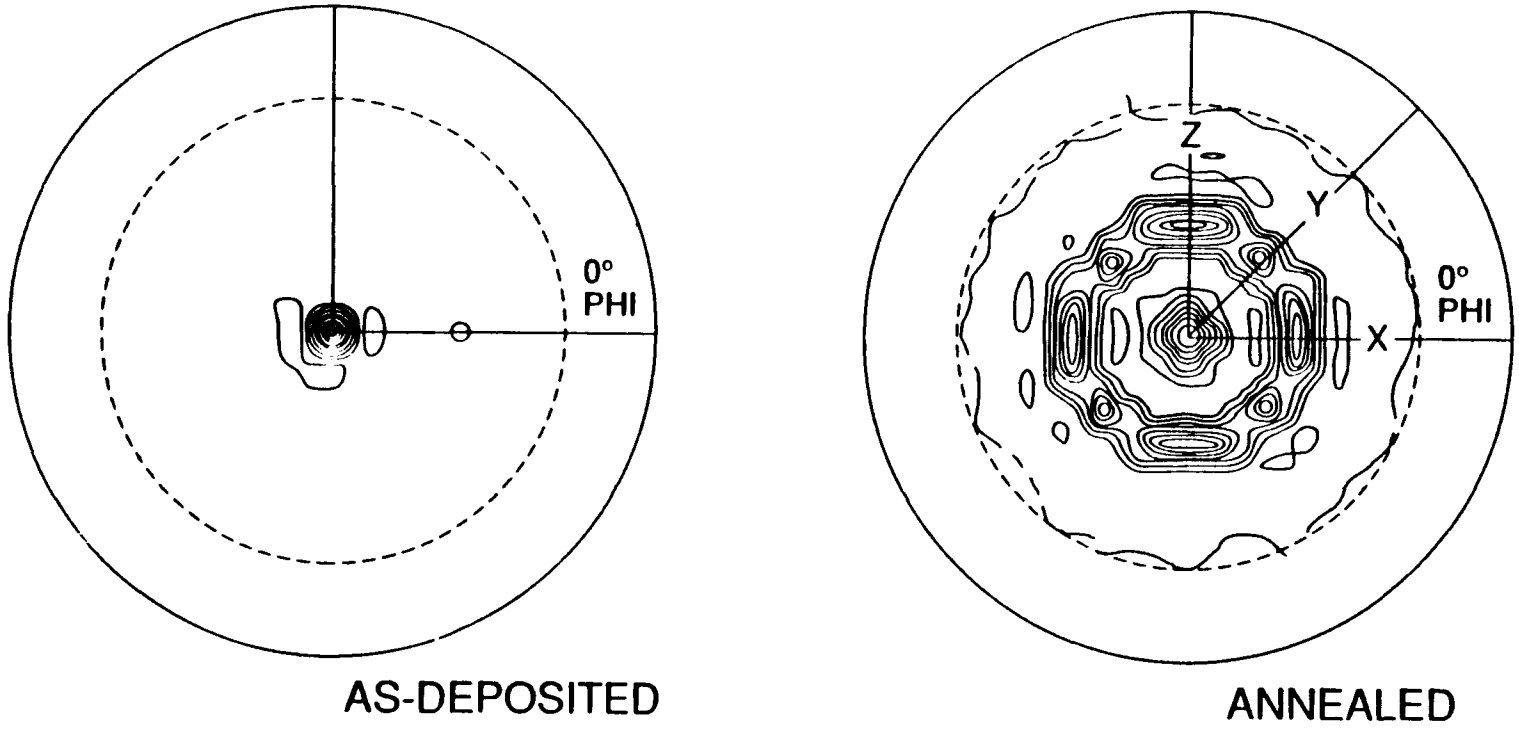


Figure 4-33. An (006) pole figure for a $YBa_2Cu_3O_x$ coating on sapphire. As-deposited and annealed (900°C in argon) analyses show the large change due to the formation of $BaAl_2O_4$.

metalorganic chemical vapor deposition were studied[92]. Earlier results in depositing $YBa_2Cu_3O_x$ on silver substrates showed a c-axis oriented growth. The film morphology for a $YBa_2Cu_3O_x$ film on Ag is shown in Figure 4-34. Since most potential fibers contain alumina, or other reactive materials, the Ag was proposed as a protective interlayer, as well as an outer layer for the $YBa_2Cu_3O_x$. Perfluoro-1-methylpropenylsilver [Silver(PF)] was fed as a powder, using the vibratory feeder with argon as a carrier gas, into a 300°C, 0.04 atm furnace. Hydrogen (1 ℓ/min) was added to assist in the reduction reaction of the organometallic compound. Smooth Ag films were deposited on both flat and fibrous polycrystalline alumina samples. By using a chemical vapor deposition technique, uniform Ag coatings were realized on each filament of a DuPont FP alumina fiber tow as shown in Figure 4-35. An x-ray diffraction pattern of the Ag film is shown in Figure 4-36, which verified the presence of Ag. Two point resistance, Auger and SEM were also used to characterize the films.

4. Silver Interlayer/Overlayer Studies

Silver films which were deposited on fiber and flat substrates were subsequently coated with $YBa_2Cu_3O_x$. An x-ray pattern of the two layered films is shown in Figure 4-37. A randomly oriented superconducting film was evident, and the presence of silver is also noted on the pattern. The absence of $BaAl_2O_4$ in the pattern provides evidence that the Ag protected the film from reaction with the substrate. Electrical tests for the Ag/$YBa_2Cu_3O_x$ coated samples were inconclusive. One sample, which was annealed at 550°C for 16 hours in oxygen, was superconducting at 72 K. Most of the samples,

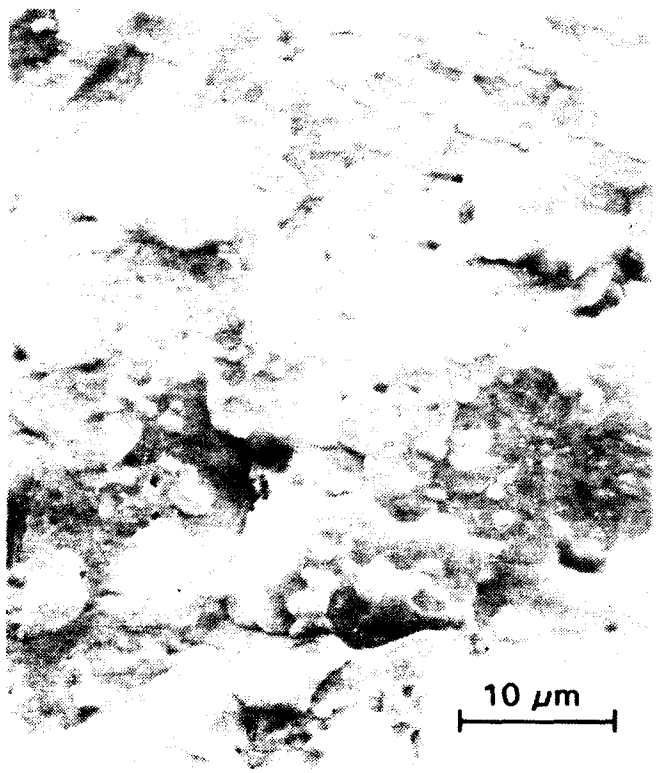


Figure 4-34. $YBa_2Cu_3O_x$ films were successfully deposited on planar silver substrates. The film orientation was not as extensive as compared to single crystal oxide substrates.

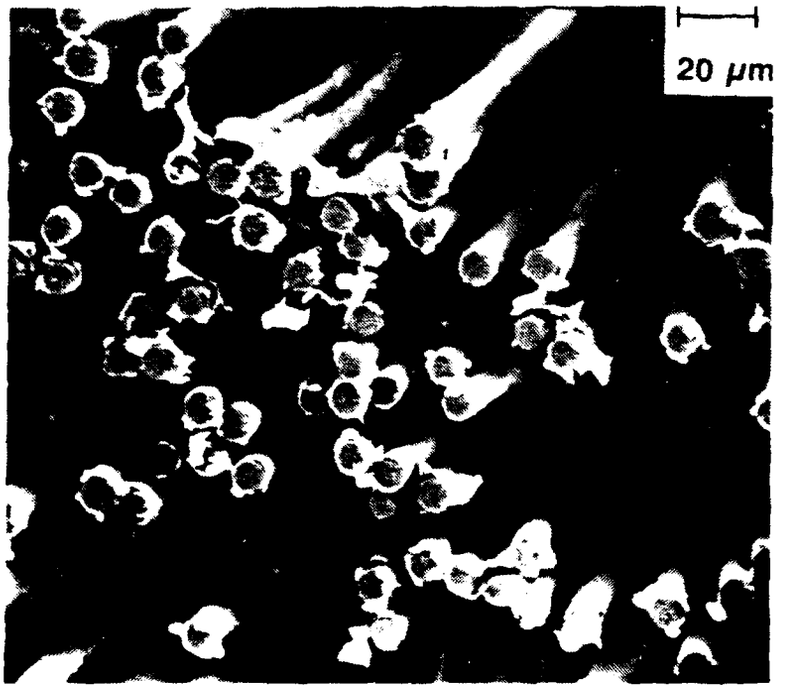


Figure 4-35. Micrograph showing a DuPont FP alumina fiber tow that is completely infiltrated and coated with an Ag film.

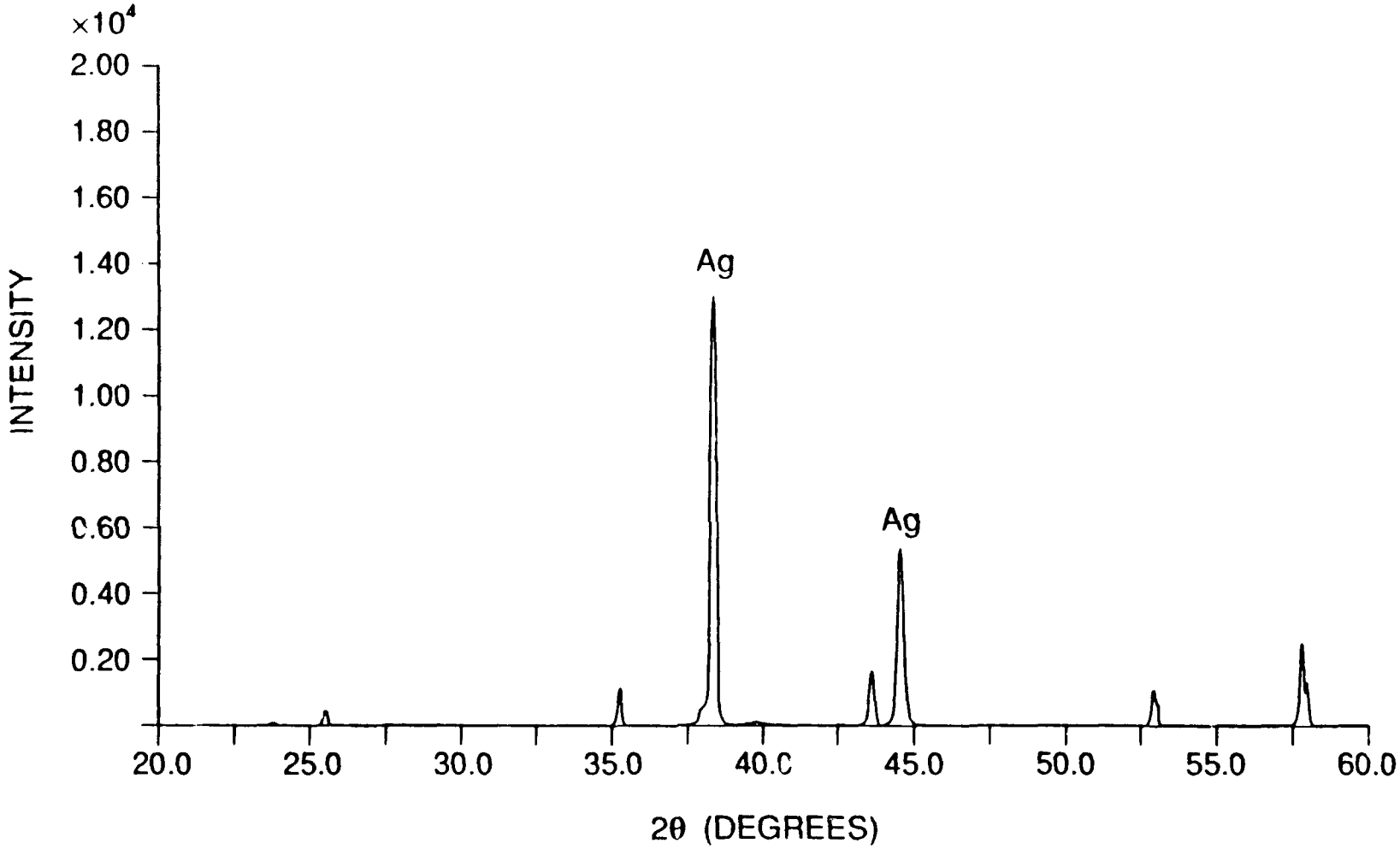


Figure 4-36. X-ray diffraction pattern of coating produced from Ag(PF) on polycrystalline alumina. The only phase detected was Ag.

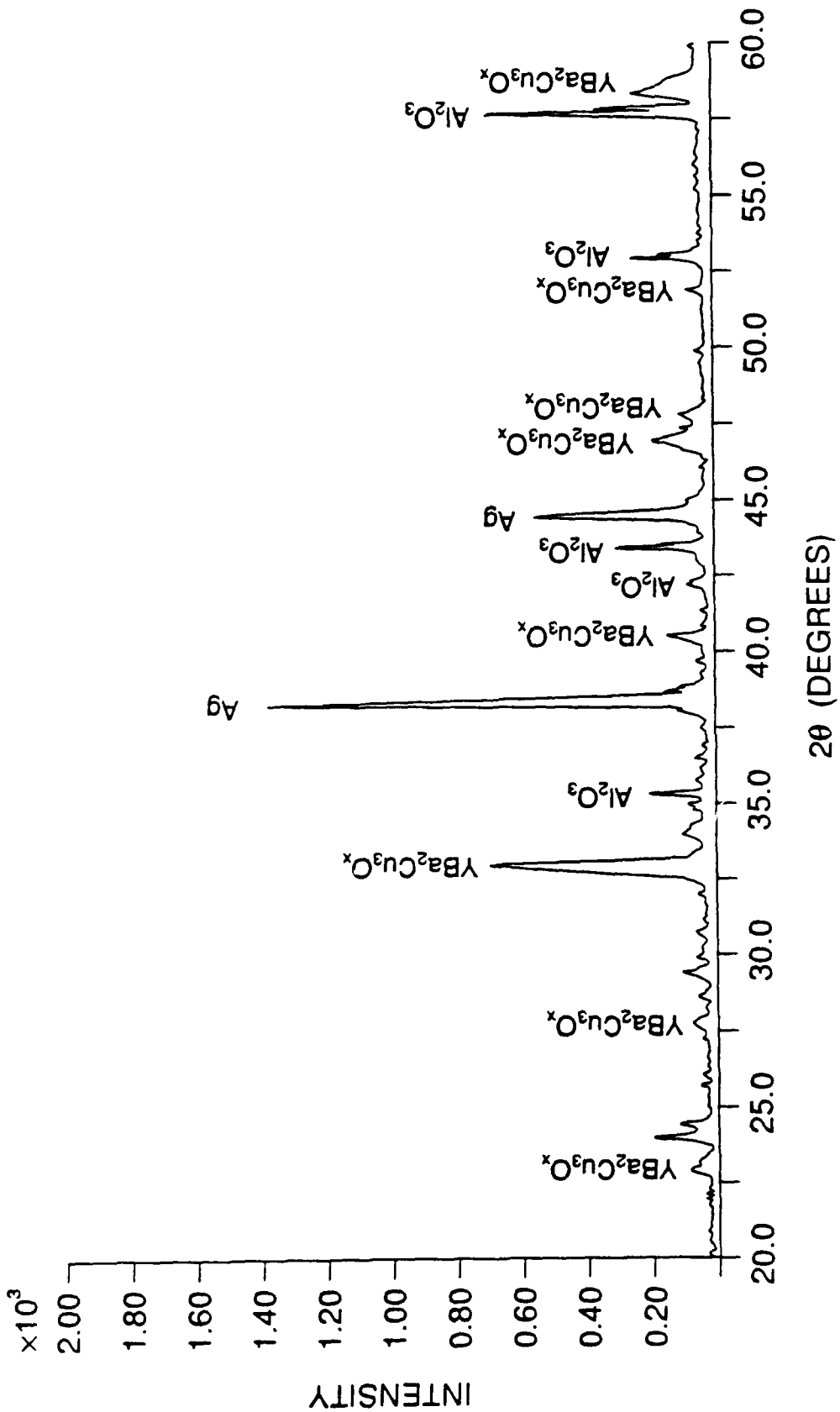


Figure 4-37. X-ray diffraction pattern of $YBa_2Cu_3O_x$ deposited over a Ag film produced from Ag (PF). The pattern indicates that the $YBa_2Cu_3O_x$ was randomly oriented and that Ag was present; no $BaAl_2O_4$ was observed.

however, exhibited semiconducting behavior or had a resistance exceeding the sensitivity of the equipment. The random structure of the deposited films observed using SEM contributed to the poor electrical results.

Silver deposits on top of $YBa_2Cu_3O_x$ coated substrates were also accomplished using the deposition procedure described previously. The major concern in this work was avoiding the reduction of oxygen in the $YBa_2Cu_3O_x$ structure during processing in a hydrogen environment. Before and after coating cell volume calculations, completed using x-ray diffraction results, indicated a cell volume decrease following the Ag deposition. The decrease corresponded to a reduction in oxygen from 6.6 to 6.1--the desired oxygen content is 6.8. The $YBa_2Cu_3O_x$ film was completely coated with Ag, showing that silver could be used as an effective protective overlayer coating for the material.

B. Fiber Coating

1. Deposition Work

The deposition of $YBa_2Cu_3O_x$ onto fibers was pursued throughout the three year effort. The main project goal, to develop a flexible superconducting wire that could be used to produce a magnet, relied on the strength and flexibility of the individual fibers in a fiber tow, and on the ability to coat each filament, resulting in a high volume fraction of superconducting material which would transport the required current. Short lengths of fibers were included in each batch run prior to receiving the

continuous fiber coating furnace, and over 150 experimental runs were completed using the continuous fiber coater.

a. Substrates (Fibers and Tapes)

Several types of fiber tows, monofilaments, and tapes were used as substrate materials. Fibers which contained silicon (including the Nextel, Sumitomo, Nicalon, and HPZ materials described in Table IV) were found to react with the coating and were extremely brittle. This was attributed to the formation of impurity phases between the film and substrate. Films deposited on Al_2O_3 fiber tows and sapphire monofilaments were more encouraging. Saphikon, Inc. provided 250, 150, and 75 μm diameter sapphire monofilaments for use as substrates. A resistance versus temperature curve for a $YBa_2Cu_3O_x$ coating on Saphikon is shown in Figure 4-38. A critical temperature of 82 K was observed for the film shown in Figure 4-39. A uniform approximately 10 μm coating was deposited on the filament.

Films deposited on polycrystalline Al_2O_3 fiber tows (FP Al_2O_3 from DuPont) also contained some superconducting phase as shown in the x-ray diffraction pattern in Figure 4-40. The presence of $BaAl_2O_4$ can also be seen. Uniform coatings were typically observed on the fiber tows as shown in Figure 4-41. A second DuPont fiber (PRD-166) which consisted of 85% Al_2O_3 + 15% ZrO_2 was favored for a majority of the experimental runs, since it exhibited a higher strength over the FP fiber. Frequently, the FP fiber would break during an experiment as it was pulled through the coater, due to the low fiber strength and fiber nonuniformity.

The final substrate type used in the fiber coating furnace

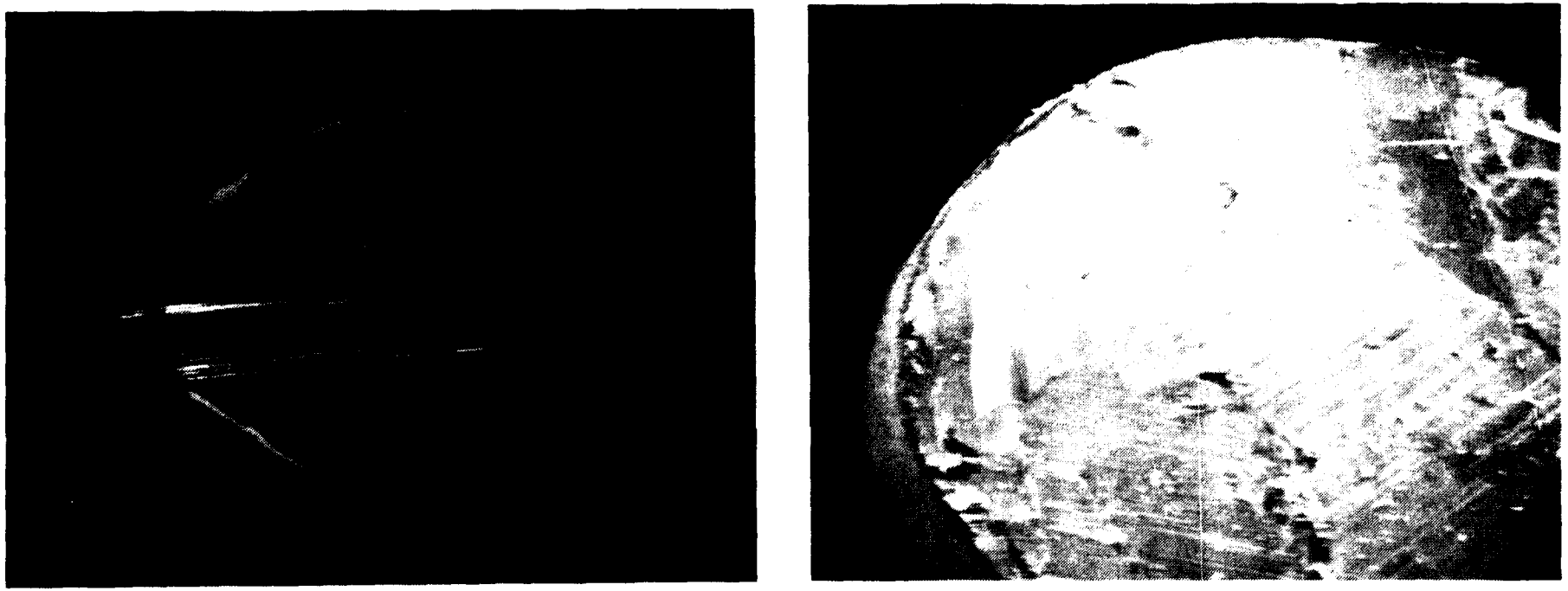


Figure 4-38. Micrographs of single crystal Al_2O_3 (Saphikon) fibers show a uniform 1-2 μm thick $YBa_2Cu_3O_x$ coating.

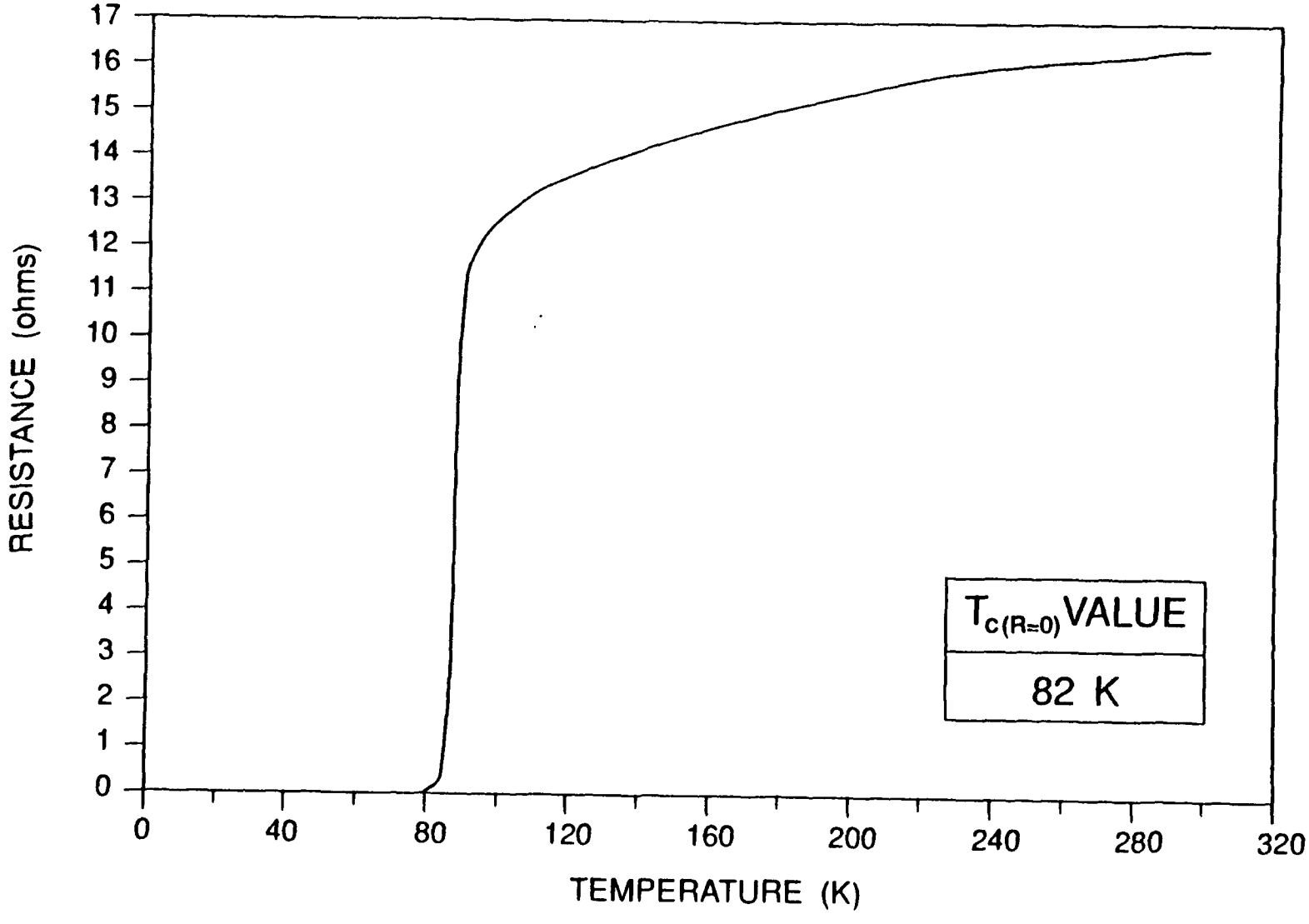


Figure 4-39. Resistance vs. temperature measurement for a $YBa_2Cu_3O_x$ coating on a Saphikon (sapphire) fiber shows a critical temperature of 82 K.

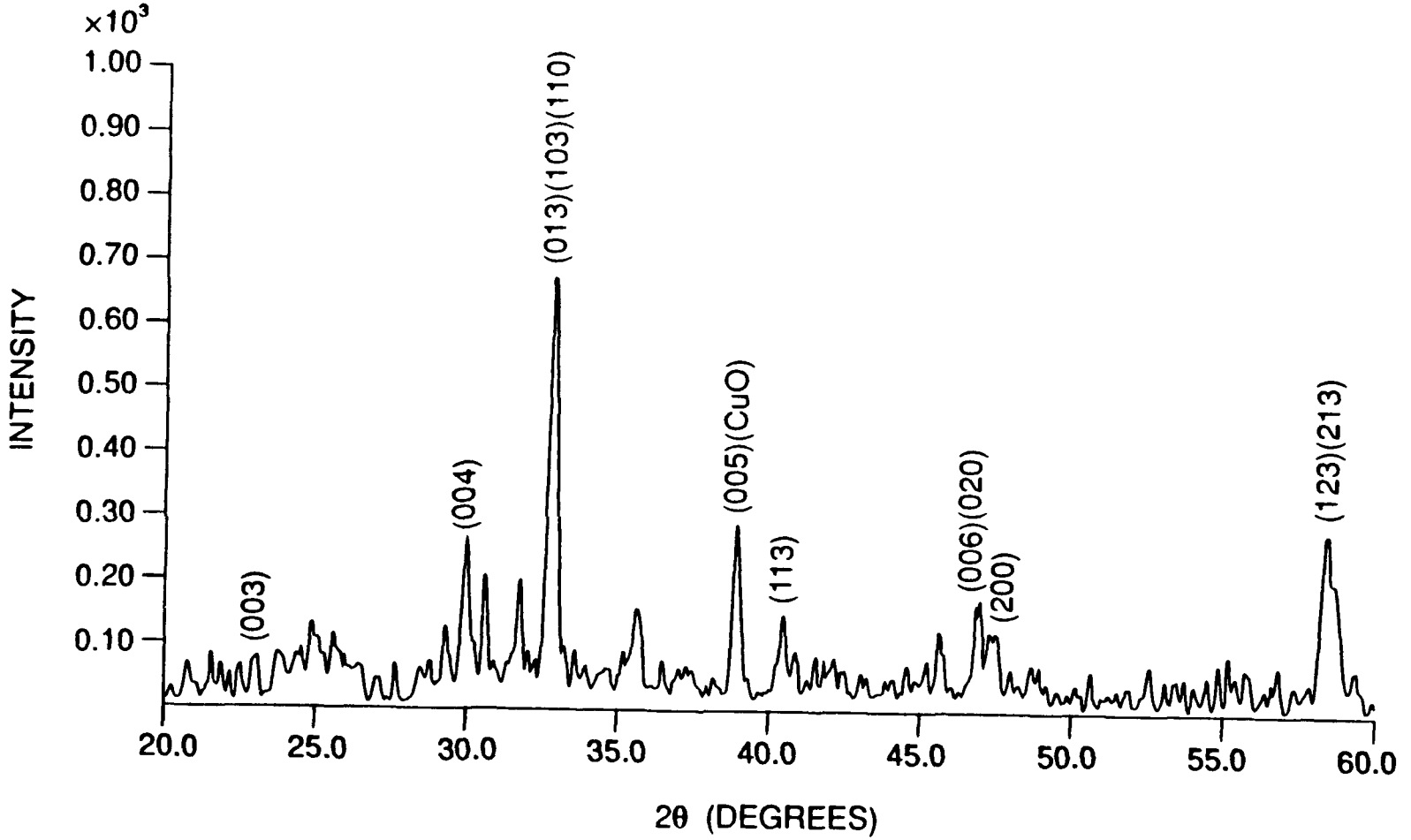


Figure 4-40. X-ray diffraction pattern of polycrystalline Al_2O_3 fiber coated with $YBa_2Cu_3O_x$ shows some c-axis oriented growth with a large unoriented $YBa_2Cu_3O_x$ content.

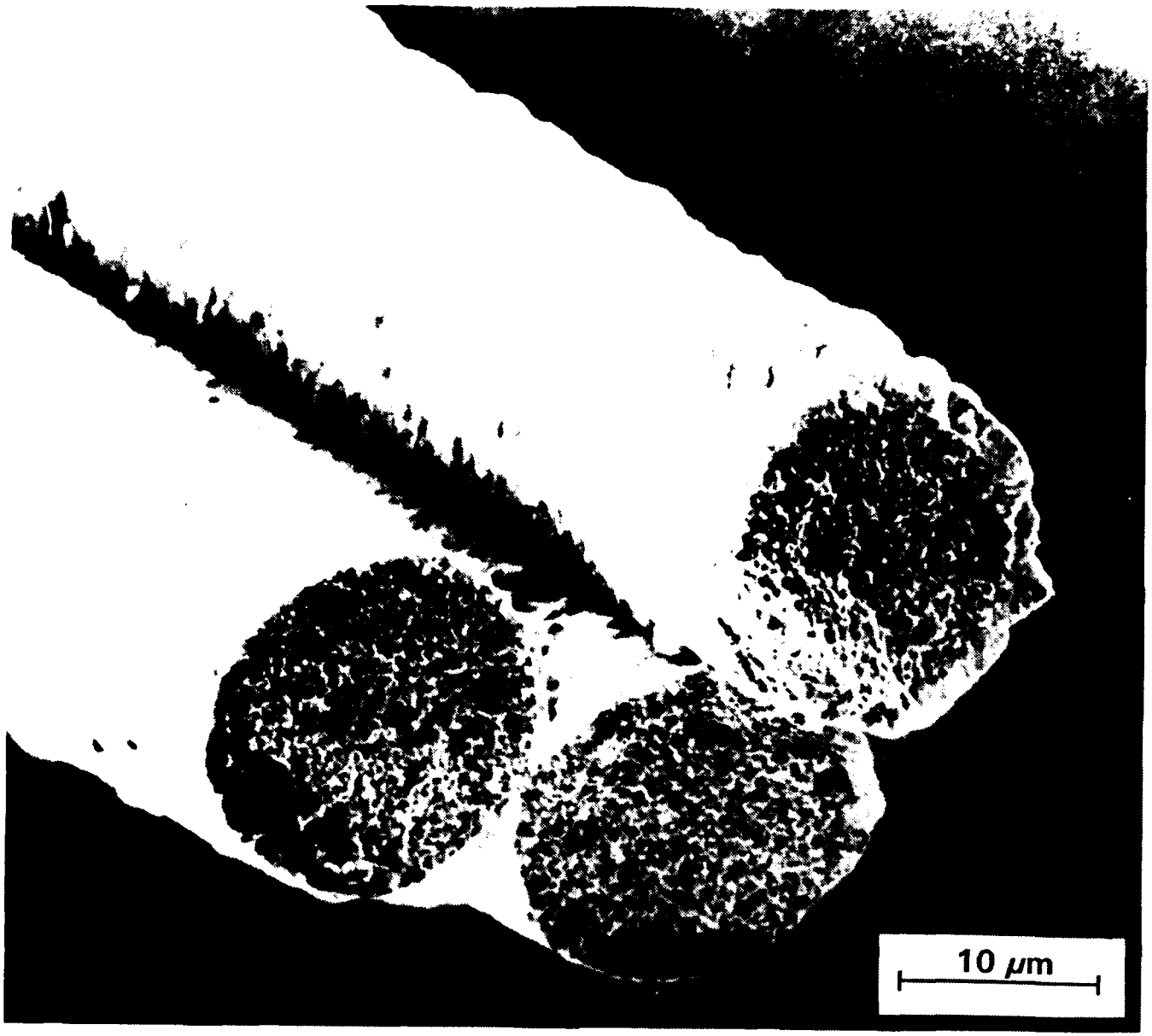


Figure 4-41. Uniform coatings of $YBa_2Cu_3O_x$ were observed on the filaments of fiber tows. The micrograph shows a 1 μm thick coating on FP Al_2O_3 fibers.

consisted of thin, flexible, metal tapes. Both Kanthal and Ag tapes were used. Tapes were used to simplify the composition analysis for materials pulled through the reactor, since the composition on fibers was extremely difficult to determine over a long length. The 1 cm wide x 0.5 mm thick tape was pulled through the coater during the run, and was also held stationary to determine compositional profiles along the length of the coater. Strips of Kanthal tape were also used to profile the composition, and determine an appropriate reaction zone for the coater.

Although carbon fibers were incompatible with the high temperature, oxygen environment of the $YBa_2Cu_3O_x$ deposition process, they were considered a viable substrate material as long as an oxidation protective coating was deposited prior to the $YBa_2Cu_3O_x$ deposition. Commercial carbon fibers provided the smallest diameter (5-7 μm) substrates, which would allow for the desired high volume fraction of superconductor/tow.

b. Parametric Studies

The $YBa_2Cu_3O_x$ films were deposited over a range of conditions - both processing and equipment parameters were varied in order to optimize the coating quality and uniformity. A summary of these parameters and the ranges investigated are given in Table X. Since the fiber was moving through the coating furnace, it was not possible to complete the normal in-situ anneal (cooling in oxygen) for the experiments. Following deposition, all the fibers were annealed using the furnace shown in Figure 3-3.

A nine run temperature-pressure study was completed in order to pinpoint the desired operating conditions. Films deposited at

Table X. Processing & equipment parameters varied in the fiber coater.

Reagent Feed Rate	1.1 g/min – 0.4 g/min
Oxygen Flow Rate	1 – 5 ℓ/min
Fiber Type	See Table IV, mainly used FP Al_2O_3, PRD-166
Fiber Pulling Speed	stationary to 30 cm/min
Reaction Zone Length	15 to 40 cm
Temperature	500 – 900°C
Pressure	0.04 – 1 atm

the lower temperatures were subsequently annealed and cooled in oxygen to transform the tetragonal to orthorhombic structure of the $YBa_2Cu_3O_x$. All of the low pressure runs (0.04 atm) had smooth uniform coatings as shown in Figures 4-42 to 4-44 for the 500, 700, and 900°C temperatures. The high pressure runs appeared nonuniform and nodular as shown in Figure 4-45.

The fiber pull rate was kept fairly slow during the deposition (6 cm/min). The deposition rates for the continuous fiber coater were lower than for the horizontal batch furnace. The deposition rate of the $YBa_2Cu_3O_x$ was extremely slow, typically 2 μm/h, so the slower speed was desired to obtain a reasonably thick coating. A vibration device was used to minimize fibers from becoming agglomerated by coating during the run. This device applied and released tension on the fiber as it was pulled through the coater at a controllable rate.

Composition of the deposited film was determined using EDS analysis of the coated tapes. Both static and moving profiles were completed. A static profile refers to a coating deposited when the fiber was stationary in the furnace. The thickness of the film was used to determine the maximum coating rate in the coater for any given conditions. Figures 4-46 and 4-47 present the Y:Ba:Cu coating composition deposited on Kanthal tape as a function of position in the furnace. Figure 4-48 defines the reaction zone length for the coater. The hot zone was approximately 33 cm; modifications to the coater were made to prevent any subsrate/reagent contact outside the hot zone. As shown, the stationary profile exhibited a large increase in Cu content near

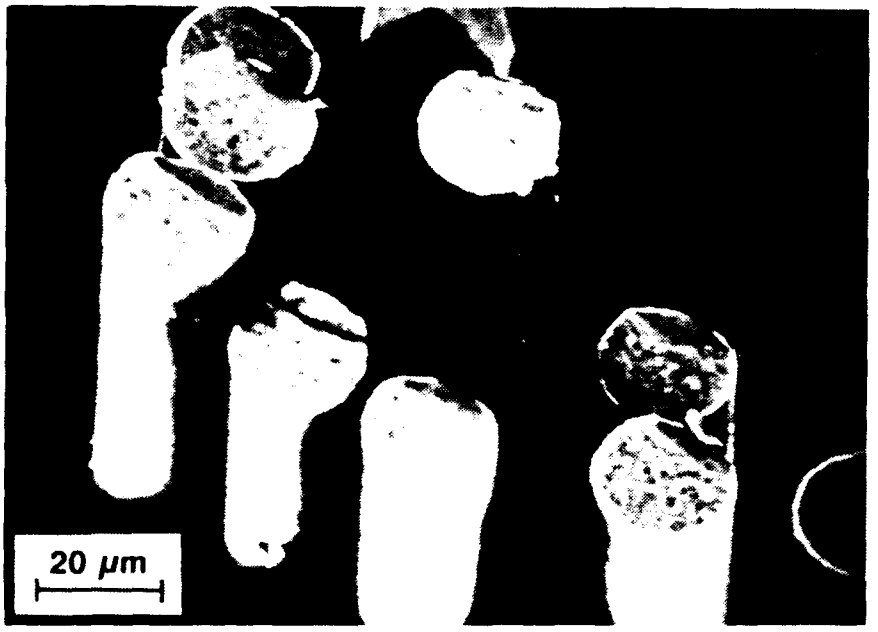


Figure 4-42. SEM micrograph for a fiber tow coated at 500°C, 0.03 atm showing a uniform, amorphous coating on each filament.

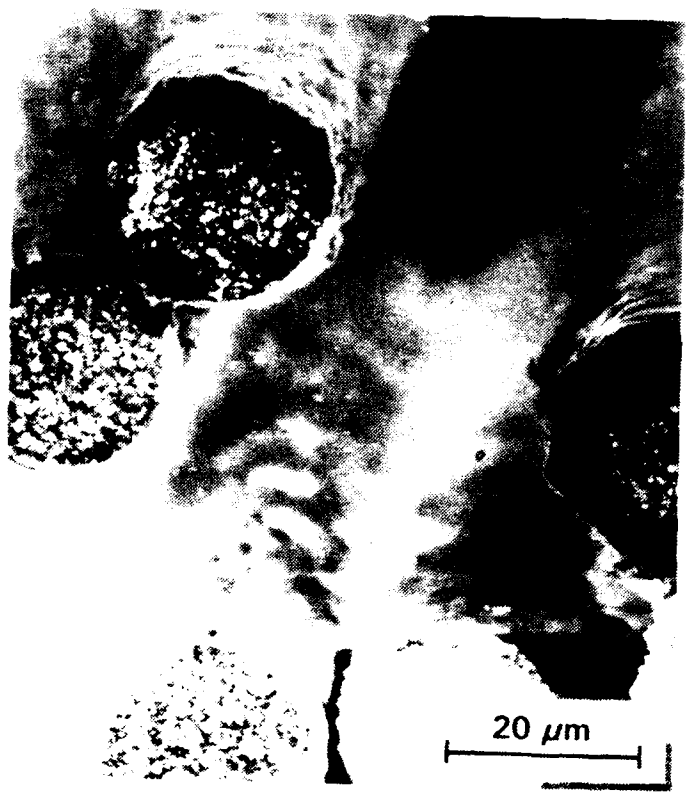


Figure 4-43. SEM micrograph for a $YBa_2Cu_3O_x$ film deposited at 700°C, 0.03 atm on FP Al_2O_3 showing a fairly smooth, uniform coating.

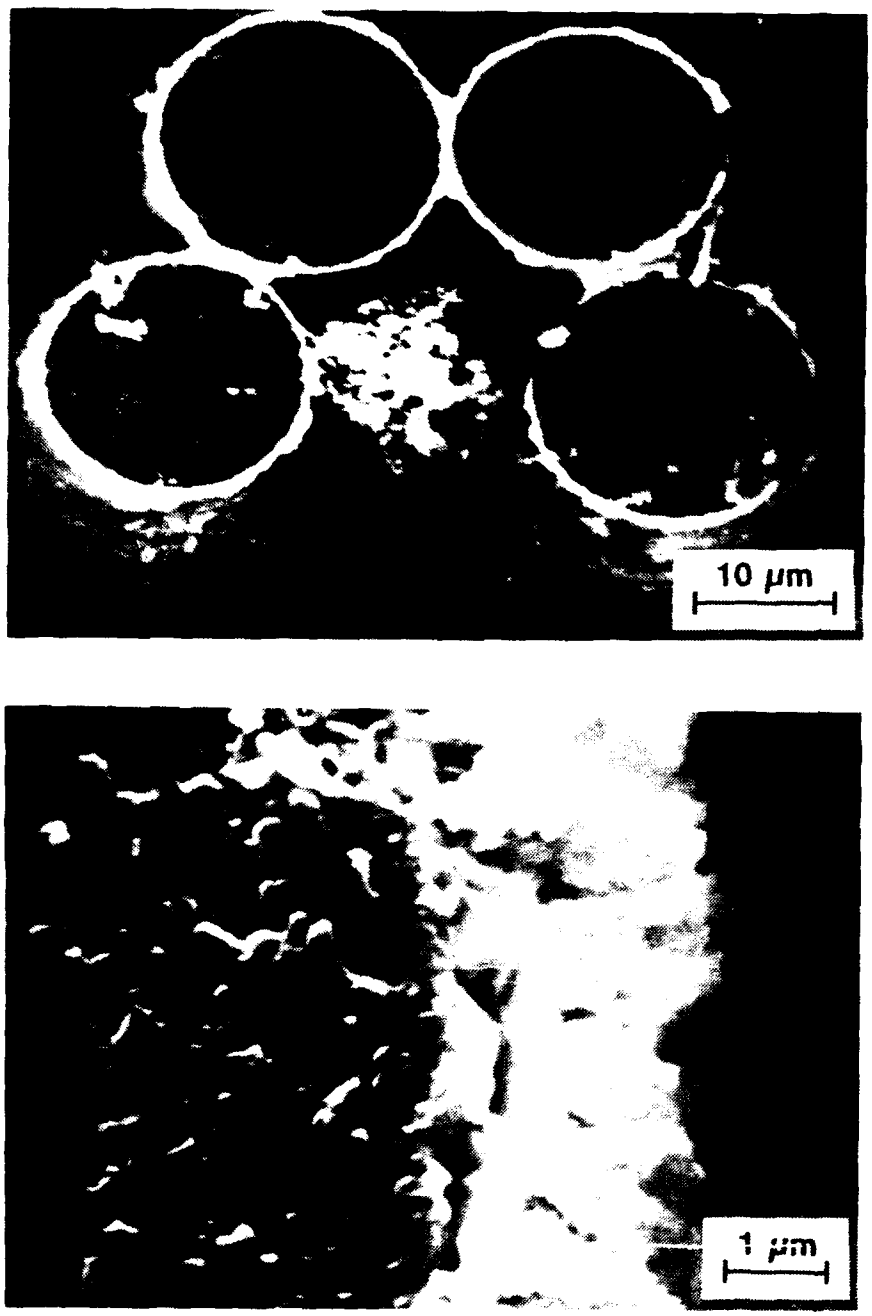


Figure 4-44. SEM micrograph for an FP Al_2O_3 fiber tow coated at 900°C, 0.03 atm shows a uniform, crystalline deposit around each filament.

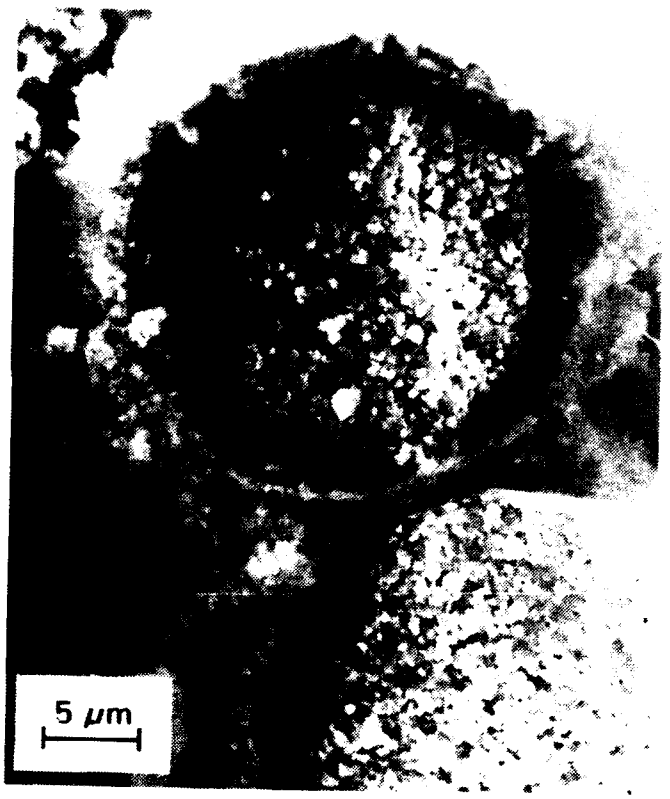


Figure 4-45. SEM micrograph of a 900°C, 1 atm deposition run on FP Al_2O_3 fiber. The morphology was extremely rough and the fibers were clumped together.

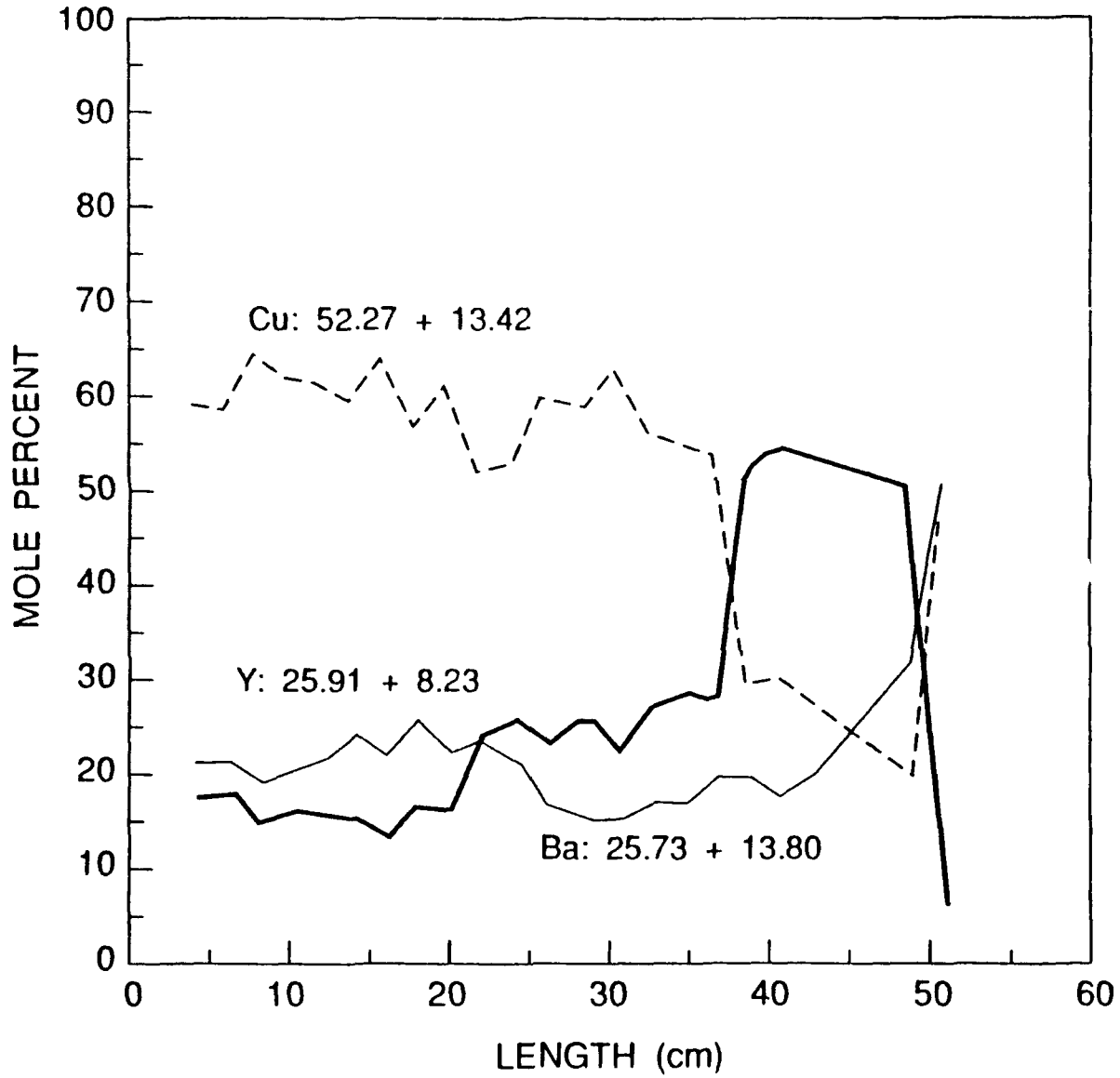


Figure 4-46. EDS composition for Y:Ba:Cu vs. position for a Kanthal tape held stationary in the continuous fiber coater. A depletion of reagents was responsible for the widely varying profile as the length into the furnace increased.

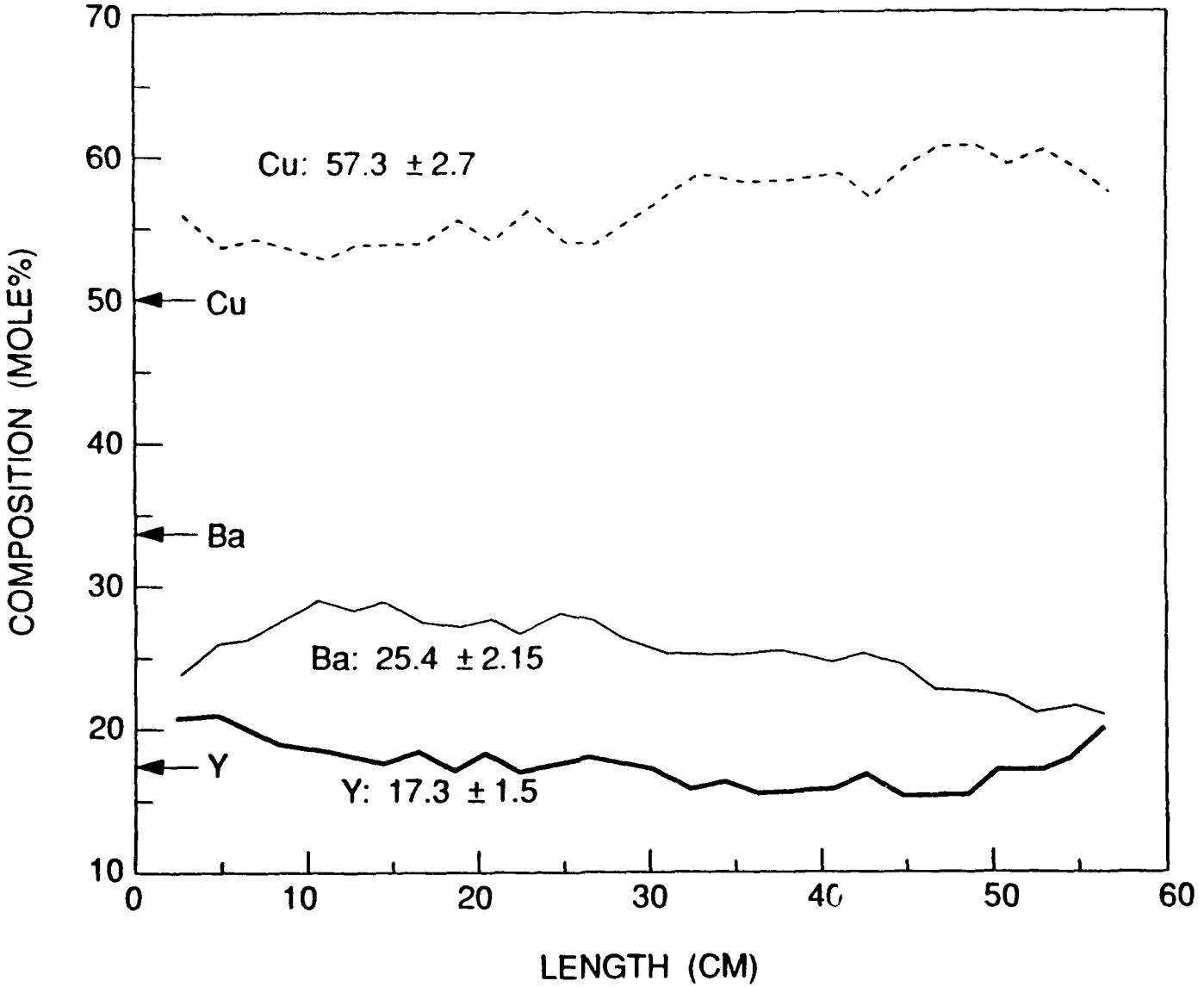


Figure 4-47. EDS composition profile vs. position into the continuous fiber coater for a moving Kanthal tape. A uniform profile was observed which is close to the ideal $YBa_2Cu_3O_x$ composition.

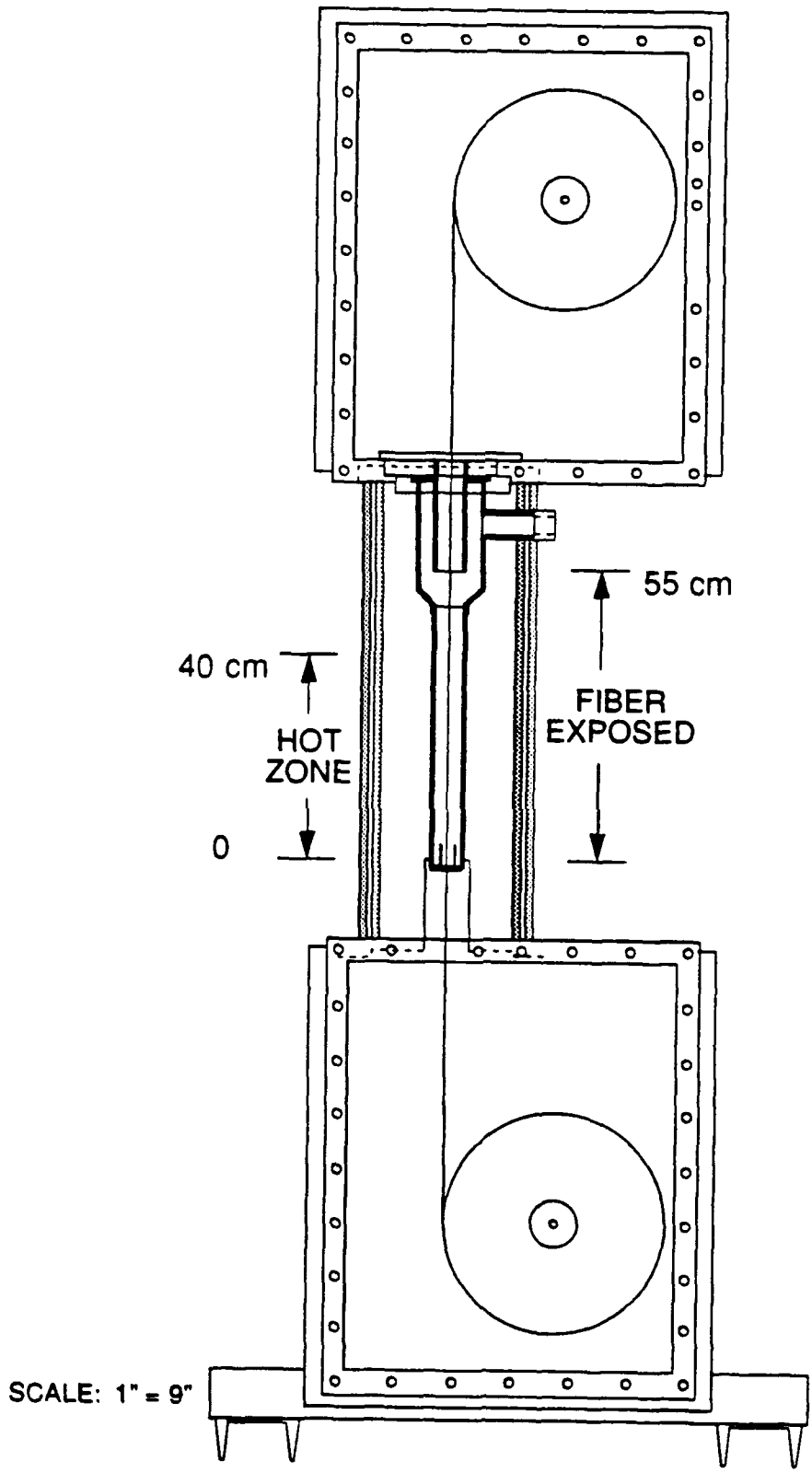


Figure 4-48. Schematic of the continuous fiber coating furnace defining the hot zone used after completing the composition profiles.

the top of the hot zone, due to the depletion of Y and Ba reactants. The moving tape, however, showed a fairly uniform profile, close to the desired Y:Ba:Cu ratio. Figure 4-49 shows the EDS signal from the Kanthal tape with the stationary profile given in Figure 4-46. The signal gives an indication of coating thickness as a function of position along the coater. The coating was much thicker in the central 15 cm of the hot zone.

c. Deposition on Silver

Silver coated tows were also coated with $YBa_2Cu_3O_x$ in the continuous fiber coating furnace and resulted in the highest critical temperatures determined on fiber tows. A Ag coated, $YBa_2Cu_3O_x$ coated tow of FP Al_2O_3 exhibited a critical temperature of 72 K as shown in Figure 4-50. Scanning electron microscopy and EDS analysis of the fiber was used to determine if the Ag remained at the film/substrate interface. The fiber, shown in Figure 4-51, did have some Ag at the interface - but a majority diffused into the grain boundaries of the coating.

An 18-run deposition/annealing study was completed on a series of FP Al_2O_3 tows initially coated with Ag[93]. The $YBa_2Cu_3O_x$ deposition was made at 500°C, 0.04 atm in the horizontal furnace. Subsequent annealing in various Ar/O_2 mixtures was used to initiate the tetragonal to orthorhombic phase transition. SEM and critical temperature measurements were used to characterize the as-deposited and annealed films. A summary of the results is shown in Table XI. The optimal annealing conditions were 835°C, at 2.9×10^{-4} pO_2. These films resulted in critical temperature onsets of 82 to 89.5 K. The microstructure of the as-deposited film is shown in Figure 4-52.

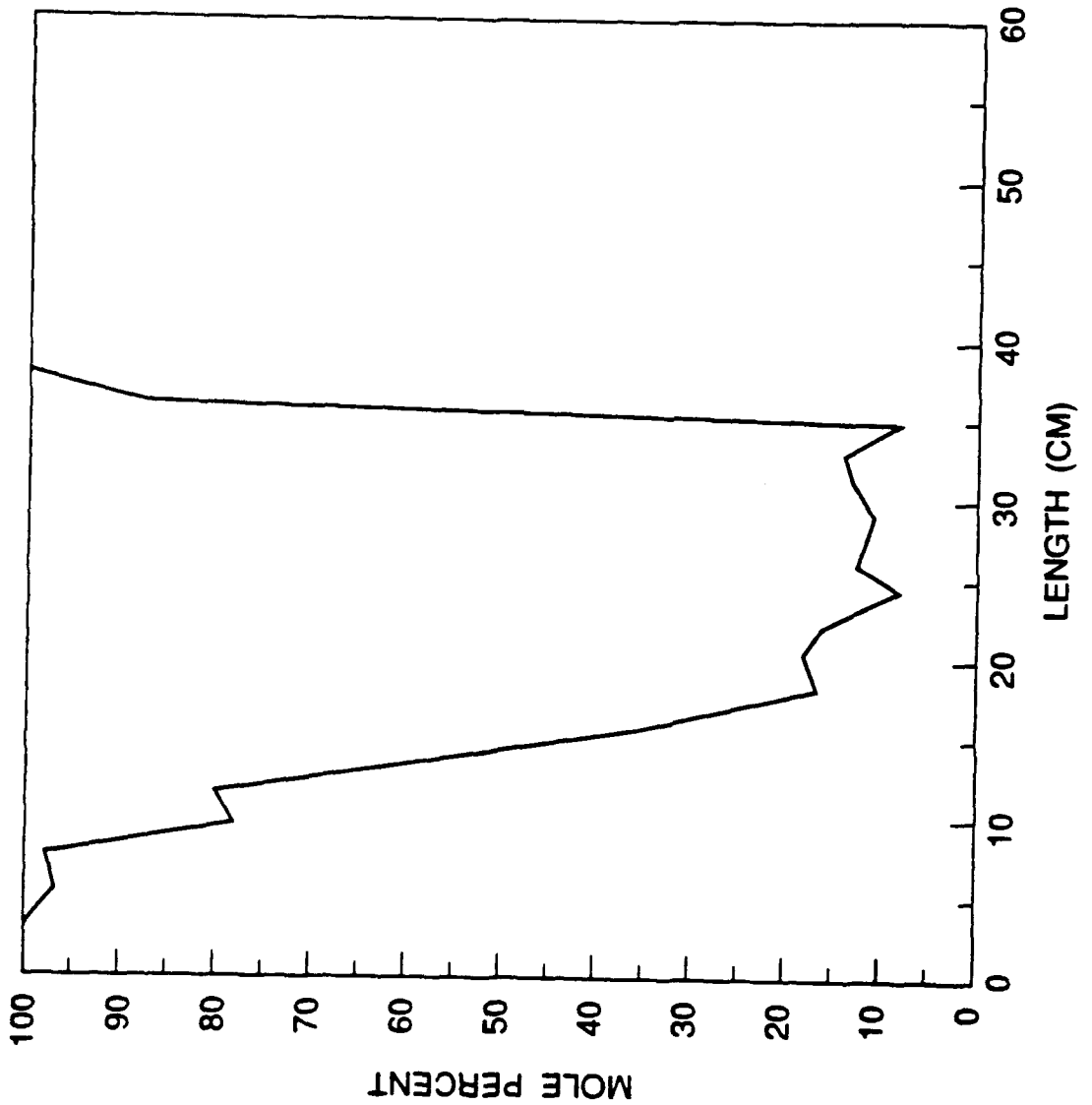


Figure 4-49. EDS signal for the $YBa_2Cu_1O_x$ coated Kanthal tape vs. position gives an indication of the film thickness. The thickest film was deposited between 15 and 35 cm into the hot zone.

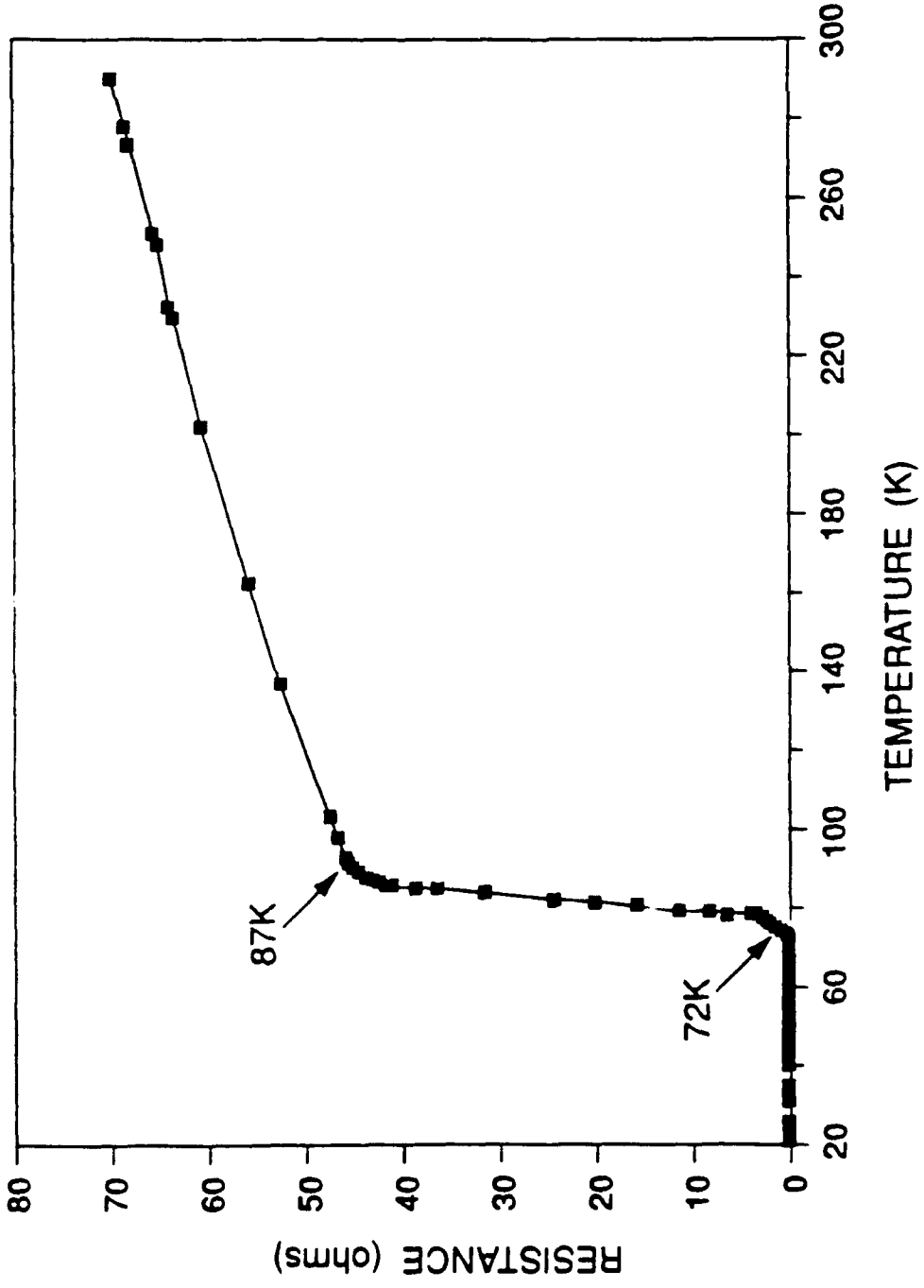


Figure 4-50. Resistance versus temperature curve of a polycrystalline Al_2O_3 fiber tow which was coated with Ag and then $YBa_2Cu_3O_x$. R = O Ω at 72 K.

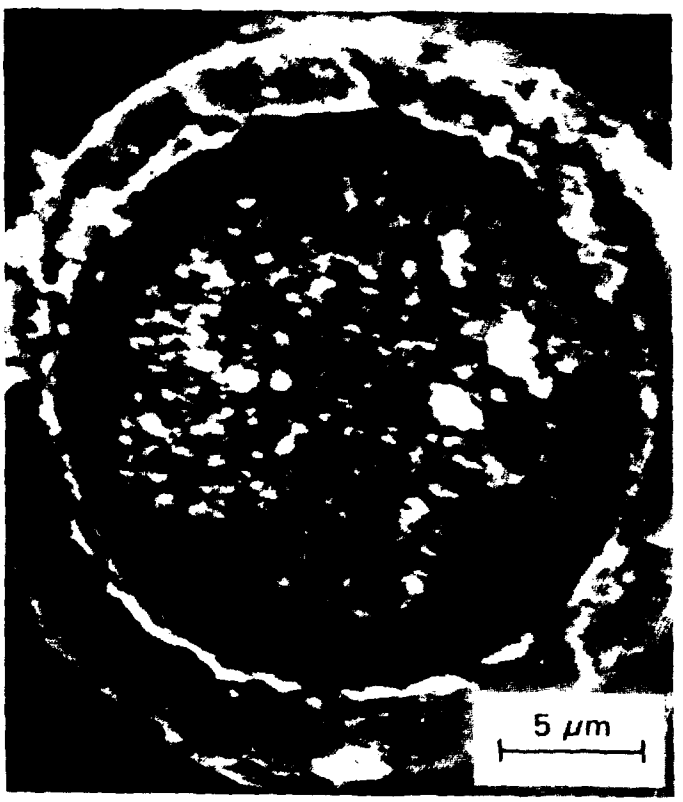


Figure 4-51. Cross section micrograph of an Ag coated/123 coated FP Al_2O_3 fiber. EDS indicated the presence of Ag at the fiber/coating interface and throughout the film.

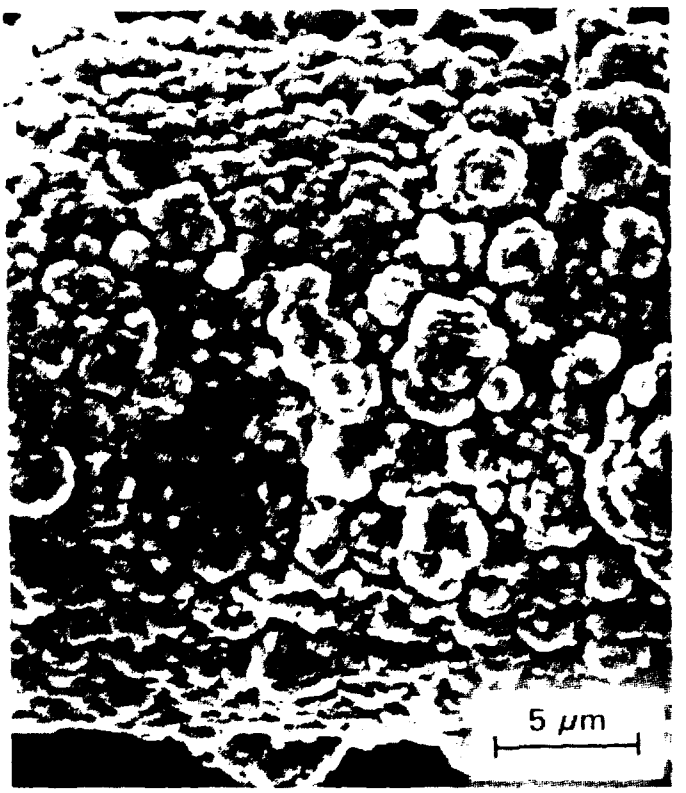


Figure 4-52. SEM micrograph of as-deposited $YBa_2Cu_3O_x$ on a strand of FP Al_2O_3 tow. An extremely nodular and rough surface was observed for this low temperature (500°C) deposition.

Table XI. Summary of electrical results of annealed coated fibers.

Anneal Conditions		Electrical Properties			
Temp (°C)	pO_2 (atm)	2-Pt. R (Ω)	4-Pt. R (Ω)	Trans.	Onset T (K)
740	1.0	3.4	.382	no	---
740	1.0	2.9	.102	no	---
740	2.8×10^{-2}	3.0	.038	no	---
740	2.8×10^{-2}	323	256	yes	85
740	2.9×10^{-4}	66	50	yes	85
740	2.9×10^{-4}	16	11.117	yes	86
795	1.0	2.8	.02	no	---
795	1.0	3.1	.198	no	---
795	2.8×10^{-2}	71.6	27	yes	83
795	2.8×10^{-2}	2180	1696.3	no	---
795	2.9×10^{-4}	101	.82	yes	86
795	2.9×10^{-4}	5.7	2.39	no	---
835	1.0	3.3	.276	no	---
835	2.8×10^{-2}	2.9	.0726	no	---
835	2.8×10^{-2}	3.9	.735	no	---
835	2.9×10^{-4}	2.7	.02	yes	89.5
835	2.9×10^{-4}	3.3	.32	yes	82
835	2.9×10^{-4}	588	365	yes	89
835	2.9×10^{-4}	1598	1270	yes	89

The morphology is consistent with earlier results; the Ag was found to remain at the interface prior to annealing.

2. Modeling

a. Stress Modeling

Analysis of the thermal and mechanical stresses in superconducting $YBa_2Cu_3O_x$ coatings on fibers was completed by a joint effort between Georgia Tech and the Oak Ridge National Laboratory[94]. The sources of stress included as-deposited (residual), thermal expansion mismatch which occur during cooling, and bending stresses which would develop on winding the coated fibers into a coil geometry. The analysis was completed for stresses in the axial, radial, and tangential directions, as defined in Figure 4-53, for a 4 μm thick coating on a 20 μm diameter fiber. The influence of varying coating thickness and fiber diameter were also evaluated.

Thermal stresses considered included the stresses developed due to the anisotropy of the coating with no fiber, the thermal expansion mismatch between coating and fiber, and the combination of the two sources. The combination of the coating anisotropy and coating/fiber mismatch is shown in Figure 4-54, along with schematics of expected coating damage modes for thick or thin coatings. The deposits would be expected to segment along the length when the axial tensile strength of the coating is exceeded; delamination of thick coatings would be expected to occur when the radial tensile strength is exceeded.

The critical bending radius for a coated fiber was calculated by using the most severe axial tensile stress due to bending along

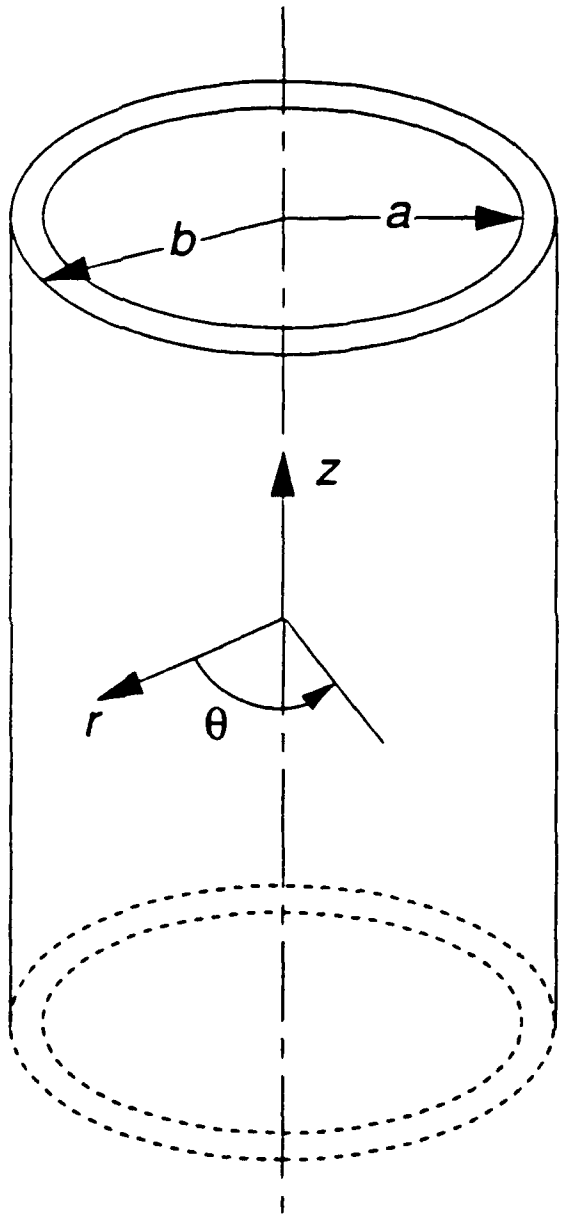


Figure 4-53. Fiber/coating geometry used to define the axial, radial, and tangential stresses in the model

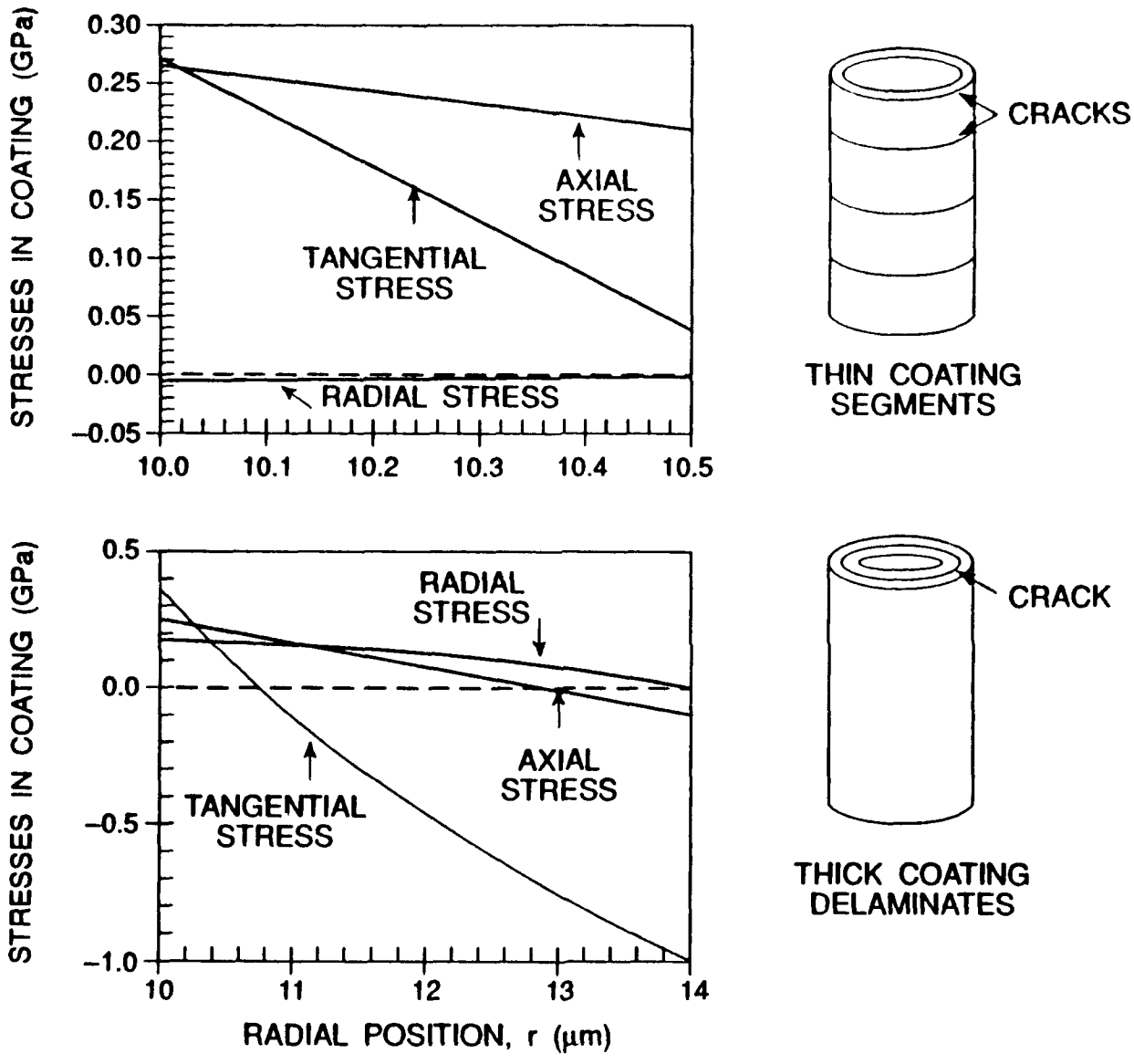


Figure 4-54. Coating anisotropy and thermal expansion stresses due to mismatch between the fiber and coating are plotted as a function of coating thickness. The schematics show expected damage modes for thin and thick coatings.

the y-axis in the convex surface shown in Figure 4-55. The resulting segmentation damage expected was predicted to occur when the axial tensile stress exceeds the tensile strength of the material. Figure 4-56 shows the critical bending radius versus coating thickness for three levels of coating strength for a 10 μm fiber radius. The critical bending radius was predicted to decrease as the coating thickness or coating strength increased.

The analysis for a 4 μm thick $YBa_2Cu_3O_x$ film on a 10 μm radius Al_2O_3 fiber indicated that the potential for segmentation or cracking of the coating was reduced when the coating has a preferred orientation. Isotropic coatings resulted in tensile stresses in the axial and tangential direction greater than the reported tensile strength of $YBa_2Cu_3O_x$. Choice of coating thickness and fiber radius were emphasized as controlling factors in reducing potential damage to the coating. For example, segmentation of the coating could be inhibited by depositing a thicker coating or choosing a smaller diameter fiber.

b. Process Modeling

Modeling of the $YBa_2Cu_3O_x$ deposition process was completed in order to study the competition between the reaction kinetics and the diffusion of the reactant specie into the fiber bundle for various temperature, pressure, reactant concentration, and fiber bundle geometries. Uniform coatings around each filament were desired, so fast diffusion and a slow reaction process was favored. The model was developed by completing a mass balance around a cross section of the fiber bundle (shown in Figure 4-57), assuming ordinary diffusion and constant physical properties of the

reactants. A BASIC computer program was written which calculated the reactant concentration profile as a function of position into the fiber bundle.

The solution to the reaction/diffusion competition resulted in a Modified Bessel Function series expression. A dimensionless, scaleable parameter called the Thiele modulus was calculated for given processing parameters. The Thiele modulus related the ratio of the rate of reaction to the rate of diffusion as a function of temperature, pressure, and concentration. A small Thiele modulus was favored in order to predict a uniform deposition on each filament.

For the deposition of $YBa_2Cu_3O_x$, the experimentally observed deposition rates varied from 1 to 240 μm/h, which indicates an extremely slow reaction. The activation energy for the deposition was presented by Schmaderer et al.[58] and is shown in Figure 4-58. The plot of log growth rate versus 1/T (K^{-1}) had a slope of 73 kJ/mole. Since the rate of deposition was always extremely small compared to the diffusion, the resulting Thiele modulus values ranged from 10^{-1} to 10^{-9}. The predicted concentration profiles were constant as a function of position into the fiber. Uniform deposition was predicted over a 500 to 1000°C range in temperature, and a 0.04 to 1 atm range of pressure. Most of the experimental runs resulted in uniform coating as shown in Figure 4-59.

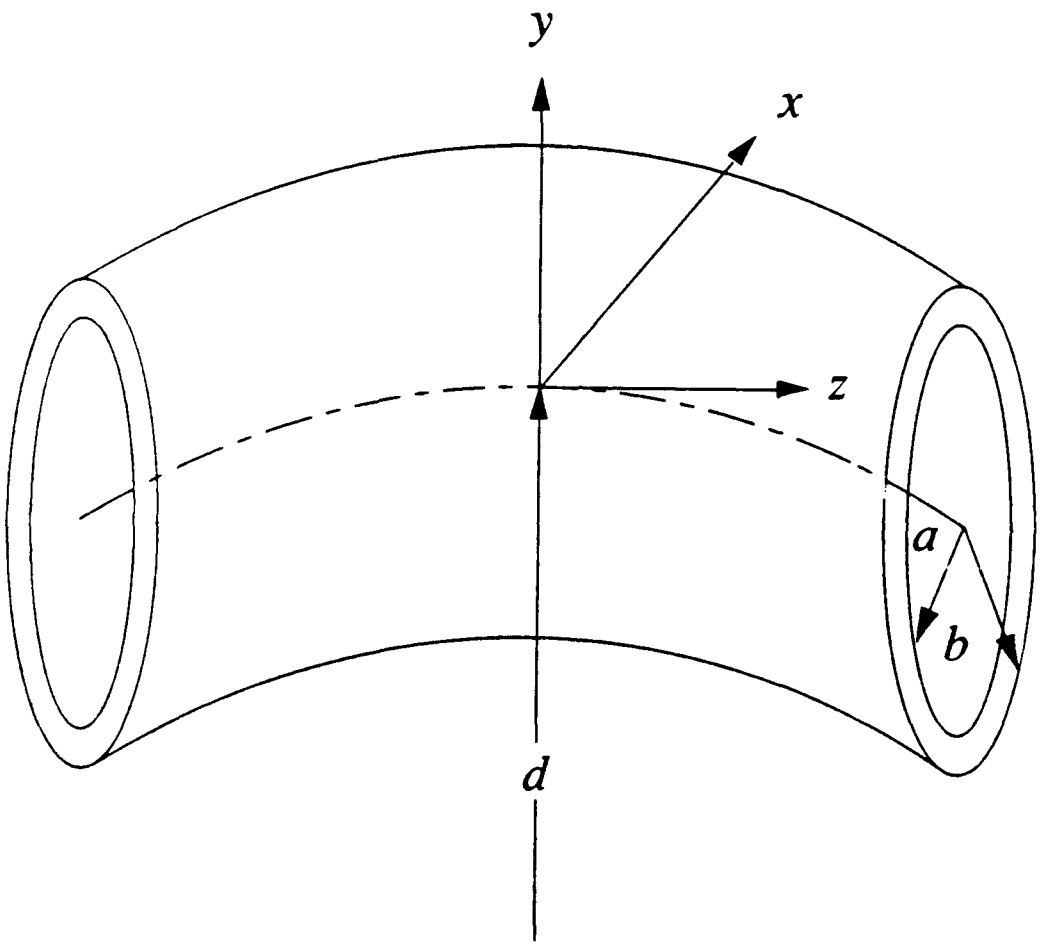


Figure 4-55. Fiber / coating bending geometry defines the maximum axial stress expected along the y-axis. *d* corresponds to the critical bending radius.

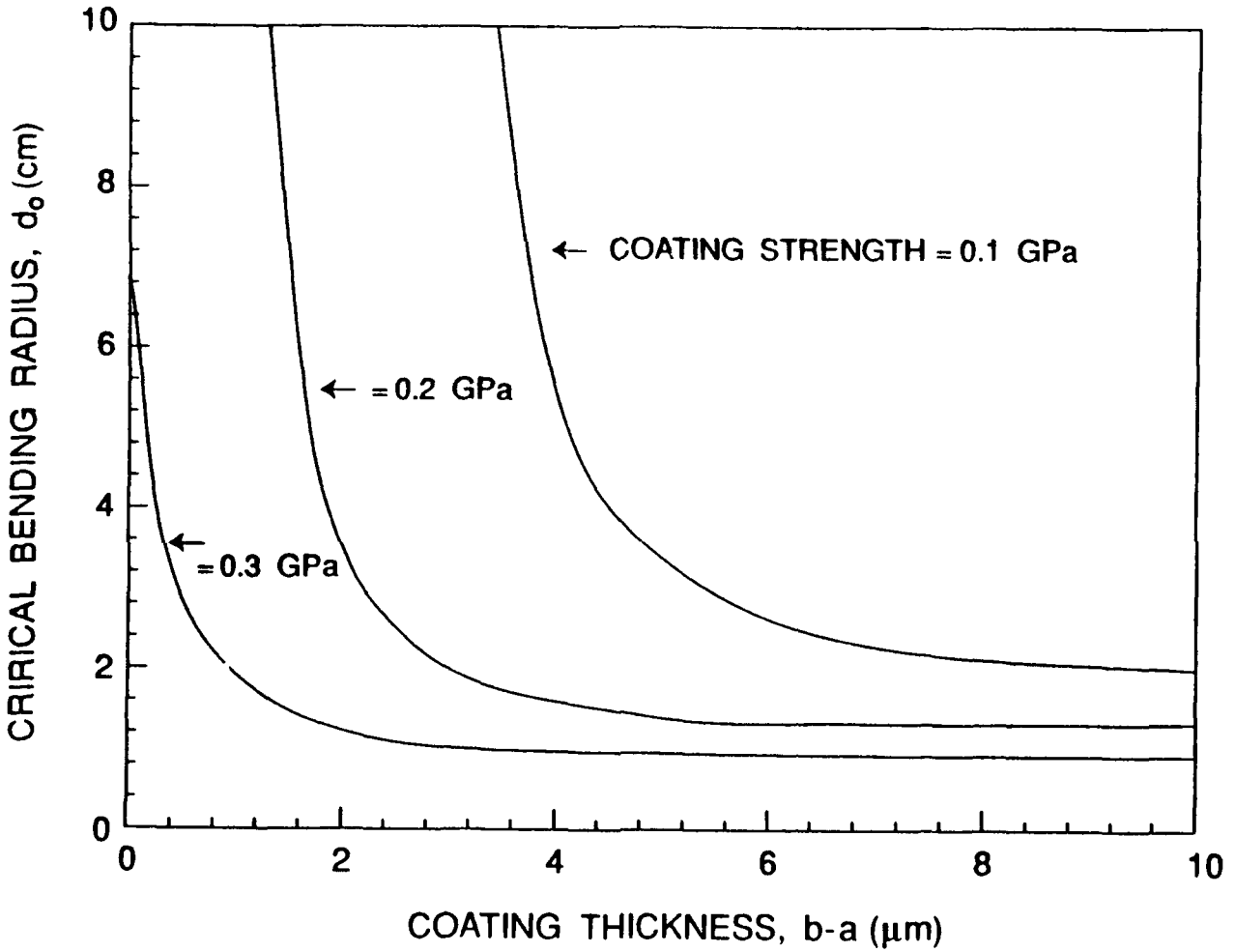
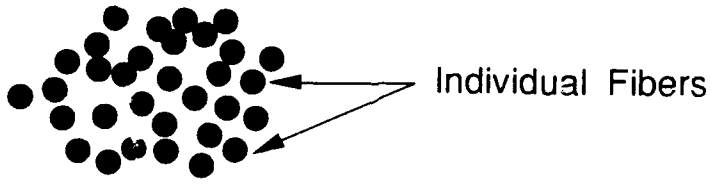


Figure 4-56. Critical bending radius versus coating thickness for a $YBa_2Cu_3O_x$ coating on Al_2O_3. The critical radius is shown to decrease as the coating thickness and coating strength increased.

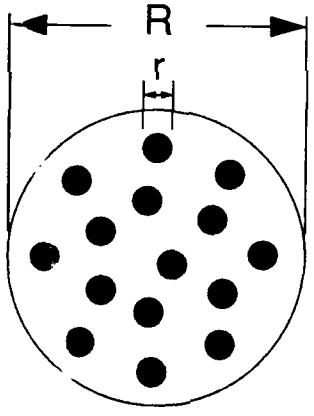
ACTUAL



Porosity Between Fibers
(~ 10 μm)

$$\% \text{ Porosity} = 1 - \frac{\pi r^2_{fiber} \times (\#\text{ of fibers})}{\pi R^2_{tow}}$$

MODEL



Fiber Tow Cross section

$$S_V = \text{Reaction Site Factor} \Rightarrow \frac{\text{Deposition Sites}}{\text{Area of Circle}}$$

$$= \frac{2\pi \sqrt{r} \times 200}{\pi R^2}$$

Figure 4-57. Physical and theoretical schematics of the geometry defined for the process model. Both the porosity and the reaction site factors were used to calculate parameters for the Thiele modulus.

reactants. A BASIC computer program, listed in Appendix C, was written which calculated the reactant concentration profile as a function of position into the fiber bundle.

The solution to the reaction/diffusion competition resulted in a Modified Bessel Function series expression. A dimensionless, scaleable parameter called the Thiele modulus was calculated for given processing parameters. The Thiele modulus related the ratio of the rate of reaction to the rate of diffusion as a function of temperature, pressure, and concentration. A small Thiele modulus was favored in order to predict a uniform deposition on each filament.

For the deposition of $YBa_2Cu_3O_x$, the experimentally observed deposition rates varied from 1 to 240 μm/h, which indicates an extremely slow reaction. The activation energy for the deposition was presented by Schmaderer et al.[58] and is shown in Figure 4-58. The plot of log growth rate versus 1/T (K^{-1}) had a slope of 73 kJ/mole. Since the rate of deposition was always extremely small compared to the diffusion, the resulting Thiele modulus values ranged from 10^{-1} to 10^{-9}. The predicted concentration profiles were constant as a function of position into the fiber. Uniform deposition was predicted over a 500 to 1000°C range in temperature, and a 0.04 to 1 atm range of pressure. Most of the experimental runs resulted in uniform coating as shown in Figure 4-59.

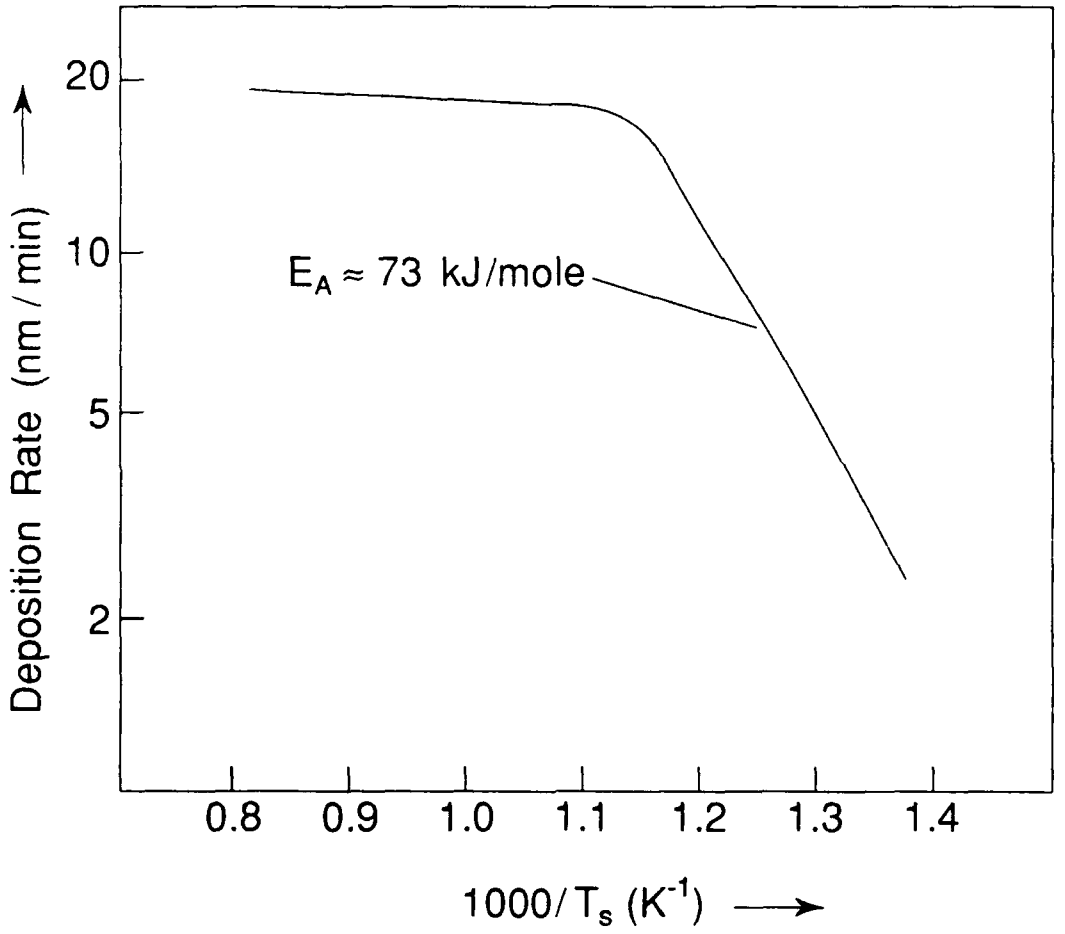


Figure 4-58. Experimental deposition rate for $YBa_2Cu_3O_x$ versus the reciprocal absolute deposition temperature indicates an activation energy of 73 kJ/mole.

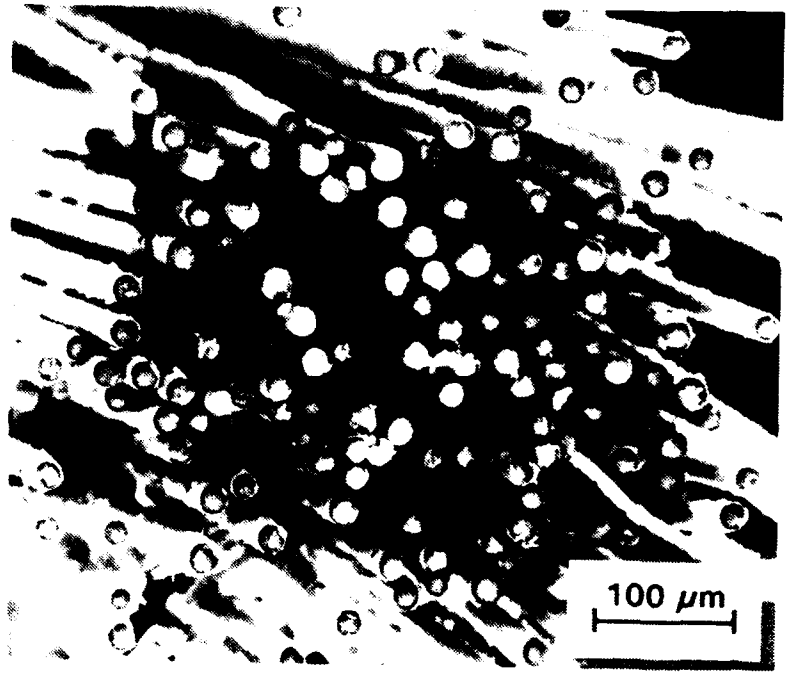


Figure 4-59. Fiber tow (DuPont PRD-166) shows a uniform deposition of $YBa_2Cu_3O_x$ around each filament.

DISCUSSION AND CONCLUSIONS

The chemical vapor deposition of $YBa_2Cu_3O_x$ onto flats and fiber substrates was investigated over a three year period. Several aspects of the deposition process, substrate selection, and characterization were systematically improved throughout the work, and problems were continually identified and corrected which enabled the superconducting material to be deposited onto a wide variety of materials -- specifically onto individual filaments of a ceramic fiber tow.

A. General Observations

1. Process Description

The powder feeding deposition process was favored over the vaporization method for introducing the organometallic reagent materials into the coating system. This technique delivered more reagent in a controllable fashion, with relative operational ease as compared to utilizing three separate vaporizers. Using powder, however, presented its own set of problems. First, the quality and consistency of the powder varied during the experimental work. This problem was eventually limited by storing the organometallic compounds in a vacuum desiccator. Second, the mixing of the three reagents as powder followed by screening caused segregation of the denser reagent. The Cu reagent was found to settle in the delivery line during the experiments (See Table X). This problem was

partially overcome by careful cleaning of the supply lines after each run, and the use of a high carrier gas flow rate. Another problem involved the pyrophoric nature of the powder, which was eliminated by grounding the delivery lines to prevent static charge buildup, and by mixing the oxygen farther downstream in the process. Finally, the uniformity of the feed rate was improved by the design of a rotating powder feeder.

Even though the powder handling presented numerous problems, it was favored highly over the vaporizer approach. When operating the system at the conditions favorable to $YBa_2Cu_3O_x$ deposition (875°C, 30 torr), nearly 80% of the films contained the orthorhombic $YBa_2Cu_3O_x$ phase as determined by x-ray diffraction. Electrical results were less favorable; the variability in coating quality regarding impurities and uniform substrate coverage was high.

The general trends for depositing high quality (based on electrical performance) c-axis oriented $YBa_2Cu_3O_x$ films on flat samples include operating the furnace at or above 850°C. Films with a-axis oriented $YBa_2Cu_3O_x$ films formed at temperatures below 850°C. Smoother, more uniform films were also deposited at lower pressures. The typical operating pressures during deposition were 20-30 torr. Smoother films were also deposited when using a slow powder feed rate; this resulted in a more efficient delivery of reagent to the furnace. The input composition most frequently used was higher in Ba content than the $YBa_2Cu_3O_x$ composition. This reflects the poor volatility of the Ba reagent as indicated by several researchers and/or lower deposition efficiency for Ba.

Finally, the post-deposition treatment most favorable was determined to be a slow cool at 1 atmosphere and 1 ℓ/min oxygen flowing through the furnace. The general processing trends were found to deposit smooth, glassy looking c-axis oriented $YBa_2Cu_3O_x$ films.

2. Substrate Selection

The substrate selection played an important role in the deposition of the $YBa_2Cu_3O_x$ compound. The study of reaction layers, and the eventual development of Ag interlayers proved necessary in order to deposit quality films on polycrystalline alumina substrates. The presence of Al in any substrate material necessitated the use of a protection layer to prevent $BaAl_2O_4$ formation. This point was extremely important when considering the potential fiber substrates available for continuous fiber coating.

The use of single crystal substrates was determined to favor the deposition of higher quality, epitaxial $YBa_2Cu_3O_x$. The highest critical temperatures achieved during this work were on $LaAlO_3$, which has a close lattice match with the $YBa_2Cu_3O_x$. This result also extended to the fiber coating work. The best coatings obtained on fibers were on single crystal alumina (Saphikon) filaments (250 μm diameter). The polycrystalline fiber tows available, which commonly contained either Al, Si, or both, probably had interaction layers between the film/substrate. The use of silver interlayers, specifically on DuPont FP Al_2O_3 and PRD-166 fiber, prevented the reaction layer from forming. These films were determined to be superconducting, however the c-axis orientation was probably poor and there was low critical current density.

3. Characterization

Film and fiber characterization techniques were investigated throughout the project. Characterization of flat samples, by SEM/EDS and x-ray diffraction, was useful in giving feedback to the processing studies, resulting in the development of higher quality films. Electrical characterization proved to be time-consuming (mainly due to problems in contacting the voltage and current leads), however, the feedback into the composition studies was critical in improving the deposited films.

Characterization of fibers was a complex problem. Scanning electron microscopy analyses were useful in determining coating uniformity and thickness; however, EDS was inaccurate due to the sample morphology. No standard was available for analyzing the fibers, and only qualitative values for the Y, Ba, and Cu content were obtained. X-ray diffraction of fibers indicated the presence of some superconducting phase; the intensity of the substrate peaks was commonly as high as any of the coating peaks. Finally, electrical characterization of the fiber tows resulted in several unique methods for mounting electrical contacts to multifilament samples. Silver paint which was dabbed onto a tow spread at the contact point provided the best electrical contacts. The contacts, however, were considered to be poor, and very limited testing was completed.

B. Accomplishments

The deposition of $YBa_2Cu_3O_x$ coatings onto flats and fibers resulted in several achievements. First, a unique powder feeding

approach for the mixture of organometallic reagents was developed and patented (currently pending). A continuous fiber coating furnace was designed, fabricated, and used to deposit uniform coatings of $YBa_2Cu_3O_x$ onto multifilament tows, monofilaments, and thin tapes, in order to provide flexible material for magnet fabrication. Magnets were not fabricated because of the apparent low critical currents of the coated fiber tows.

The deposition of superconducting films was studied in over 900 experiments using both processing and characterization to optimize the coating quality based on electrical performance. Superconducting films were deposited on both flats and fiber substrates; the highest critical temperature achieved was 91 K on $LaAlO_3$. The highest critical current achieved was 3.6×10^4 A/cm^2 at 0 Tesla, 77 K obtained on a single crystal flat partially stabilized zirconia substrate. An MOCVD process for depositing interlayers of Ag was developed in order to circumvent substrate/coating interaction which limited the quality of the films, specifically for the films deposited on the commercially available multifilament fiber tows. Characterization techniques were pioneered for $YBa_2Cu_3O_x$ property determination on fiber tows; and included several post-deposition annealing studies. Finally, theoretical studies using deposition parameters and stress calculations of coated fibers were modeled to give insight into improving the process of coating fiber tows with $YBa_2Cu_3O_x$.

APPENDIX A

The efficiency of reaction is calculated for both the conventional CVD approach and the powder feeding process is calculated in this section. It is important to realize that the efficiency calculations will vary depending on the type of system used, ie, hot or cold walled reactors. Several assumptions are required for each calculation, and will be stated prior to the calculation.

Based on molar evaporation rate data presented by Schmaderer for the conventional vaporizer method, the Y, Ba, and Cu organometallic reagents enter the furnace at between $2\text{-}4\text{x}10^{-4}$ moles/hour. If all three elements enter the reactor at the same rate, the Cu will limit the reaction efficiency since $YBa_2Cu_3O_x$ contains 3 moles of Cu. If the Cu is introduced into the reactor at $3\text{x}10^{-4}$ moles/hour, in 1 hour, $3\text{x}10^{-4}$ moles of Cu will enter the system. Assuming a 100% efficient reaction, $1\text{x}10^{-4}$ moles of the $YBa_2Cu_3O_x$ compound will be deposited. (3 moles of Cu/1 mole of $YBa_2Cu_3O_x$) The volume of $YBa_2Cu_3O_x$ corresponding to this molar amount is ($1\text{x}10^{-4}$ moles $YBa_2Cu_3O_x$ x 666 g/mole)/6.8 g/cm^3 = 0.0098 cm^3. A 1 cm^2 flat substrate would have a 98 μm coating based on this calculation. Studies using conventional CVD deposition of $YBa_2Cu_3O_x$ report that 1 μm films are deposited in 1 hour. Therefore, for conventional vaporization of the material, a 1% efficient process is realized.

For the powder feeding approach to $YBa_2Cu_3O_x$ deposition, a mixture of organometallic powders are introduced into the system. Since the deposition is ususally completed in 20 to 30 minutes, the amount of reactant used in the calculation has been extrapolated

for a run lasting 1 hour. The molar ratios of metals in the organometallic reactants $Y(TMHD)_3$:$Ba(TMHD)_2$:$Cu(TMHD)_2$ is 0.00234:0.00345:0.0026 for a typical ratio and amount of powder fed into the system. The amount of Y, therefore, limits the reaction for this technique. Again, assuming a 100% efficient deposition, a coating of 1590 μm thickness is calculated to form on a 1 cm^2 substrate. The typical deposition in a 20 minute period using this technique is 1 μm; in 1 hour, a 3 μm coating would be realized. The efficiency of this approach is thus about 0.2%.

It is important to see that the efficiency is approximately equal for both processes; however, the rate of deposition using the powder feeding approach is much faster, and can be changed based on the amount of powder input into the system. An example of this fact was the 40 μm thick coating deposited with the powder feeding technique in 5 minutes.

Data Used: for a 20 minute run, 2.79 g is fed into the furnace with a weight ratio of $Y(TMHD)_3$:$Ba(TMHD)_2$:$Cu(TMHD)_2$ = 1:2.34:2.24

$Y(TMHD)_3$ = 0.5 g/638.72 g/mole = 0.00078 moles in 20 minutes
= 0.00234 moles in 1 hour

$Ba(TMHD)_2$ = 1.17g/503.85 g/mole = 0.0023 moles in 20 minutes
= 0.0069 moles in 1 hour

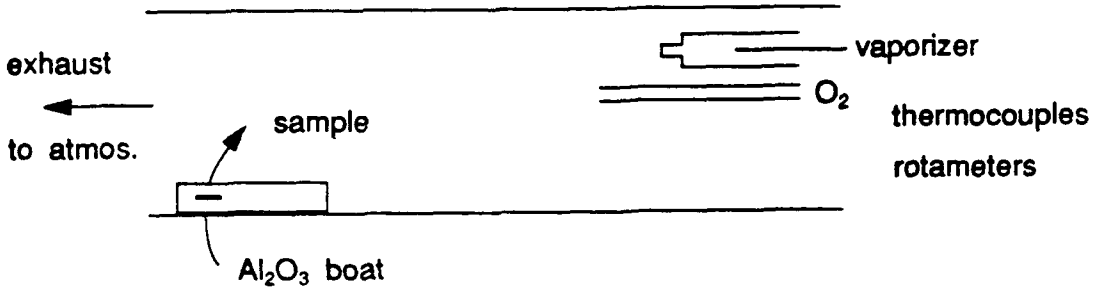
$Cu(TMHD)_2$ = 1.12g/430.05 g/mole = 0.0026 moles in 20 minutes
= 0.0078 moles in 1 hour

Since $YBa_2Cu_3O_x$ has 1 mole of Y, 2 moles of Ba, and 3 moles of Cu, the limiting molar feed rate is Y, since 0.00234< (0.0069/2) < (0.0078/3).

APPENDIX B

The following appendix includes brief summaries of the processing conditions and initial visual sample characterization for the horizontal batch furnace runs from run ST-18 to run ST-715.

Original Setup



ST-18 Heated zone 1 to 570°C. Loaded ~2 gm reagent in each vaporizer. Moving vaporizers to get T was successful but no coating was seen. Ba & Cu vaporizers were put at the wrong T -- need to label everything carefully. No characterization. M2, M3, M4, M5, M6

ST-19 Measured substrate position as 12½" from exhaust end; used ~1 gm of each reagents, but we didn't lose Cu? No deposit was seen on the substrate. Vaporizers were coated indicating some backflow. EDAX showed no coating.
On 8/29-8/30 did system leak checks using Ar/H_2 mixture.
M7, M8, M9, M10, M11, M12

ST-20 Modifications: 1) Profiled across vaporizers. 2) Ran Ar purge through the vaporizers at the start. T across vaporizer = 60°C. Lost Cu, little Y or Ba. Al_2O_3 boat was black; ESCA showed CuO coating. M13, M14

ST-21 Modifications: Thermocouples are placed at the midpoints of the vaporizers. Had problems with vaporizer temperatures including overshoot. All 3 vaporizers lost weight. Al_2O_3 substrate gained 0.003 g. No characterization completed.
M16, M17

ST-22 1st high temperature run. All 3 vaporizers lost weight but no weight gain on the substrate. No characterization completed.

9/9 meeting:
1) Extended vaporizer tubes
2) Try to put vaporizers in same position
3) Suspend the sample
4) Duplicate runs ST-21 and 22
5) Pursue vacuum runs

Note: Furnace may not be operating at same voltage on the top and bottom shell.

ST-23 Completed with new tube extenders and sample suspended. A black coating was seen on ½ of the Al_2O_3 substrate. All vaporizers lost weight. EDAX indicated Y & Cu at 1.06:1 ratio. Spectra dominated by Al. M19

New holder
Inconel 885 was used.

9/22 - Rotameters were replaced with Mass Flow Controllers.

ST-32 High T, new sample holder w/MgO. Showed some discoloration but lost weight due to hydration. No characterization completed. M33

Vacuum Runs

ST-33 Started using the new procedure. Noticed a blue-green coating on the O_2 extended tube. (Strem) Al_2O_3 coated, black at top, greenish at the bottom. Gained .0009 g. 11.4 Torr during run. EDAX completed showed Y, Ba, Cu at 5.6:4.2:1. Here is where we start getting small amounts of Cu, but were vaporizing a large amount. M34

ST-34 Same as ST-33 except for reagent type. (Kents) Al_2O_3 was coated with spotty green marks. Y temperature was overshot. M35

9/27 Maintenance
*O-rings were checked and cleaned. Y, Ba, & Cu thermocouples were changed. New, longer extension tubes were used to insure the tubes are inside the holder.
*Also, from now on, we will cool down completely with O_2.
EDAX completed. Green spots were Y. EDAX nearly 100%Y.

ST-35 High T, vacuum run. Substrate was coated on <u>both</u> sides. Lost a large amount of Y. EDAX showed mainly Y. M36

ST-36 Attempted to duplicate the Japanese work. All (tmhd's), high T, low flowrates. P = 3-5 Torr. Sample had a slight discoloration. EDAX showed Y:Ba:Cu = 25:59:16. M37

ST-37 <u>No vacuum</u>, high T, no coating on the sample; however, the run was used to profile the cool down T for the system. No characterization completed. M38

ST-38 Lowered Y temperature to 150°C. Increased Cu to 190. Sample was coated fairly uniform (brownish-black). Some backflow occurred. EDAX analysis. M39, M40

ST-39 Sample placed behind holder. No coating was seen on the sample or fibers. Cu extention tube clogged. No characterization completed. M41

- Changed Procedure to indlude weighing and cleaning of extention tubes.

ST-40 Same conditions as ST-39. Al_2O_3 and fibers showed discoloration and gained weight. Sample was at least 2" out of the hot zone. M42, M43

October 13-20, 1988

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-45	880	S-Y (TMHD)	125	50	0.15
	~6	S-Ba(TMHD)	253	100	0.95
		S-Cu(ACAC)	170	50	0.89
		O_2		100	

10/11 Al_2O_3 had a two color deposition of it; it was a dark brown/black color near the center and turned a light brown/tan near the edges. Under a light microscope the deposition was very smooth and even, except for a spotted region near one of the edges. The backside of the alumina was very clean. Al_2O_3 substrate broke while removing it. EDAX showed the Y:Ba:Cu ratio to be 1:1.61:2.31. M-55

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-46	890	S-Y (TMHD)	115~120	50	0.08
	~6.9	S-Ba(TMHD)	253	100	1.09
		S-Cu(ACAC)	170	50	0.72
		O_2		100	

10/12 Once again the Al_2O_3 broke into two pieces while it was being removed; one piece was lost and could not be found. The remaining piece did, however, have some light deposition on it which was mud-red in color. This coating was very smooth and did not have much texture at all. On the back of the substrate there were more of the black specks which had been noted in earlier runs. The extension tubes broke the Inconel wire which was holding them together presumably because the wire was too tight and/or too far back on the extension tubes. EDAX showed the Y:Ba:Cu ratio to be 1:1[illegible].71:3.57. M-56

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-47	870	S-Y (TMHD)	120	50	0.14
	~6.9	S-Ba(TMHD)	253	100	0.93
		S-Cu(ACAC)	170	50	0.66
		O_2		100	

10/12 Temperatures were all over the place, overshooting by as much as 15-20°C on each vaporizer. However, the Al_2O_3 picked up a non-uniform grey/black deposition on both sides. The coating on the front looked pretty splotchy, but was fairly uniform in some areas. The backside appeared to be a thinner, more uniform grey coating. The Ba vaporizer leaked, as it has for quite a while, and left a fairly big deposit on the Cu vaporizer. Also, the substrate holder developed a flaky coating on its back side. Cajon fittings

used to keep the tubes together and, for the most part, worked. M-61, M-63

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-51	895	S-Y (TMHD)	120	50	0.08
	~5.8	S-Ba(TMHD)	250	100	0.56
		S-Cu(TMHD)	150	50	0.66
		O_2		100	

10/19 M_8O show some slight discoloration near one corner but other than that was very clean. Al_2O_3 that was hanging behind the substrate holder did not show any discoloration at all. Sumitomo fibers did not show any discoloration at all. Questionable if tubes are being held tight enough together. Problems arose with the O_2 mass flow controller during the annealing which must have worked themselves out because it worked fine after an hour or so. No EDAX yet completed. M-64, M-66

white except for some light green and black tinges where they were exposed to the flow. Nextel 440 changed from orangish to white with no other discoloration. Nicalon fibers started black and finished black. Ba vaporizer was wrapped with two layers of teflon tape and it did not leak. This was the first run of a ten run series which varied flow rates. No EDAX yet completed. M-73, M-74, M-75, M-76, M-77, M-78

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-55	900	S-Y (TMHD)	130	100	0.14
	~8.4	S-Ba(TMHD)	250	200	0.83
		S-Cu(TMHD)	150	100	0.81
		O_2		100	

10/25 MgO pieces fell so that one partially covered the other, and thus, only one piece showed a coating. This piece had a brown film that was thin enough to see through. Its heaviest concentration was near the edges where it was in contact with the substrate holder. Al_2O_3 had a fairly heavy brown-orange coating on it where it was exposed to the flow. Run #2 using flow variance. No EDAX yet completed. M-79, M-80, M-81, M-82, M-83, M-84

October 31 - November 2

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
St-59	899	S-Y (TMHD)	135	100	0.12
	6.7	S-Ba(TMHD)	253	50	0.84
		S-Cu(TMHD)	150	25	0.72
		O_2		100	

10/31 MgO had a thin brown film, variable density, no discoloration of the Al_2O_3. No resistance across film at Room T. No characterization completed. M-100 to M-104

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
ST-60	898	K-Y (TMHD)	132	25	0.42
	7.4 torr	S-Ba(TMHD)	260	200	0.71
		S-Cu(TMHD)	159	25	0.50
		O_2		100	

11/1 MgO had a very thin, nonuniform deposit, brownish black in color. Fiber had a little discoloration. The Al_2O_3 had an orange-yellow discoloration. M-105 to M-109

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-61	900	K-Y (TMHD)	135	50	0.36
	35	S-Ba(TMHD)	253	100	0.35
		S-Cu(TMHD)	162	50	0.15
		*O_2		1000	

11/1 First vertical furnace run since Spring. See sketch. Several startup problems were worked out. Ran O_2 flow too high. Al_2O_3 under the hearth was coated on both sides (brownish purple). The strip of Al_2O_3 had a variable coating along its length. Cu and Y (compounds) crystals were growing in the vaporizer valve. Cooled under O_2 in vacuum, then under Ar at 1 atm. M-100 to M-112

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
ST-62	896	K-Y (TMHD)	139	25	0.52
	6.4 torr	S-Ba(TMHD)	258	50	0.79
		S-Cu(TMHD)	165	25	0.92
		O_2		150	

11/2 Lost every bit of Cu? Mgo had a dark, blackish, nonuniform coating. SiC fiber had a brown discoloration. Vaporizer temperatures were a little high. O_2 ran high, and we had to adjust the valve seat. No characterization yet. M-113 to M-117

November 3 - 10, 1988

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-64	895	K-Y (TMHD)	131	25	0.64
	7.8	S-Ba(TMHD)	250	200	0.89
		S-Cu(TMHD)	150	100	0.86

11/3 The MgO substrate had a very dark discoloration. The Al_2O_3 had an orangish color, and the fibers had very little discoloration. High Ba, high Cu, low Y flows. M-122 to M-126.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-65	900	K-Y (TMHD)	135	50	0.05
	23	S-Ba(TMHD)	253	100	N/A
		S-Cu(TMHD)	164	50	0.71

11/3 The hanging substrates (lower) had fairly thick, dark grey coatings. Al_2O_3 strip had light flow patterns along its length. Cooldown was under 1 atm O_2. Cu vaporizer was completely clogged. M-127 to M-130.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-66	900	K-Y (TMHD)	144	50	0.10
	23	S-Ba(TMHD)	253	100	0.32
		S-Cu(TMHD)	160	50	0.94

11/4 Completely overshot the Cu temperature to over 300°C. Y was also overshot. The hanging samples had a dark black coating. Resistance was measurable, at 250 ≫! The high overshoot may have caused excess Cu. Also Ba pressure was ~20 psi when we opened the valve. M-131 to M-134.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-67	900	K-Y (TMHD)	146	50	0.14
	22	S-Ba(TMHD)	254	100	0.33
		S-Cu(TMHD)	164	50	0.75

11/7 MgO hanging below had an orangish-red coating. The Al_2O_3 had the same color with a smooth and even texture. No visible coating was seen on the long Al_2O_3 strip. The Cu valve was clogged completely even though we insulated the valve. M-136 to M-139.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-68	900	K-Y (TMHD)	138	100	0.17
	8.5	S-Ba(TMHD)	254	200	0.74
		S-Cu(TMHD)	156	25	N/A

11/7 MgO had a slight thin even blackish coating. The Al_2O_3 piece had very little coating. The fibers had no visible discoloration. (Note: 9th run in 10 run study.) M-140 to M-144.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-69	900	S-Y (TMHD)	145	50	0.11
	20.5	S-Ba(TMHD)	255	100	0.29
		S-Cu(TMHD)	164	50	0.76

11/8 MgO was put on the hearth; it's too hard to hang below. The hearth fell during the run, giving an irregular coating on the substrates. The hanging Al_2O_3 was partially black in color. M-150 to M-153.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-70	900	S-Y (TMHD)	130	100	0.45
	7.5	S-Y (TMHD)	255	50	0.50
		S-Y (TMHD)	155	100	0.88

11/8 MgO had a slight cloudy grey deposit; the coating was very thin and you could see through it. The fibers had no discoloration. M-145 to M-149.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-75	878 6.6	S-Y (TMHD) S-Ba(TMHD) S-Cu(TMHD) O_2	123 254 150	25 25 200 100	0.48 0.50 0.71

11/15 Ran high Cu, low Y, Ba flowrates. MgO sample was brownish-coating appeared fairly thick. M-169.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-76	900 17.8	S-Y (TMHD) S-Ba(TMHD) S-Cu(TMHD) O_2	132 255 160	50 100 50 100	0.10 0.29 0.61

11/15 Ran oil bath again at 120°C, injector was at 280°C. Cu did not clog. MgO was brick red--when checking the lines, we located a problem with the O_2 flow and corrected it. Since this sample and other samples are oxygen deficient, we are annealing them. M-170 to 173.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-77	900 17.2	S-Y (TMHD) S-Ba(TMHD) S-Cu(TMHD) O_2	134 252 153	50 100 50 100	0.80 0.26 0.55

11/16 Y controller had a 15° variation in temperature. MgO sample was black with a few orange spots. The coating was fairly thick. None of the vaporizers clogged. M-174 to 177.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-78	900 14.4	S-Y (TMHD) S-Ba(TMHD) S-Cu(TMHD) O_2	148 253 156	50 100 50 200	0.73 0.33 0.68

11/16 Y overshot temperature by 50°C. Both Y and Cu valves clogged. Ran double O_2 to ensure enough oxygen for the reaction. MgO was mainly black and brown and very thick. M-178 to 181.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-82	900	Y	130	200	.06
	22.6	Ba	254	400	.37
		Cu	160	50	.56

11/22 Samples were orangish in color due to an O_2 deficiency that resulted when the O_2 line was melted by the oil bath lines. Cu vaporizer showed an increase in pressure due to a clog that resulted in the Cu vaporizer. Samples need to be annealed. Oil temperature started out at 150°C but was later reduced to 125°C because of a high injector temperature. EDAX showed Y:Ba:Cu to be 1:0.41:7.60. M-195, M-196, M-197, M-198

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-83	.5 hr	Y	-	50	-
	900	Ba	-	100	-
	11.3	Cu(ACAC)	-	50	-

11/22 First run external vaporizers horizontal furnace. (See diagram for set.) Unsure of vaporizer temperatures because the thermocouples did not record vaporizer temperatures. O_2 line melted during run and had to be replaced. Y and Cu vaporizers seem to be blowing powder up the stem. System has major leaks in it. MgO was coated with a light aqua-green coating and had increased density near the edges. Vaporizers were a mess and hard to clean. No EDAX completed. M-194

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-84	900	Y	152	100	.05
	14.2	Ba	218	200	.23
		Cu	176	50	.06

15 minutes run

11/28 Run aborted when injector temperature reached 450°C after flows were started. (Run started early when injector temp reached 450°C without flows) MgO had three distinct colors of discoloration on it: orange, brown and maroon. The maroon area was rough and uneven; other areas were smooth and even. Al_2O_3 (sh) had orangish discoloration on the lower half of one side. Al_2O_3 (lh) had some light orangish discoloration on the lower portion of the strip. Seven new carbon felt pieces had been added to the tip of the injector to bring the total number to 16 in addition to the soffil. Oil set to 250°C, however, never reached set temperature. No EDAX completed. M-199, M-200, M-201, M-202

flow patterns visible on it. during the run the hearth had broken from its place and was sitting in the cone parallel to the flow. As a result, the Al_2O_3 pieces were irregularly coated, it at all. M-209, M-210, M-211, M-212

	12	Ba	250	400	.07
		Cu(ACAC)	150	50	.14

12/8 Helped some of the temperature control by wrapping the vaporizers better. MgO had a thin black coating; denser at the edges. Getting a better feel for the setup and take down.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-94	900	Y	130	50	.01
	20.8	Ba	250	750	.29
		Cu(ACAC)	150	25	.05

12/9 Used graphite (SiC coated) extension tube on the top of the injector. Using ½" ball valve for Ba vaporizer. Injector T at start was hot ~450 C. MgO was black and thick, but not very even. Running maximum Ba flowrate to make sure we can deposit Ba in this furnace.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-95	900	Y	130	100	.07
	22.6	Ba	250	650	.36
		Cu(ACAC)	150	30	.07

12/12 Based on S-94 results, our guess at flowrates for 123 deposition. Waiting for Ba to do box runs. MgO was black with both specks and flow patterns present. Injector T again net ~450 C during heatup.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
ST-96	900	Y	130	100	.07
	15.8	Ba	250	550	.07
		Cu(ACAC)	164	40	.03

12/13 The MgO substrate fell either before or during the run, and had a thin blackish coating.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-97	900	Y	140	100	.2
	21	Ba	253	550	.29
		Cu(ACAC)	150	40	.08

12/13 Another best guess at flowrates before receiving the 10g $Ba(tmhd)_3$. The MgO had a blackish coating with a greenish area. No resistance could be measured. The cones had to be repainted with SiC after the run.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-102	900	Y	130	200	0.07
	23.6	Ba	250	750	0.31
		Cu	150	10	0.19
		O_2		200	

12/19 Run had to be restarted due to a Ba tape burnout. Using a 4' tape and more insulation, MgO coating was thick and black. Resistance was in the MΩ's.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-103	900	Y	130	50	0.06
	21.0	Ba	250	750	0.43
		Cu	150	10	0.18
		O_2		200	

12/20 Again, the Ba heating tape burned out and was replaced. The MgO was thick and black. You could see the wire lines outlined in the coating. The Sumitomo fell out during the run. There was MΩ resistance on both sides.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-104	900	Y	130	100	0.12
	19.8	Ba	250	500	0.35
		Cu	150	25	0.24
		O_2		200	

12/20 The MgO substrate had a black splotch on it. The Hearth had slipped sideways during the run. The Sumitomo looked good and both were given to Dr. Hill for analysis.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-105	900	Y	130	200	0.08
	21.3	Ba	250	750	0.36
		Cu	150	50	0.28
		O_2		200	

12/22 MgO coating was very thick - black and silverish. The resistance was <1MΩ. The oil bath has developed a slight leak out the back and needs attention.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-111	900	Y	130	200	0.08
	21.6	Ba	250	250	0.18
		Cu	150	10	0.29
		O_2		200	

1/6 The MgO fell out of the basket at some point during the run. The coating was black with an orange tint. The hearth also moved parallel to the flow during the run. The resistances were in the MΩ's.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
ST-112	900	Y	150	100	0.07
	16.1	Ba	220	500	0.1
		Cu	160	50	
		O_2		200	

1/6 The horizontal furnace was used to run Ba(hfa) reagent. Flows were determined using a Japanese paper. (Ba conditions 500 cc/min Ar, 220°C) The MgO was a uniform blackish gray.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-113	900	Y	130	200	0.08
	21.6	Ba	250	250	0.30
		Cu	150	50	0.56
		O_2		200	

1/9 The MgO had a black coating (R ≅ MΩ's) on half of the sample - an orangish coating on the other half. The Sumitomo was also blackened during the run.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-114	900	Y	130	50	0.15
	20.2	Ba	250	250	0.31
		Cu	150	10	0.21
		O_2		200	

* Duplicating S-107

1/10 The MgO substrate had a black, thick, even coating. No resistance was observed. The Sumitomo also had a good black coating. The Y vaporizer T varied from 130 to 155°C during the run.

January 12 - 20, 1989

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-116	900	Y	136	50	not
	13	Ba	265	100	reported
		Cu	177	50	
		O_2		200	

1/12 Horizontal run using the same conditions as ST-85; the Japanese conditions. MgO had a uniform black coating on the surface facing the flow.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-117	900	Y	131	200	.08
	22.3	Ba	257	500	.26
		Cu	150	25	.25
		O_2		200	

1/12 Midpoint of box with double the Y flow (200). Resistance was ~7 MΩ. MgO had a thin black/orangish coating. The Sumitomo had a black coating.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
ST-118	900	Y	140	50	0
	11.2	Ba	248	100	1.12
		Cu	160	50	0.13
		O_2		200	

1/12 Repeated ST-85; Japanese runs. MgO had a thin brownish coating. Infinite resistance was measured. A large amount of Ba was lost, and no Y was lost. Possible error in weight measurements.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-119	900	Y	135	.08	
	22.8	Ba	255	500	.23
		Cu	150	25	.25
		O_2		200	

1/13 Started new Y (made by Kent). Repeat of S-117. MgO had a thin black spotty coating; Sumitomo had a black coating on it. At some point during the run, the hearth turned vertical.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
ST-124	900	Y	126	100	.02
	90	Ba	237	400	.11
		Cu	145	25	.26
		O_2		200	

1/18 The conditions were again matched to the vertical run from S-123. By accident, the carrier gas lines were not hooked up. The pressure during the run was op torr. Downstream, the lines were hot. MgO was dark black; resistance was ~300 KΩ's.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-125	900	Y	140	100	.06
	21	Ba	255	400	.22
		Cu	156	25	.14
		O_2		200	

1/19 (15 min. run) Flowrates were midpoint with slightly less Ba flow. Run to compare with the 1 hour runs. The MgO had a poor coating; only 1/8 of the surface was coated black.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
ST-126	900	Y	146	100	.02
	15.1	Ba	250	400	.14
		Cu	155	25	.14
		O_2		200	

1/19 Ran Ar through Y vaporizer during heatup for 10 minutes. Ran same conditions as S-125. The MgO sample slid out during heatup and had no coating; it will not be analyzed.

Run	System Temp & Pressure	Reagent	Reagent Temp	Flow	Amount Lost
S-127	900	Y	140	100	.11
	22	Ba	285*	700	.42
		Cu	153	25	.33
		O_2		200	

1/20 *Ba T reached 336°C probably a fluke because of a bad thermocouple extension wire connection. A high Ba flow was run since no Ba was found on earlier sample. The MgO had a thick black coating. Resistance was in the 100's of KΩ's.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-132	900	Y(Ar)	131	100	n/a
	20.3	Ba	253	500	.25
		Cu(Ar)	151	25	n/a
		O_2		200	

1/26/89 Ran only Ba vaporizer. Everything was set up the same. Put Al_2O_3, MgO, and a previous MgO run that was Ba poor in the furnace. A glassy coating, brownish/yellow in color was observed. Coatings were uneven. A white coating was seen inside the upper graphite chamber (BaO?).

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-134	900	Y	131	100	.05
	20.3	Ba(Ar)	253	500	n/a
		Cu(Ar)	148	25	n/a
		O_2		200	

1/27/89 Ran only Y vaporizer with everything set up the same. The extra Ar line (150 cc/min) was not heated during the first 40 minutes. MgO piece M-242 was put in the run along with a new MgO piece. M-242 had a high Ba and Cu content.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-136	900	Y	131	100	.03
	21.6	Ba	252	500	.22
		Cu(Ar)	150	25	n/a
		O_2		200	

1/30/89 Only Ba and Y vaporizers were run with two MgO and Al_2O_3 substrates. The coatings were thin and white. A rainbow spectrum was seen on one of the MgO samples. The Al_2O_3 had some green-orange dots.

Run	System Temp & Pressure	Reagent	Reagent Temp	Reagent Flow	Amount Lost
S-137	900	Y	130	75	.05
	24	Ba	250	750	.32
		Cu(Ar)	150	25	
		O_2		200	

1/31/89 Same as S-136 except a higher Ba flow was used. The Ba heating tape burned out but was replaced before the run started. The run was stopped 10 minutes short due to decreasing oil in and out temperature. Substrates had thin, white, translucent coatings.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-133	1:1:1	-	900	50	0.25

Flowrates
Ar: n/a - run made on horizontal furnace
O_2:

Anneal: Backfill and cool with pure O2

Date: 1/26/89

Comments: This was a test run to check the effectiveness of the powder feeder. MgO had a uniform coating, thin and transparent. Tried to locate leaks.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-135	2:1:1	-900		0.25	

Flowrates
Ar: n/a - run made on horizontal furnace.
O_2:

Anneal: Backfill and cool with pure O2

Date: 1/29/89

Comments: Similar to ST-133

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-139	1:2:1	-	900	60	0.25

Flowrates
Ar: n/a
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 2/2/89

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-145	1:1.5:2	-	900	73.5	6.6

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 2/7/89

Comments: Started using screen -- powder size = 149μm. Used only Al_2O_3 substrate. Plumbed an additional O_2line to furnace to avoid tee and minimize powder backflow. Run lasted 30 minutes; most of the powder was used early on. Thick black coating, 15 KΩ's, was observed.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-146	1:1.5:2	-	900	73.5	6.6

Flowrates
Ar: 0.15 ℓ/min
O_2: 0.75 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 2/8/89

Comments: Developed an MgO holder to hang a sample near where the Al_2O_3 strip was placed. A thick black coating on half the substrate was observed. Resistance = 600KΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-147	1:1.75:1.25	-	900	74.0	6.6

Flowrates
Ar: 0.15 ℓ/min
O_2: 0.75 ℓ/min

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-150	1:2:2	-	900	26.4	7

Flowrates
Ar: 0.15 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: First SS chamber run. Quiet and clean, but you can't see the powder. The MgO had a light coating. Fibers had no coating. Postulate that you need some gas assisting powder down the tubes before the tee. Also may need more O_2 flow.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-151	1:2:2	-	900	26.8	10

Flowrates
Ar: 0.15 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Ran the system with the O2 flow going directly into the chamber. MgO had a thick, uniform black coating, 0.6 20 MΩ's. Fibers were also coated.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-152	1:2:2:	-	900	47.4	6.5

Flowrates
Ar: 0.15 ℓ/min
O_2: 5 ℓ/min

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-155	1:22.25	-	900	48.5	6.5

Flowrates
Ar: 0.15 ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Repeated ST-154. MgO had a thick, uniform black coating (KΩ range). Fibers had a light brownish coating. Replumbed the system to allow for higher O_2 flows.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-156	1:1.75:2.25	-	900	48.2	6.5

Flowrates
Ar: 0.15 ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Used new cone; new painted chamber. First run with new, desiccated Ba, M-323. MgO had a thick black coating in the KΩ range. Fibers were coated. The run was cut short (25 min.) because the % power dropped from 20 to 17%. Problem with the SCR. Had to order a new part. Switched to H-furnace.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-157	1:1.75:2.25	-	914	30.7	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Plumbed O_2 line from V to H furnace to get high flow. Used large feed (4g). Feed path cut to 1/2 along ledge in order to control rate. Ran at fastest setting to insure movement. MgO was thick, black, resistance form 1000 to 59Ω.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-158	1:1.75:2.25	-	905	31.1	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Duplicate ST-157. MgO was extremely thick, with corner to corner 200 Ω's. Lost most the powder in 30 minutes.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-159	1:1.75:2.25	-	911	30.1	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: 5 minute run seeing if we can control feed rate. We still lost ≈4g, so we have to restrict the powder path more. MgO was thick and black; 300 to 1000 Ω's. Fiber had a thick black

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-162	1:1.75:2.25	-	906	31.2	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Put in a second reduction in the feeder path near the base of the ramp. The coating was lighter than before, the 30 min. run lost 3.8g. Resistance was 500-1000Ω corner to corner.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-163	1:1.75:2.25	-	897	30.4	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: 5 minute run with similar setup as St-162. (Changed O_2 tank) MgO had very light, uniform black coating. Resistance was 1-3 KΩ corner to corner. Fiber was lightly coated. Lost only ≈1g of powder.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-164	1:1.75:2.25	-	912	30	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-167	1:1.75:2.25	-	900	47	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Vertical is fixed. Moved feeder over. MgO has a very thick coating with resistance in the KΩ's. This 5 minute run lost 2.44g. This doesn't compare to the horizontal furnace feed rate of 1g.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-168	1:1.75:2.25	-	900	53	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Ran 30 minutes, lost 4.35g (again higher than in the horizontal furnace). Suspended a Sumitomo tow down center of graphite chamber. Good coating at the ≈ position of the substrate in our typical setup. Light brown at top.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-169	1:1.75:2.25	-	900	54	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-172	1:1.75:2.25	-	914	30.6	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: 5 minute run using powder ≥ 44 μm. Light coating was observed in the MgO with 10KΩ resistance. Sumitomo and Nicalon were lightly coated.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-173	1:1.75:2.25	-	914	30	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: 15 minute run checking the 44 μm powder. MgO and Sumitomo were coated dark black.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-174	1:1.75:2.25	-	906	31.2	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: 30 minute run. MgO was black and uniform - 700 to 900Ω. Sumitomo and Nicalon were lightly coated.

Comments: Ran without path restrictors. Only ran 10 minutes. (We expected to lose most of the powder in a few minutes). Resistance on MgO was 1-5KΩ's, so we didn't duplicate our early success.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-178	1:1.75:2.25	-	906	26.5	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Repeated ST-177 with only 5 minute run time. Verified loss of powder in a few minutes. Resistance on MgO was 1-5KΩ's, so we still can't duplicate early success.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-179	1:1.75:2.25	-	914	27.6	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Ran for 7 minutes. MgO was thick, black, with a resistance of 1-5KΩ's. Fibers were also coated thick

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-180	1:1.75:2.25	-	907	28.7	10

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-183	1:1.75:2.25	-	912	28.2	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Ran for 30 minutes. Annealed in 5 1/min O_2 under vacuum for 30 minutes at 900°C. Resistance was 500Ω's. Coating was dark black and looked good. Need to anneal!

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-184	1:1.75:2.25	-	909	29.2	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Repeated ST-183 with similar results. Sumitomo and Nicalon looked good. Annealed for 30 minutes.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-185	1:1.75:2.25	-	912	28.1	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-186	1:1.75:2.25	-	910	30	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Anneal 50 minutes. Ran MgO and $SrTiO_3$ at the same time. Coatings looked fairly thick and black. Resistances were 50-90Ω for the MgO; 500Ω for the $SrTiO_3$. A large amount of black powder was present between the O_2 line and the sample in the tube.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-187	1:1.75:2.25	-	910	28.6	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Anneal 1 hour. Run completed to duplicate earlier results. MgO had a thick, black coating; resistance = 150-200Ω. The Sumitomo and Nicalon looked good. Note: Only a 15 minute run to vary thickness.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-188	1:1.75:2.25	-	895	26.9	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-191	1:1.75:2.25	-	910	29.4	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Anneal 1 hour. Cu(acac) // Calculated the proper moles of Cu(acac) to be input and ran for 30 minutes., Resistance was 500-1000Ω. Fibers were lightly coated black from the process.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-192	Y only	-	915	28.5	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Anneal 1 hour. Given to Jack, only put $Y(tmhd)_3$ in the bowl. Ran for 15 minutes. A greenish coating was observed on the Al_2O_3 and Sumitomo fiber.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-193	1:1.75:2.25	-	900	49	10

Flowrates
Ar: ℓ/min
O_2: 10 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-196	1:1.75:2.25	-	900	85.2	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min (10 help carrying of powder to furnace)

Anneal: Backfill and cool with pure O_2

Date:

Comments: Anneal 1 hour in horizontal furnace. Same as ST-195 except we used 10 1/min O_2 to help carry the powder. The post anneal resistacne was 250Ω. The fibers were black with a uniform coating.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-197	1:1.75:2.25	-	900	97	10

Flowrates
Ar: 1,5 ℓ/min
O_2: 5 (used 10 1/min Ar directly to furnace) ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: Anneal 1 hour in hodrizontal furnace. Ran using Ar carrier instead of O_2 carrier. The cone was broken in half either near the end of the ru n, or during cooldown due to O_2 eating the graphite. No visual damage to the heating element. MgO resistance was 20-60Ω's. Maybe Cu rich?

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-198	1:1.75:2.25	-	900	69	10

Flowrates
Ar: 1,5 ℓ/min
O_2: 15 ℓ/min

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-201	1:1.75:2.25	-	912	22.8	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: Anneal 1 hour under vacuum. Put in a series of fibers including Nextel, Sumitomo, Nicalon, and Saphikon along with a MgO substrate. Resistance on the MgO was 3MΩ's. The pressure was ≈10 torr lower than before, so we'll repeat this run at a higher pressure.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST202	1:1.75:2.25	-	915	26	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 4/5/89

Comments: Ran several substrates including polycrystalline Al_2O_3, Nextel mesh, and several fibers. The Al_2O_3 had resistance of 500-4000Ω. The coating looked uniform and black. Note: Started skimping on reagents (only 4g/run).

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-203	1:1.75:2.25	-	915	24.3	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-206	1:1.75:2.25	-	900	123.5	10

Flowrates
Ar: 10 ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: Annealed in the horizontal furnace. MgO was coated black and had a "pile" of coating on the surface. Samples were organgish-black before annealing and black afterward.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-207	1:1.75:2.25	-	900	110	10

Flowrates
Ar: 10 ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: The MgO before and after annealing was black and uneven. Resistance was 5-5-KΩ's. We're starting to lump up the coating right above the injector - may need to get a gas diffuser.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-208	1:1.75:2.25	-	914	26.1	10

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

ST-211	1:1.75:2.25	-	900	65	5

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: MgO was only slightly coated - we slowed down the feeder rate and carrier flow. Had Nicalon, Sumitomo, and AVCO fibers - only the Sumitomo looked a little organish. No annealing completed. Used acetates.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-212	1:1.75:2.25	-	900	61.1	4

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: A very poor run - nearly a redo of ST-211; used acetates. The powder still clumped on the MgO above the injector. The run was cut short ofr 20 minutes.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-213	1:1.75:2.25	-	900	78.2	10

Flowrates
Ar: 5 ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: Ran wiht Y and Cu tmhd and Ba acetate. Didn't lose as

Anneal: Backfill and cool with pure O_2

Date:

Comments: First attempt at liquid feeding of reagents. The capillary plugged and the run was stopped. Noticed puddles of reagent and liquid on the glass just below the spray.

Anneal: Backfill and cool with pure O2.

Date:

Comments: Cl_2 gas formed and flooded the room - i.e., the O_2 line was not hooked up. Need to be more careful with the setup. No coating observed.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-220	1:1.75:2.25	2.06	900	106.5	10

Flowrates
Ar: 10 ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: Tried the gas diffuser again in the vertical furnace. The MgO was sparsely coated - no resistance was measured. The SiO_2 middle piece was coated more than anything.

Run	(Y:Ba:Cu) Reagent wt Ratio	Liquid Carrier	Temp (°C)	Pres	Feed Rate
STL-221	≈1:1:1	CCl_4	905	751	30 ml @ 1 ml/min

Flowrates
Ar: 500 cc/min
O_2: ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: No coating observed - a yellowish deposit was noticed near the exhaust end of the tube. Still having problems with the atomizer flow during the run.

Date:

Comments: Reduced carrier and feeder rate MgO measured ≈100 Ω's and was a uniform black coating., The gas diffuser appeared to be working okay. (Only ran for 25 minutes.) Also moved the quartz inner chamber lower and insured a gas tight seal for the powder/gas feed. No SiO_2 spaces in the gas diffuser.

Run	(Y:Ba:Cu) Reagent wt Ratio	Liquid Carrier	Temp (°C)	Pres	Feed Rate
STL-225	1:1.75:2.25	H_2O	905	570	30 ml @ 1 ml/min

Flowrates
Ar: 500 cc/min
O_2: ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: Liquid run with acetates and water - tried various pressures throughout the run. A light coating was observed on the Al_2O_3 substrate.

Run	(Y:Ba:Cu) Reagent wt Ratio	Liquid Carrier	Temp (°C)	Pres	Feed Rate
STL-226	1:1.75:2.25	H_2O	904	669	33 ml ml/min

Flowrates
Ar: 500 cc/min
O_2: ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: Liquid run with acetates - tried an intermediate vacuum to try to maintain spray and avoid freezing. Used a heating tape around the inlet end to assist in heating the reagent.

Date:

Comments: Reworked the system - Ar is used through the bowl, O_2 added downstream, and 2 grounding wires are attached. The unannealed MgO sample had R=500 KΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-230	1:1.75:2.25	4.12	898	33.8	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: Tested the system and annealing schedule. Annealed at 900°C, 1 ℓ/o_2 for 5 minutes, then cooled to 450°C and held for 1 hour. Resistance in the MgO was 2-3 MΩ's. Coating was gray and smooth.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-231	1:1.75:2.25	4.8	916	26.2	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: Same as St-230 except we held the 1st anneal at 900°C for 30 minutes. The MgO looked smooth and black. Resistance was 40 Ω's. We need to hold at 900°C for at least 30 minutes.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate

minutes, 760 torr, 1 ℓ/min O_2 at 900°C, then at 450°C for 1 hour. MgO fell out of holder. No resistance. Sumitomo and Nicalon are coated black and thin.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-235	1:1.75:2.25	n/a	916	26.8	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: The O_2 ignited again - processing error. The bowl was backfilled with O_2 at 900°C following the run instead of Argon. MgO was coated black. Resistance was 600-1000 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-236 #1	1:1.75:2.25	3.06	918	30.9	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: MgO was black/charcoal with resistance of 150-450 Ω's. This is the 1st run in a series of composition variations. A more complete summary is elsewhere. These runs use Kent Barefield's reagents. $Y(tmhd)_3$ M-569, $Ba(tmhd)_2$ M-598, $Cu(tmhd)_2$ M-597.

Anneal: Backfill and cool with pure O2.

Date:

Comments: MgO was smoothly coated black\dark gray. The colors were variable. Resistance was 300 KΩ's and was variable.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-240 #4	1:1.73:2.33	6.21	911	25.4	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: The MgO was dark gray and had black spot "freckles" over the substrate. Deposition thickness looked uneven. Resistance was 80-160 Ω's. The endcap powder inlet tube was clogged with black powder and needs cleaned regularly.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-241 #5	1:1.38:1.51	5.15	918	15.2	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2.

Date:

Comments: The MgO was coated gray/black which was smooth and even. The resistance was around 45-90 Ω's. Before this run the endcap line was changed and the powder lines were blown out.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-244 #1 redo	1:1.75:2.25	3.79	919	14.8	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date:

Comments: The MgO was grayish black with a slight density variation toward one corner. The resistance was 15-50Ω's, which is much lower than the 150 to 450 Ω's from the 1st #1 run. We are worried about reproduciblity.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-245	1:1.75:2.25	4.78	900	126	10

Flowrates
Ar: 5 ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date:

Comments: We had the quartz insert remade. 1st vertical runs in 2 weeks - picked identical conditions to ST-244. The MgO had rough black coating with resistances ranging from 150-400 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-246	a:0.95:1.21	4.92	915	23	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

MgO was rough and uneven - with a big mound of deposit. The resistance was 100-300 Ω's on the front - 150 to 200 Ω's on the back side!

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-249 #8	1:1.05:2.02	4.43	917	24	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date:

Comments: The MgO was very thick and black with resistances from 80-140 Ω's. This was a new point of the composition variation.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-250 #9	1:0.79:1.35	n/a	922	23.4	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date:

Comments: The MgO fell out of the sample holder during the run and had only sparse, translucent coating.

Powder Feed Runs

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-251	1:0.79:1.35	6.49	918	23.6	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date: 6/12

Comments: Repeat of ST-250 (point 9) since the MgO sample fell. Resistance was 70-120 Ω s. The sample was black and uniform.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-252	1:0.71:0.96	3.71	914	24.5	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date: 6/12

Comments: Point 10 and final point in composition variation experiment. Extremely high Y amount. Sample had a smooth gray/black coating - Resistance was 5 MΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-253	1:0.95:1.21	5.43	900	76.4	10

Flowrates
Ar: 5 ℓ/min
O_2: 5 ℓ/min

Comments: Same as S-254 except MgO is parallel to the flow. The sample had an extremely even coating but it was fairly thin and almost translucent. Resistance was 1-2 KΩ.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-25[illegible]	1:1.18:1.68	5.39	900	86.5	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date: 6/15

Comments: Same conditions as ST-242. Vertical run with MgO parallel to the flow. Both sides were coated a thin black - definitely not as thick as perpendicular positioning. Resistance was 80-150 Ω's on both sides.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-257	1:1.38:1.51		918	15.9	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date:

Comments: Repeat of the "best" run from the study. Uniform coating (black) was observed on the MgO substrate. The resistance was 20-30 Ω's corner to corner.

Comments: Run was stopped after 20 minutes due to a clog in the gas lines. First multiple MgO run. Set up 5 substrates ≈1" spacing. All the substrates were coated black - no variations in coating thickness was seen. Resistances corner to corner varied from 20 to 250 Ω in no distinct order.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-261	1:1.38:1.51	6.64	919	14.8	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Blew out the powder feed lines before the run - will repeat before every run. Again put in Saphikon - but it was lost during the run. MgO resistance was 10-14 Ω's. It took 90 seconds to backfill system with Argon before the O_2 anneal.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-262	1:1.38:1.51	5.37	911	13.2	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Multiple MgO runs. A variation in coating was noted; however, all substrates were coated black. Deposition looked a little "clumpier" on the back 2 away from the inlet. Resistances ranged form 50-80 KΩ's at the back position at 110 KΩ's at the front. Some question of sample placement was discussed. They may have been 2" from the normal spot.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-264	1:1.38:1.51	4.96	912	10.7	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date:

Comments: Multiple MgO run. The deposition appeared to vary in thickness based on position. The front MgO was flaky - the back was translucent. The colors varied from grey to black. The cooling waster was left off during the run. Resistances ranged from 180 to ∞ Ω's, but with no real pattern.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-265	1:1.38:1.51	4.86	970	16	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2. ½ hr 900°C, 1 atm, 1 l O_2/min

Date:

Comments: Note: higher temperature. Massive problems - running MgO & Saphikon. The powder lines plugged twice. We speculate that the previous run had deposits which had to be scraped out of the inlet tube. Saphilon recovered - MgO was black with resistance = 45-100 Ω.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
St-266	1:1.38:1.51	4.82	911	14.4	10

Flowrates

950°C, so the processing was at 910°C. The $SrTiO_3$ doubled its weight, and the resistance was 70-100 Ω's. The Saphikon looked good. A thermocouple was inserted at the gas inlet end to provide a better temperature measurement. The multimeter read ≈7 during the run.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-269	1:1.38:1.51	5.83	920	18.6	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date: 7/19/89

Comments: Ran MgO & Saphikon. Backfilled with (O_2 instead of Argon. Annealed using the same procedure. Substrates were black and uniformly coated. Resistance was 90-120 Ω's. a lot of powder had deposited on the tube walls.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-270	1:1.38:1.[illegible]	5.83	920	18.6	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2. ½ hr, 900°C, 1 atm, 1 l O_2/min

Date: 7/20/89

Comments: Ran MgO, Al_2O_3, flat Ag substrates. All were coated black. The silver had resistance of 70-100 Ω's. the MgO and Al_2O_3 did not get coated as well, and their resistances were 130-500 Ω's and 1000-5000 Ω's respectively.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-273	1:1.38:1.51	6.48	919	16.7	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 7/25/89

Comments: Ran Saphikon and Ag wire. Now there are 2 pieces of Ag wire coated. The fibers were coated thick and black on the areas exposed to the flow.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-274	1:1.38:1.51	6.44	919	16.5	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 7/25/89

Comments: Ran polycrystalline ZrO_2 (M-218) for the first time. Coating was thick and black - some areas looked darker and grainier than others. The resistance was 30-50Ω's.

7/26/89 A check of the feed rate using atmospheric conditions with no gas flow indicated that we lose ≈90% of the powder in ≈5 min at the feed set of 10. We determined that feeding at 7 to 10 minutes followed by 10 for 10 minutes uniformly moved the powders.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-275	1:1.38:1.51	6.59	920	17.8	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Observations with the new pot: A spark was observed coming form the powder feeding area, the rate was fairly constant. Even though the feed rate was 10, we did not lose all the powder. We lost ≈75% in 15 minutes. The rate slowed as the powder amount decreased. Some problems occurred. 1) The vibrator fell off the neoprene pads and was not level. 2) The Lexan cannot be cleaned with Acetone, methanol works all right.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-278	1:1.38:1.51		500		10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/2/89

Comments: Annealed at 500°C. Ran a diamond substrate from D-29, low temperature. Annealed at low temperature. Sample had high resistance (MΩ's) and was multicolored (black to uncoated).

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-279	1:1.38:1.51	13.75	900		10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/4/89

Comments: *Prior to ST-279, the water lines for the system were changed, the Inconel tube was cleaned, a new end cap and powder lines were used (since we were running acetates). The scrubber was also cleaned out.

Ran Y, Ba, Cu acetates. (Higher weight to achieve same powder volume.) The powder feeds very fast. Only ran for 5 minutes. MgO was half coated black. Resistance was 200-500 KΩ. A large amount of powder was found in front of the sample holder.

Comments: Same run with acetates with lower flow and lower feed rate set. The MgO looked better than previous samples, but was still rough and grainy. Resistance was 20-40 MΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-283	1:1.38:1.51	9.59	900	10.4	7-7.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/9/89

Comments: Repeat of ST-282 with acetates. The run was stopped early due to excessive noise from the chamber. Sample looked poor. Following this run, the system was cleaned again for use with tmhd's. Replaced end cap and lines. New $Y(tmhd)_3$ was obtained from Kent.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-284	1:1.38:1.51	7.04	906	24.7	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/14/89

Comments: Ran MgO to duplicate earlier ST-241 run. Heated up 2 zones of the furnace. Sample was further in. Thermocouple placed in furnace. Lexane top replaced. Noted T rising during powder feeding. MgO was shiny, smooth and grey/black. Resistance was 20-40 Ω's.

Compare with ST-285 regarding annealing.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-288	1:1.38:1.51	6.71	908	22.9	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/16/89

Comments: Another fiber run using the quartz holder. Ran Sumitomo, Nextel, and Nicalon. A thin grey/black coating was observed - heaviest on the ends of the fibers.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-289	1:1.38:1.51	6.71	890	22.6	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date:

Comments: Repeat of preferred conditions -- MgO had a black coating 40-80Ω's. Samples from ST-285 and ST-287 were annealed following this run. Resistances were 50-80 Ω's and 1000 Ω's respectively.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-290	1:1.38:1.51	4.23	885	22.2	7-10

Flowrates
Ar: 5 ℓ/min

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-293	1:1.38:1.51	5.15	910	23	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/22/89

Comments: Chose another point from the input composition study. The MgO was coated black, thick, and even with a resistance of 80-160 Ω's. The Sumitomo was coated also, with some thickness variations noted.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-294	1:1.38:1.51	6.49	895	1atm	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/23/89

Comments: Started the T,P variations study. Samples are annealed for 30 min under O_2 at their processing temperatures. The MgO had a resistance of 4.5 KΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-295	1:1.38:1.51	5.32	895	1atm	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/24/89

O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/25/89

Comments: The MgO had a good, thick, uniform black coating with no measurable resistance. Sumitomo was thick, black and uniform.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-299	1:1.38:1.51	5.33	470	23.2	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/28/89

Comments: The MgO was thick, black, and uniform with no measurable resistance. This low T run in the T,P variation. The Sumitomo was also coated uniform and black.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-300	1:1.38:1.51	7.69	680	0.5 atm	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/29/89

Comments: Standard anneal at 700°C. The MgO had a thick black - dark brown coating with a translucent brown ring around the outside. Resistance was 2-6 MΩ's. Sumitomo was coated dark brown.

Date: 8/30/89

Comments: Annealed at 700°C for 30 minutes. Part of T,P study. The MgO appeared black with some orangish color and very rough. Resistance was 6 MΩ's. All powder fed in 13 minutes.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-304	1:1.38:1.51	6.65	480	0.5 atm	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/1/89

Comments: Annealed at 500°C for 30 minutes. The MgO was coated a deep black, smooth and uniform. The Sumitomo was also coated black across the entire sample. Resistance was 2 MΩ's for this T,P point.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-305	1:1.38:1.51	7.79	475	22.6	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/1/89

Comments: Used substrate from D-37; processed and annealed at 500°C. The substrate had a thick, uniform black coating. Unmeasurable resistance.

Comments: Ran one of the Ag 1" disks. Powder fed completely in 10 minutes. The sample looked good ≈2 KΩ resistance. There was a ¼" circle of rougher and darker coating on the sample.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-309	1:1.38:1.51	6.45	8.75	16.3	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/6/89

Comments: The MgO had a smooth uniform coating, but the resistance was 9 KΩ in this duplicate of ST-241. We have been starting the anneal as soon as the powder is gone and leaving the feed rate set at 10. We should change this procedure.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-310	1:1.38:1.51	5.96	895	24	7.75-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/7/89

Comments: Varied the feed rate set to ensure powder fed for at least 20 minutes. The MgO was coated a dark silver/grey. The coating was smooth with a resistance of 70-120 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-311	1:1.38:1.51	6.37	900	24	7.5-8.5

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-314	1:1.38:1.51	6.3	895	23.6	7.5-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/11/89

Comments: Ran 3 substrates in multiple holder. One plain MgO and Ag coated MgO and Al_2O_3 from Mike. All 3 samples were coated thick and black. The plain MgO looked a little grainy -- possibly out of the hot zone. Resistances were ≈100 to 400 Ω's. (Sent to Karren More.)

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-315	1:1.38:1.51	6.3	843	24	8-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/11/89

Comments: Silver disk ran at 850°C. The 1" area had 2 distinct coatings. The top 3/4 was very smooth and the bottom 1/4 was uniform but grainy. The top resistance was 2-9 Ω's; the bottom was 100-200 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-316	1:1.38:1.51	3.85	890	23.7	8-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-319	1:1.38:1.51	7.04	900	24.6	8-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/13/89

Comments: Another possible ORNL run; MgO and Saphikon. One small spot on the MgO was noted; the samples were smooth, black and uniform. The resistances were 30-60 Ω on MgO; 100 KΩ on Saphikon. The feeder appears to back up at the funnel at low feed rates.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-320	1:1.38:1.51	6.22	900	24.5	8-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/14/89

Comments: MgO and Saphikon for Los Alamos for surface resistance measurements. The MgO had a grainy patch, but otherwise the coatings were smooth and black. The resistances were 20-50 Ω for the MgO, 1-5 MΩ for the Saphikon.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-321	1:1.38:1.51	7.47	895	25	8-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/19/89

Comments: Reruns of ST-242 conditions (higher Cu and less Ba). The MgO had a small triangular dark splotch - otherwise the coating was smooth and even - a resistance of 30-50 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-325	1:1.18:1.68	6.77	905	24.5	8-8.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/19/89

Comments: The samples were coated smooth and even, resistance on the MgO was 50-120 Ω's. Following this run, the O_2 bottle was changed.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-326	1:1.38:1.51	6.77	920	16	7.5-8.0

Flowrates
Ar: 2.5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/21/89

Comments: The run was made with half the Argon flow. Some powder growths were hanging out of the inlet feed tube. The coating was black and a little grainy. Resistance on MgO was 40-50 Ω's. Started heating up under vacuum!

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-301	1:1.38:1.51	6.21	480	1 atm	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/29/89

Comments: Standard anneal at 500°C. The MgO was coated black, smooth and even. The resistance was constant at 1 MΩ. The Sumitomo was coated 4-5" in length.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-302	1:1.38:1.51	6.21	710	1 atm	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 8/20/89

Comments: Standard anneal at 700°C. 15 minute run due to line clog. MgO had little or no coating with no measurable resistance. The Sumitomo was coated orangish-black.

Powder Feed Runs

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-303	1:1.38:1.51	6.02	750 varied	1 atm	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-306	1:1.38:1.51	9.19	920	25	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/5/89

Comments: Run stopped after 10 minutes due to clog in powder lines. Ran Al_2O_3 rods for Mike. The coating looked greenish but was uniform and smooth (211 phase?).

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-307	1:1.38:1.51	4.64	450	22.6	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/5/89

Comments: Annealed at 500°C. Used a 2nd wafer from D-37. All the powder fed in 10 minutes. The coating appeared uniform and black; however, no resistance was measured.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-308	1:1.38:1.51		920	23.5	10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/6/89

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/7/89

Comments: Ran another 1" Ag disk. The sample had a fairly uniform black coating. Resistance was below 10 Ω's. The entire surface was coated.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-312	1:1.38:1.51	6.7	900	23.8	8-8.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/8/89

Comments: Ran the polished Y_2O_3 stabilized PSZ (M-696) for Brent. The coated had a thick, black, uniform appearance over the 1.5" surface. Resistance was 40-50 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-313	1:1.38:1.51	6.32	895	24	8-8.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/8/89

Comments: Possible MgO sample for ORNL. MgO was coated a thick black for the majority of the area., Resistance was 10-30 Ω's. Mike will fracture the area which wasn't coated well.

Anneal: Backfill and cool with pure O2

Date: 9/12/88889

Comments: Ran MgO, Saphikon, and Al_2O_3 rods for Mike. Coatings appeared fairly smooth and black. Resistance on the MgO was 10-20Ω. Resistances could be measured on the Saphikon and rods, which were in the KΩ and MΩ range.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-317	1:1.38:1.51	8.43	918	24.2	9.0

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/12/89

Comments: Ran MgO and Saphikon - possible run for ORNL. Both samples were coated black - the MgO had its typical good smooth coating on the top 3/4 of the plate. Resistances were 30-60 Ω on the MgO; 9 KΩ on the Saphikon.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-318	1:1.38:1.51	8.38	910	24	8-9

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/13/89

Comments: Ran MgO and Saphikon - another possible ORNL run. Coatings were thick and black -- the Saphikon looked very good. The typical coverage pattern on the MgO was observed. Resistances were 30-50 Ω on the MgO; 1-10 KΩ on the Saphikon.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-319	1:1.38:1.51	7.04	900	24.6	8-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/13/89

Comments: Another possible ORNL run; MgO and Saphikon. One small spot on the MgO was noted; the samples were smooth, black and uniform. The resistances were 30-60 Ω on MgO; 100 KΩ on Saphikon. The feeder appears to back up at the funnel at low feed rates.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-320	1:1.38:1.51	6.22	900	24.5	8-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/14/89

Comments: MgO and Saphikon for Los Alamos for surface resistance measurements. The MgO had a grainy patch, but otherwise the coatings were smooth and black. The resistances were 20-50 Ω for the MgO, 1-5 MΩ for the Saphikon.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-321	1:1.38:1.51	7.47	895	25	8-10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/19/89

Comments: Reruns of ST-242 conditions (higher Cu and less Ba). The MgO had a small triangular dark splotch - otherwise the coating was smooth and even - a resistance of 30-50 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-325	1:1.18:1.68	6.77	905	24.5	8-8.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/19/89

Comments: The samples were coated smooth and even, resistance on the MgO was 50-120 Ω's. Following this run, the O_2 bottle was changed.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-326	1:1.38:1.51	6.77	920	16	7.5-8.0

Flowrates
Ar: 2.5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/21/89

Comments: The run was made with half the Argon flow. Some powder growths were hanging out of the inlet feed tube. The coating was black and a little grainy. Resistance on MgO was 40-50 Ω's. Started heating up under vacuum!

Comments: The MgO had two distinct areas of coating - both were smooth. ½ was grey - ½ was black. Resistance was 10-30 Ω's. The Saphikon looked good. Another T variation.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-330	1:1.38:1.51	7.69	900	25	8-8.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/25/89

Comments: Let cool below 100°C to avoid phase transformation of $LaGaO_3$ substrate. The coating looked smooth and black, the resistance was 40-50 Ω's. Sample from Los Alamos.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-331	1:1.38:1.51	5.69	935	6.5	8-8.5

Flowrates
Ar: 0.5 ℓ/min
O_2: 0.5 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/26/89

Comments: Slow backfill of furnace for this lowest pressure CVD run. Both Ar and O_2 were reduced. The MgO was coated grayish/black with a resistance of 20-40 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-332	1:1.38:1.51	0.9	898	23.4	8

Flowrates

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-335	1:1.38:1.51		845	37	8-8.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/29/89

Comments: Tried to duplicate the 850°C run. The pressure was a little high but no obvious leak was found. The MgO had a thick, black coating with resistance not recorded.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-336	1:1.38:1.51	7.8	895	36.1	8.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 9/29/89

Comments: Reran ST-241 conditions. We had planned to wash the MgO in phosphoric acid, but we had a small spill. The MgO and Saphikon were coated well, with resistance of 10-50 Ω's. The pressure was still high.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-337	1:1.38:1.51		885	37.4	8.5-9.0

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/2/89

Anneal: Backfill and cool with pure O2

Date: 10/10/89

Comments: First run with the new powder feeder (NPF). The MgO looked very smooth compared to earlier runs. Resistance was 60 to 2000 Ω's. Had problems with the vacuum and moving the feeder. A large amount of the powder was separated in the intermediate chamber.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	[illegible]	Feed Rate
ST-341	1:1.38:1.51	2.0	890	23	2.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/11/89

Comments: Added a funnel in the intermediate chamber to help catch the powder. The MgO had an extremely smooth black coating. Resistance was 50-200 Ω's. About 4 grams of powder was recovered in the intermediate chamber. Most of the powder is being fed by the feeder. There are some problems at the "webbing."

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-342	1:1.38:1.51	7.22	900	20.7	8.9

Flowrates
Ar: 5 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O2

Date: 10/11/89

Comments: Ran vertical with glass insert and bowl feeder. The coating was grainy, and the sample may have been outside the hot zone. The MgO had a clump of powder in the center. The gas diffuser didn't work very well.

Anneal: Backfill and cool with pure O2

Date: 10/13/89

Comments: Duplicated ST-344. Backfilled with O_2 instead of Argon. MgO looked smooth and uniform. Resistance was 20-100 Ω's. Sumitomo was barely coated (very thin and black).

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-346	1:1.38:1.51	7.49	900	21.5	7.5-8.5

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O2

Date: 10/16/89

Comments: Vertical run - used the Inconel spacer to mix gases. Fed the powder too fast - it was all gone in 10 minutes - ran 10 more before annealing. MgO had a very thin black coating. No measurable resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-347	1:1.38:1.51	7.49	900	21.5	7.5-8.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/16/89

Comments: Repeat of ST-344 but now we backfilled with Argon. We still have the plastic catch in the intermediate chamber. MgO had a smooth coating - resistance was 100-200 0's.

Comments: Tried slowing the feed rate in the vertical furnace. Run lasted 50 minutes before annealing. The sample (MgO) had a poor coating- no resistance was measured.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-351	1:1.38:1.51	1.96	888	20.7	2.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/18/89

Comments: The MgO had a thin glassy black coating. This was a repeat of earlier standard runs with an Argon backfill. Resistance was 75-100 O's.

*We started sweeping system and cleaning sample holder after each run.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-352	1:1.52:1.54	3.37	900	21.0	4.5-7.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/18/89

Comments: ACAC Bowl feeder with ACAC material. We saw a large T jump when powder was introduced. Temperature also jumps during backfill and O_2 anneal (up to 940°C). MgO had a thick black nonuniform coating. Resistance was 1-2 KΩ's.

Anneal: Backfill and cool with pure O2

Date: 10/23/89

Comments: ACAC Rerun of ST-354. The MgO had a smooth, uniform black coating. The results was 500-1000 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-356	1:1:1.25	5.26	895	23.0	5-6.5

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/24/89

Comments: ACAC Again, we adjusted the input acac composition. The feeder is not continuous, but the overall rate is fairly constant. The MgO looked great--very smooth. The resistance was 20-100 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-357	1:1:1.25	5.78	885	23.7	6.5-7.0

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/24/89

Comments: ACAC Duplicated previous run ST-356. The MgO had a smooth black coating--some spots along the edge appeared slightly thicker and grainy. Excellent resistance - 30-70 Ω's.

thick uniform black coating. Resistance was 500-1000 Ω's. There was another "pop" in the O_2 line which went back as far as the Ascerite tube--fortunately, the valve to the feeder was closed.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-361	1:1.38:1.51	1.47	878	24.1	2.75

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/27/89

Comments: tmhd Started using the new powder feeder - finally got $BA(tmhd)_3$ from Kent. The MgO had a thin black uniform coating with R ≈100 Ω's. The Sumitomo had a thin grayish/black coating. This run was similar to earlier standard runs to see if we can reproduce.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-362	1:1.38:1.51	1.64	875	23.7	0.275

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfilled with Argon, annealed in O_2

Date: 10/30/89

Comments: Running @ 875°C; standard conditions, using new reagents (Y, Cu) from Kent. Sample was lost in furnace.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-363	All $Cu(tmhd)_2$	6.94	900	23	6.5

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-366	1:1.38:1.51	2.20	900	23	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2

Date: 11/2/89

Comments: Using Andrew's gas diffuser, there were two distinct zones (60 Ω) and (1 KΩ). The better resistance zone had a smooth black appearance. The higher resistance was silvery and grainy.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-367	1:1.38:1.51	1.67	870	24.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 11/2/89

Comments: First run using no annealing since our ST-349 success. The MgO was smooth, black, and uniform. No resistance was measured. The Sumitomo had a black coating on areas exposed to the flow.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-368	1:1.38:1.51	1.81	874	24.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Flowrates
Ar: 5 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2. no anneal

Date: 11/6/89

Comments: Used Andrew's gas diffuser. Sample looked grainy and poor in quality. No resistance was measured.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-372	1:1.38:1.51	1.66	875	24.1	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with Argon - no anneal

Date: 11/6/89

Comments: Second run. MgO was evenly coated black. Resistance was 50-100 Ω's. Sumitomo was coated lightly black. Use to compare with ST-370.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-373	1:1.38:1.51	1.99	900	22.3	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2. no anneal

Date: 11/7/89

Comments: Duplicated S-371. Used Andrew's gas diffuser. MgO was nonuniformly coated but had a resistance of 100-300 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-377	1:1.38:1.51	1.70	900	23.7	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 no anneal

Date: 11/9/89

Comments: Moved MgO and holder up ½" in the chamber. Resistance was 50-150Ω's. The MgO appeared thick; it was very patchy. Resistance is excellent for a vertical run.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-378	1:1.38:1.51	1.62	864	22.9	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 11/9/89

Comments: PSZ - 50-80 Ω's. Both the PSZ and Saphikon had a good, uniform and black coating. Following this run, the T/C was cleaned with methanol and calibrated in boiling water.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-379	1:1.38:1.51	2.21	900	23.1	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-382	1:1.38:1.51	1.92	900	22.1	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 no anneal

Date: 11/13/89

Comments: Used gas diffuser - hung another Sumitomo tow making sure it was centered. Both the MgO and Sumitomo had thin black coatings--the MgO had 1-2 KΩ resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-383	1:1.38:1.51	1.57	902	23.9	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 11/13/89

Comments: New T for run schedule - 900°C. MgO was coated uniformly. Resistance was 75-100 Ω's. The Sumitomo had black tips near the flow. *Geoving's first superconducting run.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-384	1:1.38:1.51	1.67	900	22.0	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 no anneal

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-387	1:1.38:1.51	1.74	900	22.4	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2. no anneal

Date: 11/15/89

Comments: Used the new sample holder with no diffuser plate. The MgO had R of 20-30 Ω's, but was very grainy looking and shiny. The Sumitomo was coated on the bottom third.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-388	1:1.38:1.51	1.94	904	23.9	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 11/15/89

Comments: Used an expensive MgO substrate with a polished side. Sample broke in half. Resistance was 100-500 Ω's. The coating was black but not very smooth.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-389	1:1.38:1.51	1.77	875	21.3	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2. no anneal

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-392	1:1.38:1.51	1.80	890	24.7	*100%

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - anneal

Date: 11/17/89

Comments: Five minute run from schedule. The MgO had a charcoal appearance with resistance of 80-100 Ω's. The Sumitomo looked good over the exposed area.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-393	1:1.38:1.51	2.09	850	22.3	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 11/17/89

Comments: The MgO had 2 sections; a dull black uniform coating (R=½-1 KΩ) surrounding a shiny black circle (R=10-20 Ω's). The Sumitomo was black on the area below the holder.

Note: Calibrated MFC's before the next run.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate

Comments: Slow feed rate (run time = 55 min.). The MgO had a glassy smooth coating. The Saphikon had a uniform black coating on the exposed area. The MgO resistance was ≈100 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-397	1:1.38:1.51	1.80	908	-	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 11/29/89

Comments: Short run time = 5 minutes. MgO had a black smooth coating, the resistance was ≈50 Ω's. The Saphikon was missing. This run was made with a new batch of $Y(tmhd)_3$.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-398	1:1.38:1.51	1.94	800	22.2	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 no anneal

Date: 11/29/89

Comments: The MgO had a thick, dull black coating at this low T (800°C), directly over the injector. Resistance was 30-50 Ω's. The Sumitomo was coated black below the holder.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-399	1:1.38:1.51	1.95	900	26.6	0.25

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-402	1:1.38:1.51	1.90	904	18.4	0.25

Flowrates
Ar: 2.5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/1/89

Comments: Run with 2.5:1 Ar:O2 ratio. The MgO looked glassy, uniform and black. The Saphikon was also uniformly coated and black. The resistance was ≈90 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-403	1:1.38:1.51	1.98	850	23.4	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/1/89

Comments: An 850°C run with the Mgo sample raised 1". Excellent resistance of 9-16 Ω's. The Mgo was black and shiny but appeared to be slightly grainy.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-404	1:1.38:1.51	1.72	899	26.9	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/1/89

Comments: Considered a standard run in our run schedule. (5:1, Ar:O2) The MgO had a resistance of 300-800 Ω's.

black appearance - a darker coating was observed over part of the sample. Resistance was 30-40 Ω's. The Saphikon was missing. Sent to Dr. Christen.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-408	1:1.38:1.51	1.84	899	27.7	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/5/89

Comments: Another sample for Dr. Christen. Standard conditions. The MgO had a shiny black uniform coating. The Saphikon also had a dark coating. MgO resistance was 50-75 Ω's.

Comments: Repeat of earlier run. The MgO had a glassy uniform black coating. Resistance was 20-50 Ω's. The Sumitomo had a black coating on exposed areas.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-412	1:1.38:1.51	1.74	900	26.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/7/89

Comments: Standard run in schedule. The MgO had a uniform, glassy black coating. The Sumitomo frayed and was tangled i the T/C. The resistance was ≈20 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-413	1:1.38:1.51	1.62	800	22.9	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/7/89

Comments: Duplicated earlier run in vertical furnace. The MgO had distinct rings dark on the outer edge of the coating which we attribute to flow patterns in the furnace around the sample holder. Resistance was 10-20 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate

a big black darker splotch was on the front. It was left overnight in the furnace. Resistance was 50-1200 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-417	1:1.38:1.51	3.66	901	26.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/11/89

Comments: Another run with no intermediate chamber. The start was rough - we flowed Ar before pulling vacuum. The MgO had various regions of coating. Resistances varied from 50 to 200 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-418	1:1.38:1.51	1.74	890	26.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/12/89

Comments: Run #23 of Run Schedule; Acid washed MgO in hot phosphoric acid. This run failed due to the sample being dropped in water by accident after the run. The Saphikon looked good.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-419	1:1.38:1.51	1.70	850	19.7	0.25

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-422	1:1.38:1.51	1.71	900	27.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/13/89

Comments: This is a rerun of #23 and washed MgO. The MgO had a uniform, dull gray coating but did not have a measurable resistance. Note: the sample was left in the furnace overnight.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-423	1:1.38:1.51	1.88	900	24.0	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/13/89

Comments: Higher T run with the gas diffuser. The MgO had a very thin coating which was somewhat patchy. The resistance was 50-150 Ω's. Note: the sample was left in the furnace over night.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-424	1:1.38:1.51	1.89	875	24.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-427	1:1.38:1.51	2.02	870	24.1	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/15/89

Comments: Sample fell out of holder - probably after the run. The MgO had a resistance of 100-200 Ω's. The Sumitomo had a fairly thin black coating. The sample was left in the furnace over the weekend.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-428	1:1.38:1.51	--	850	24.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill with Argon - annealed in O_2 for 30 minutes.

Date: 12/185/89

Comments: Replaced endcap with centered powder/gas tube. First run of annealing study. Had to abort run - the fuse kept popping and the MgO and Saphikon were not coated well.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-429	1:1.38:1.51	2.25	900	21.6	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-432	1:1.38:1.51	1.61	900	20.4	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/20/89

Comments: Aborted run - the fuse on the powder feed controller kept popping, even when we ran it at 0.9. Could be a problem with the initial feed quatity into the feeder.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-433	1:1.38:1.51	1.64	831	24.2	0.25-0.50

Flowrates
Ar: 1 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/20/89

Comments: Part of annealing study. The MgO had 100-200 Ω's resistance. The T/C was moved to profile the furnace and again was directly in hte path of the powder/gas, resulting in lower T's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-434	1:1.38:1.51	1.73	840	24.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/21/89

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/26/89

Comments: The gas diffuser was accidently left in at this low temperature. The MgO had an extremely thin, patchy coating with MΩ resistance. The sample was raised ≈1".

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-438	1:1.38:1.51	2.04	880	24.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/27/89

Comments: Note: Moved T/C out of the gas stream. The MgO had two distinct areas of coating. The black coating was 15-30 Ω's and a grayish black area is alsoo present. The coating appeared smooth.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-439	1:1.38:1.51	1.78	850	22.2	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/27/89

Comments: The sample was raised 1", no gas diffuser. THe MgO had tow concentric circular regions of coating. The grayish center had 10-15 Ω's resistance. The daarker outer area had KΩ's resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-440	1:1.38:1.51	1.94	874	24.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - annealed in O2 for 30 minutes

Date: 12/28/89

Comments: No comments on the run were recorded. Presumably, the sample fell and the run had to be redone.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-441	1:1.38:1.51	1.92	850	22.8	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 12/28/89

Comments: Repeat of S-439. A similar coating was observed, the grayish center had a resistance of 15-30 Ω's. The outer area had 300 Ω's resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-442	1:1.38:1.51	2.04	860	24.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - annealed 30 minutes in O2

S-445	1:1.38:1.51	1.70	850	23	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/2/90

Comments: A repeat of our earlier good runs (S-443, 441, 439). This time the MgO was completely coated black, with a resistance of 80-150 Ω's. We don't know why.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-446	1:1.38:1.51	2.0	890	23.6	0.25

Flowrates
Ar: ℓ/min
O_2: 5 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/3/90

Comments: Part of annealing study. The MgO had a glassy black coating. The outer edges appeared grainy. The resistance was 30-80 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-447	1:1.38:1.51	1.89	850	22	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/3/90

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/4/90

Comments: Another duplication of our earlier runs in the vertical furnace (S-339). The MgO had concentric circles again-- the silver was 16 Ω's; the black outer ring was 120 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-451	1:1.38:1.51	2.06	897	9.2	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/5/90

Comments: Very humid day, had trouble feeding the powder--part of annealing study, ST-401 conditions. The MgO coating appeared extremely variable with the majority being coated grayish black and having 50-70 Ω's resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-452	1:1.38:1.51	1.54	900	19.1	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 500 cc/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/5/90

Comments: Put in gas diffuser, 900°C run. The MgO had a sparse coating with an infinte resistance. The Sumitomo had no coating. The heating element was noticed to be damaged.

O_2: 1 ℓ/min

Anneal: Backfilled with Ar - annealed for 30 minutes in O_2

Date: 1/8/90

Comments: Part of annealing study. The MgO was very non uniform in appearance, having black, gray to a greenish appearance. The Sumitomo had a dark gray coating. The resistance was 30-60 Ω's across the sample but ahd higher resistances in each individual area.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
S-456	1:1.38:1.51	2.1	880	23.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/9/90

Comments: The MgO looked more uniform in this run, the coating had a silver gray region and a darker gray region. The resistance varied from 95-150 Ω's. The Sumitomo had a black coating in some areas.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Lost	Temp (°C)	Pres	Feed Rate
ST-457	1:1.38:1.51	1.69	875	10	0.25

Flowrates
Ar: 1 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/9/90

Comments: Final run of annealing study. The MgO had a thin smooth black coating. The resistance was 50-150 Ω's. The Sumitomo ahd a light uniform grayish-black coating.

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2 - no anneal

Date: 1/11/90

Comments: Standard run with polycrystalline Al_2O_3. The sample had athin coating (black) but the resistance was over 3 MΩ's.

Comments: Ran polycrystalline PSZ substrate (out of MgO). Coating appeared black and uniform with an excellent resistance of 20-30 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-464	1:1.38:1.51	1.45	875	23.9	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 1/16/90

Comments: Polycrystalline PSZ was run to duplicate ST-463. Resistance was 20-50 Ω's and the sample appeared uniformly coated.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-465	1:1.38:1.51	2.30	870	23.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 1/17/90

Comments: Ran ESPI MgO substrate and Saphikon fiber. Resistance was 40-140 Ω's, and the sample was coated black but a little nonuniform. The Saphikon looked good.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-466	1:1.38:1.51	1.76	880	23.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

S-469	1:1.38:1.51	1.85	850	19.7	0.25

Flowrates
Ar: 500 sccm
O_2: 250 sccm

Anneal: Backfill and cool with pure O_2

Date: 1/22/90

Comments: Ran Al_2O_3 in the vertical furnace. No resistance was measured on the sample which had a light black coating.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-470	1:1.38:1.51	1.85	850	19.7	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 1/23/90

Comments: First of 20 "G" runs. CuO furnace heated to 500°C. MgO resistance was 40-60 Ω's with a fairly uniform coating. Areas of gray and black were observed.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
S-471	1:1.38:1.51	--	1000	20	--

Flowrates
Ar: 500 sccm
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: Ran AgCl in an attempt to deposit silver. Mike Shapiro will continue this work for his Ph.D. thesis.

Anneal: Backfill and cool with pure O_2

Date: 1/25/0

Comments: G run - 15 min, 900°C, 15% powder feed rate. The MgO was grayish black but glassy smooth. The resistance was 30-50 Ω's. Sumitomo was missing following the run.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-475	1:1.38:1.51	0.28	895	28.1	0.15

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 1/26/90

Comments: G run - 5 minutes, 900°C, 15% powder feed rate. The MgO was very lightly coated, discolored but glassy. Resistance was 50-75 Ω's. Coloration ranged from light gray to black.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-476	1:1.38:1.51	2.15	845	27.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 1/29/90

Comments: G run - 25 minutes, 850°C, 25% powder feed rate. The MgO was coated fairly uniform and was shiny and black. The resistance was 50 to 70 Ω's. The Sumitomo was coated dark gray.

Date: 1/30/90

Comments: Vertical furnace coating at "best" conditions using a polycrystalline PSZ substrate. The coating appeared mainly a grayish color with a resistance of 30-35 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-480	1:1.38:1.51	1.34	≈900	28.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 1/31/90

Comments: G run, 15 minute, 900°C, 25% powder feed rate. The MgO had a light gray to gray appearance with a dull finish. The resistance was 15-20 Ω's. The Sumitomo was fairly brittle when pulled. (No internal thermocouple.)

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
S-481	1:1.38:1.51	2.25	875	39.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 1/31/90

Comments: Vertical run using polycrystalline PSZ substrate. Coating was black, but the sample holder design prevented a very thick coating. Resistance was excellent - 20-30 Ω's.

Date: 2/2/90

Comments: Ran Saphikon, Sumitomo, and Nicalon fibers at low temperature. This follows Galena's result from one of our earlier fibers. The coatings appeared brownish but uniform - no resistance was measured.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-485	1:1.38:1.51	1.16	≈500	52	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 2/5/90

Comments: Ran new fiber "FP Alumina" from Du Pont at 500°C. Coating was thick, black and uniform but no resistance was measurable. The pressure gauge was not zeroed - may be the cause of the high reading.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-486	1:1.38:1.51	1.95	≈900	45	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 2/6/90

Comments: Again, the FP fiber was run but at 900°C. Unfortunately, the fiber broke and fell in the furnace prior to the run. Pressure gauge is still giving us problems.

Anneal: Backfill and cool with pure O_2

Date: 2/12/90

Comments: Received a new Ba batch. Ran MgO - coating was smooth and black over ≈40%, silver and shiny over ≈60%. Resistance was 10-25Ω's. No internal thermocouple was used, and the pressure gauge still read high (still need to zero it).

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-490	1:1.38:1.51	1.69	900	44	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 2/13/90

Comments: Ran an MgO at a little higher temperature. The coating had two areas, a black area, 4KΩ's, and a silver shiny area, 30-80 Ω's. The Sumitomo had a light gray coating on most of the fibers.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-491	1:1.38:1.51	2.92	≈900	56.3	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 2/14/90

Comments: Switched mass flow controllers to the fiber coating furnace. Running at an extremely reduced flow rate. The MgO looked uniformly coated and had a resistance of 80-170 Ω's. A growth on the feed tube was observed.

Comments: Several start up problems, including forgetting to turn on the Ar bottle, and having unstable T and flow rates were noted. The MgO was very dull looking. One half of the coating was silver, the remaining was grey and black. The resistance was 60-300 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-495	1:0.756:1.49	1.17	910	--	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 2/21/90

Comments: Composition variation - no pressure gauge - only 7 troughs were fed. MgO was lightly coated and was gray with black spots. The resistance was 8-60 KΩ's. This is the first of a 5 run study.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-496	1:1.88:2.90	1.51	850	--	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 2/22/90

Comments: Terrible flow problems were noted The O-ring on the powder feeder was accidently left off; also, the O_2 flow rate was jumping. The MgO looked uniformly silver and shiny. The resistance was 60-80 Ω's. This is the second run of the composition study.

Anneal: Backfill and cool with pure O_2

Date: 2/27/90

Comments: This is another control run to get the process back on track. The MgO was mainly silver and grainy and had an R = 50-70 Ω's. The Sumitomo was coated a grayish color. This is point 4 of the study.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Fe. ' Rate
ST-500	1:0.96:1.43	0.90	895	--	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 3/1/90

Comments: Fifth and final run from the composition study. Only 5 troughs were filled. The MgO had light, thin splotchy coating. The resistance was 350 to 1200 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-501	1:1.38:1.51	1.97	900	--	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 3/2/90

Comments: Standard run - we are trying to handle the powder better - keeping track of which part of the mixed batch we are using. The MgO was mainly dark gray with a resistance between 20 and 35 Ω's.

Date: 3/12/90

Comments: Start of a second composition study of 5 runs. No internal thermocouple, no pressure gauge. The MgO was coated a splotchy grayish silver and had to be redone due to the fact that the powder catch was left on the system.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-505	1:0.76:1.49	2.91	890	--	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 3/13/90

Comments: Again, we tried to start the composition study - this time with no powder catch. At the end of this run, we had a spark, so the O_2 was immediately shut off during cool down. The MgO was coated a dull gray, but no resistance was measured.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-506	1:1.88:2.9	4.46	880	-	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 3/16/90

Comments: Finally starting the 5 run composition study. The internal thermocouple is in place. The MgO was coated very dull silver/gray. The resistance was 5-9 MΩ's. Note: the sample set in the furnace over the weekend.

Date: 3/21/90

Comments: Fourth composition study run. The MgO fell back into the holder during the run and so this had to be done.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-510	1:2.65:3.18	4.12	923	-	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date:

Comments: Running with the new lexan plate for the powder feeder. The T/C was usually high. Fourth run of the composition study. The MgO was extremely variable in color and appearance (red/gray/yellow). The resistance was 8-22 MΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-511	1:0.96:1.43	2.60	925	-	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 3/23/90

Comments: Final run of composition study, again the T/C read much higher than usual. The MgO was 70% dark gray, 30% light gray in appearance. The resistance was 16-22 MΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate

Comments: Repeat of ST-513; unfortunately, the sample fell out again. No resistance was measured. Reinforced importance of loading the MgO so it wouldn't fall out.

Date: 4/3/90

Comments: Another repeat of our standard run. Resistance on the MgO was 90-100 Ω's. The coating appearance was mainly gray and dull. Note: we mixed up 10 g of powder and finished a Y batch.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-518	1:1.381.51	3.40	919	-	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 4/4/90

Comments: First run for Dave and Bruce. Standard conditions. The MgO was coated gray with resistance of 80-90 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-519	1:1.38;1.51	4.01	905	-	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 4/6/90

Comments: Prior to this run, the powder feeder broke, and the lovejoy connector was replaced. Also, we added an argon diffuser before the feeder. The sample was loaded the day before. Its appearance was light silver to dark gray and had a resistance of 25-40 Ω's.

Date: 4/10/90

Comments: Ran a silver coated polycrystalline Al_2O_3 substrate for Mike. Changed the composition of the feed powder. The substrate had a dark gray and silver coloration, the resistance was 1-5 KΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-523	1:1.50:1.75	3.80	905	-	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 4/10/90

Comments: Run changed composition with an MgO substrate. The sample had some light green area but was mainly black and gray. The resistance was 140-160 KΩ's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-524	1:1.50:1.75	-	-	-	0.25

Flowrates
Ar: 500 cc/min
O_2: 100 cc/min

Anneal: Backfill and cool with pure O_2

Date: 4/11/90

Comments: The powder feeder had problem, so the substrates were coated very lightly - only one trough was fed. Resistance measured on the MgO was 30-50 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-526B	1:1.38:1.51	4.02	870	31.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 4/16/90

Comments: Ran a silver coated Al_2O_3 substrate for Mike. The silver coating appeared to be gone after the processing. The front surface appeared to be unevenly coated and had a resistance of 300-400 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-527	1:1.38:1.51	3.75	865	33.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Date: 4/16/90

Comments: Standard run - we still haven't gotten a verification on the superconductor quality. The MgO had two areas of coating. The light gray area (80%) had a resistance of 200-300 Ω's; the dark gray area had 20-30 Ω's.

Run	(Y:Ba:Cu) Reagent wt Ratio	(grams) Powder Fed	Temp (°C)	Pres	Feed Rate
ST-528	1:1.38:1.51	4.67	880	28.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O_2

Powder Feed Runs

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-532	1:1.38:1.51	-	900	27.7	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O_2, furnace cooled with flowing O_2

Date: 4/23/90

Comments: Standard run - resistance 200-400 Ω. MgO coated uniformly but the resistance varied widely.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-533	1:1.38:1.51	-	900	27.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O_2, furnace cooled with flowing O_2

Date: 4/24/90

Comments: Standard run - resistance 90-110 Ω. Sumitomo coated well near end facing flow. MgO rainy coating spotted with light grey. Samples left in O_2 overnight.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-534	1:1.38:1.51	-	873	29.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O_2, furnace cooled with flowing O_2

Date: 4/19/90

Comments: Standard run - resistance 120-130 Ω. Sumitomo coated dark grey but not uniform. MgO coated black and dark grey.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-538	1:1.38:1.51	-	-	31.9	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool furnace with flowing O2

Date: 5/2/90

Comments: Standard run - resistance 125-130 Ω. Used ACAC reagents because Ba wasn't ready. MgO grainy but evenly coated. Large jump in pressure; no Ar rotameter.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-539	1:1.38:1.51	-	915	28.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool furnace with flowing O2

Date: 5/3/90

Comments: Standard run - resistance 3-4 mΩ. Leftover powder. Annealed for 30 min at 600°. No change in MgO color or resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-540	1:1.38:1.51	-	-	27.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, anneal for 30 min at 600°

Date: 5/3/90

Comments: Standard run - resistance 1500Ω. t(23)≈955°-no t/c No change in MgO color or resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-544	1:1.38:1.51	-	880	26.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 on CuO

Date: 5/9/90

Comments: Standard run - resistance 30-150 Ω. MgO resistance varied with color: black (30-40 Ω) dark gray (120-150 Ω)

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-545	1:1.38:1.51	-	930	30	0.10

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool down with O2 from CuO furnace

Date: 5/10/90

Comments: Run stopped after 7 min when pressure jump noted. Sample were coated. 1st Al2O3 coated black with mΩ resistance. Others coated but no resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-546	1:1.38:1.51	-	876	26.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cooldown with CuO furnace

Date: 5/10/90

Comments: Standard run - resistance 15-30 Ω. Powder blown by Ar, so fed unevenly. MgO dark gray to light gray.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-550	1:1.38:1.51	-	880	25.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 from CuO furnace

Date: 5/16/90

Comments: Standard run - resistance 50-70 Ω. Saphikon coated even and black in front of sample holder. Lightly coated black/green in back. MgO varied from black to silver. Ran without O2 first five minutes.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-551	1:1.38:1.51	-	870	25.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 5/16/90

Comments: Standard run, resistance 40-80 Ω. Powder clumpy from humidity. Saphikon evenly coated black in front of sample holder only. MgO coated black to grainy silver.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-552	1:1.38:1.51	-	880	25.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 5/17/90

Comments: Standard run, resistance 30-60Ω. PSZ had good, even coating, but seemed to scratch. Saphikon coated evenly and dark.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-556	1:1.38:1.51	-	895	25.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 5/21/90

Comments: Standard run - resistance 80-10 Ω. PSZ coating very thin, semitransparent because not enough powder packed coating was brown gray.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-557	1:1.38:1.51	-	-	25.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 from CuO furnace

Date: 5/22/90

Comments: Standard run - resistance 20-50 kΩ. Powder blew to middle of feeder (½ gram). Ar line clogged during last five minutes. PSZ coating even and clean. No t/c TZ3≈930

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-558	1:1.38:1.51	-		26.1	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 5/23/90

Comments: Standard run - 60-120 Ω resistance. MgO very smooth and even but thin. No t/c TZ3≈933

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-562	1:1.38:1.51	-	875	26.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 6/1/90

Comments: Standard run, resistance 1-6 kΩ. Saphikon coated well. MgO coated well, but a little thin on one side (had higher resistance, too).

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-563	1:1.38:1.51	-	-	26.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 6/1/90

Comments: Standard run, resistance 60-70 Ω. Saphikon broke. MgO uniformly gray. No t/c T(Z3)≈931°C

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-564	1:1.38:1.51	-		25.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 6/4/90

Comments: Standard run - 100-200 Ω. Saphikon broke twice. Huge leak in system. PSZ looked very weird. No t/c t(z3)≈927°C

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-568	1:1.38:1.51	-	840	25.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 6/8/90

Comments: Standard run - resistance 10-160 Ω. Rotated cap 90°.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-569	1:1.38:1.51	-	850	26.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 6/11/90

Comments: Standard run. Silver coated well only where powder blowing on it.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-570	1:1.38:1.51	-	840	26.1	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 6/12/90

Comments: Standard run. Cap rotated 1/3 turn. Coated a little better.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-574	1:1.38:1.51	-	850	25.6	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 6/14/90

Comments: Refilled powder feeder twice without breaking vacuum. Run time ~15 min. Sample holder slid back. Slight coating. No resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-575	1:1.38:1.51	-	850	24.9	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 6/15/90

Comments: Refilled powder feeder twice without breaking vacuum. Run time ~15 min. Used steel rod to hold rube. No resistance.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-576	1:1.38:1.51	-	-	25.7	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with O2

Date: 6/27/90

omments: Standard run. CuO furnace broken. Pressure high first reading because a valve wasn't open. Powder not feeding continuously - small gaps coating blotchy in some areas, good in others. Resistance 5-20 Ω.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-580	1:1.38:1.51	-	875	25.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with O2, no CuO furnace

Date: 7/6/90

Comments: Standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-581	1:1.38:1.51	-	880	25.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with O2, cool with O2 through CuO furnace

Date: 7/10/90

Comments: Standard run. Silver tube done.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-582	1:1.38:1.51	-	850	26.1	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with O2 through CuO furnace

Date: 7/11/90

Comments: Standard run - resistance 30-200 Ω. MgO looked smooth, uniform, grayish black. Sumitomo looked good. Beginning of composition study #1.

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-586	1:0.71:0.96	-	845	25.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Cool and backfill with O2 through CuO furnace

Date: 7/16/90

Comments: Standard run - resistance 0.6-1.2 mΩ. Sumitomo had variable coating MgO had thick, dark and smooth coating point #10 on comp study #1

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-587	1:1.75:2.25	-	860	25.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with O2 through CuO furnace

Date: 7/16/90

Comments: Standard run - resistance 50-150 Ω. Sumitomo frayed, coated dark in middle, light on both ends MgO coated well, gray and black, smooth point #1 on comp study #1 new yttrium

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-588	1:1.38:1.51	-	860	25.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with O2 from CuO furnace

Date: 7/17/90

Comments: Standard run - resistance 30-200 Ω. Sumitomo not coated evenly - heavy at tip, greenish black at middle MgO smooth with silver and gray area point control on comp study #1

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-592	1:1.05:2.02	-	865	25.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with O2 through CuO furnace

Date: 7/19/90

Comments: Standard run - resistance 200-600 Ω Sumitomo MIA MgO shiny and smooth on ends but rough in middle point #8 in comp study #1

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-593	1:0.79:1.35	-	870	25.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with Ow through CuO furnace

Date: 7/20/90

Comments: Standard run - resistance 150-400 Ω Sumitomo MIA MgO smooth, dull, gray point #9 in comp study #1

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-594	1:1.38:1.51	-	865	26.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with O2 through CuO furnace

Date: 7/23/90

Comments: standard run - resistance 50-70 Ω Sumitomo MIA MgO smooth, shiny and uniform first of annealing experiment

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-598	1:1.38:1.51	-	850	24.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool with O2 through CuO furnace

Date: 7/27/90

Comments: run only 20 min

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST-599	1:1.38:1.51	-	870	20.9	0.25

Flowrates
Ar: 3.5-4 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill with Ar, cool with O2 through CuO furnace

Date: 7/30/90

Comments: problem with Ar line, only got 4 ℓ/min at best end of sumitomo coated very dark and thick resistance 0.4-50 kΩ MgO had non-uniform and dull coating

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST603	1:3.55:3.03	-	810	24.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnance.

Date: 8/17/90

Comments: Run only 23 min
Resistance 80-200Ω
Sumitomo coated thick at tip - varied on rest of fiber
MgO dull, blotchy
Point #2 on composition study #2

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST604	1:2.10:2.02	-	810	24.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with CuO furnance

Date: 8/20/90

Comments: Standard run-resistance 30-40Ω
Sumitomo coated non-uniform
MgO glassy, blotchy
Point #1 on composition study #2

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST605	1:1.38:1.51	-	820	24.7	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST608	1:4.34:2.36	-	810	23.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnance

Date: 8/24/90

Comments: Standard run-resistance 100-500Ω
Sumitomo had variable coating, brown blotches
MgO not smooth, scrathy
Point #7 on composition study #2

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST609	1:4.34:2.36	-	810	24.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfi[illegible] and cool with O2 thru CuO furnance

Date: 8/27/90

Comments: Standard run-resistance 50-200Ω
MgO blotchy, non-uniform
Sumitomo coated well on tip, less in middle
Ar flow jumped in last five minutes
New copper
Point #7 on composition study #2

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST610	1:1.38:1.51	-	400	23.5	0.25

Flowrates
Ar: 5 ℓ/min

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool wiht O2 thru CuO furnance

Date: 8/30/90

Comments: Run 30 min Al2O3 piece suspended in front of sample holder to test CVW
Resistance 2-200Ω
Well coated in general vary degrees of coating on substrate and in holes
New barium and yittrium

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST614	1:2.37:2.24	-			0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal:

Date: 8/31/90

Comments: Abort due to problem in powder feeder

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST615	1:2.37:2.24	-		24.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnance

Date: 9/14/90

Comments: Standard run-resistance 18-70Ω T(23)~920
Sumitomo tip coated well, looks infiltrated
MgO looked uneven, had big crack diagonally
Point control on composition study #3

Date: 9/6/90

Comments: Run 34min
Powder feeder kept quitting, very jerky
Sumitomo thick and black but not infiltrated
MgO blotchy - resistance 60-140Ω
T(23) ~ 920
Point #3 on composition study #3

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST619	1:2.73:2.18	-		24.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnacne

Date: 9/10/90

Comments: Standard run - resistance 90-125Ω
Sumitomo frayed and unevenly coated
MgO smooth, variable reistance
T(23) ~920
Point 4 on composition study #3

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST620	1:2.372.24	-		26.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnace

Date: 9/10/90

Comments: Standard run - resistance 300-400Ω
Large pressure head
T(23) ~ 925
MgO smooth, grey
Sumitomo coated thick and black on outside fibers
Point control on composition study #3

Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnace

Date: 9/12/90

Comments: Standard run - resistance 500-600Ω
MgO blotchy
Reaction tube too far out, assumed its been like that
Composition study #3

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST625	1:2.37:2.24	-	880	26.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnace

Date: 9/13/90

Comments: Standard run - resistance 800-1200Ω
Tube back in place
Pressure high at end
MgO coated unevenly and granular

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST626	1:2.37:2.24	-	450	24.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnace

Date: 9/13/90

Comments: T(23) ~ 500
MgO coating light brown
No measurable resistance

Comments: 30 min run
Methanol contaminated powder
MgO had coating on edges
Large crack on surface
Point #3 on composition study #4
Resistance 200-500Ω

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST630	1:3.03:1.92	-	870	25.8	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnace

Date: 9/19/90

Comments: Standard run - resistance 10-130Ω
MgO grey and black
Large leak rate
Point #3 on composition study #4

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST631	1:2.37:2.24	-	870	25.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with O2 thru CuO furnace

Date: 9/21/90

Comments: Standard run - resistance 50-100Ω
MgO dull and shiny differnt places
Control point on composition study #4

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST635	1:2.37:2.24	-	880	26.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 9/25/90

Comments: Substrate hollow tube x74-19

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST636	1:2.37:2.24	-	845	26.1	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 9/26/90

Comments: Substrate zirconia tape no coating

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST637	1:2.37:2.24	-	860	26.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 10/3/90

Comments: Leak in plexiglass run aborted

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST641	1:2.37:2.24	-	860	23.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 10/3/90

Comments: MgO freckled, 2-1-1 looked present
Standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST642	1:2.37:2.24	-	440	24.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 10/4/90

Comments: MgO brownish black, uneven
Low temp run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST643	1:2.37:2.24	-	700	25.4	0.25

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST646	1:2.37:2.24	-	860	25.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 10/8/90

Comments: MgO fell into holder during run
Uniform grey
One side resistance 60-80Ω
Other side 15-25Ω

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST647	1:2.37:2.24	-	920	25.7	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool in pure O2

Date: 10/9/90

Comments: Greenish MgO
100Ω resistance
Standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST648	1:2.37:2.24	-		26.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool in pure O2

Date: 10/10/90

Date: 10/12/90

Comments: PRD lost in furnace
MgO smooth, silver green
resistance <= 0.5Ω
Silver/123 run in vibratory feeder (16g)
Ar backed down from 5l/min to 2l/min

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST652		-		35.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool in pure O2

Date: 10/12/90

Comments: Moring silver coated alumina into and out of hot zone

Run	(Bi:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST653	1.76:1.48:6.86:1	-		25.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: backfill and cool in pure O2

Date: 10/15/90

Comments: 15 min run, no T/C
sample clumpy
MgO resistance 2000-3000Ω

Run	(Bi:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST654	2:3:1.38:2	-	870	27.1	0.25

Run	(Bi:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST657	3:3:1.4:3	-	915	27.6	0.50

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 10/22/90

Comments: MgO speckled
Resistance 110-3mΩ

Run	(Bi:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST658	3.5:3:1:3.25	-	925	28.3	0.50

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 10/22/90

Comments: MgO speckled - resistance 30-100Ω

Run	(Bi:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST659	3.5:3.0:1.4:3.25	-	870	28.2	0.50

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 10/23/90

Comments: Refilled powder tray once
MgO resistance 3-5kΩ

Alumina substrate
Resistance 150-600Ω

Run	(Bi:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST663	1:1.48:.686:1	-		29.3	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date:

Comments: 5 samples - MgP, MgO, PSZ, Al2O3, MgO
1st MgO had most coating

Run	(Bi:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST644		-			1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 10/29/90

Comments: Physically forced powder in
PSZ well coated
MgO blotchy

Run	(Bi:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST665	1.5:1:0.5:1	-		29.2	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Run	(Bl:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST668	2:1:0.5:1.5	-		27.5	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 11/6/90

Comments: PSZ resistance 5-50kΩ, brown, speckled
MgO immeasurable resistance splotchy
No T/C

Run	(Bl:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST669	2:1:0.5:1	-		30	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 11/7/90

Comments: PSZ brownish - red
Splotchy crystals
Resistance 500-1000Ω
Refilled tray twice
no T/C

Run	(Bl:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST670	2:1:0.5:1	-		30	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 11/14/90

Comments: Not very well coated - Silver taped sumitomo

Run	(Bl:Sr:Ca:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST673	3.5:3.0:1.0:3.25	-	888	27.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 11/15/90

Comments: Alumina and Sumitomo

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST674	1:2.37:2.24	-	860	30.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal:

Date: 11/19/90

Comments: Coating uniform
Green on corners
MgO resistance 4mΩ

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
St679	1:2.37:2.24	-	890	28.4	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 11/27/90

Comments: Alumina coated with silver
Resistance 400Ω
Sumitomo Black and dark grey

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST680	1:2.37:2.24	-	880	27.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 11/28/90

Comments: MgO dark spot 600-100Ω Rest 1-2kΩ
Standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST681	1:2.37:2.24	-	860	29.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 11/29/90

Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 12/3/90

Comments: Al2O3 (Silver coated/sputtered)
Resistance 35-200Ω
Vaarying colors of grey

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST685	1:2.37:2.24	-		30.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date: 12/5/90

Comments: Standard run - Resistance .15-1.5mΩ
PSZ varying coating

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST686	1:4.79:4.22	-			0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool in pure O2

Date:

Comments: Run aborted

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST689	0.3:1.44:1.26	-	870	54.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Cool and backfill with pure O2

Date: 12/7/90

Comments: Al2O3 light coating - Reistance 330-700Ω - Standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST690	.281:.984:1.55	-	860	30.5	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Cool and backfill with pure O2

Date: 12/11/90

Comments: Light smooth coating - Resistance 200-300Ω - standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST690(II)	.281:.984:1.55	-	897	30.2	0.25

Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 12/13/90

Comments: MgO good coating (65-600)Ω

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST693		-	900	28.7	1.00

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 12/14/90

Comments: MgO dark uniform coating - 200-700Ω resistance

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST694	1:1.38:1.51	-		29.4	0.15

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 12/17/90

Comments: Powder feeder kept shutting off - run took longer than normal
LaAlO3 black with blue stripe - resistance 150-200kΩ

Anneal: Backfill and cool with pure O2

Date: 12/18/90

Comments: MgO granular and grey - resistance 0.8mΩ

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST698	1:2.37:2.24	-	875	29.4	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 12/19/90

Comments: MgO and FP - resistance 123kΩ-3.4mΩ - Standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST699	1:1.38:1.51	-	850	29.9	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 12/19/90

Comments: Standard run - MgO dark with 30-50Ωresistance

Date: 12/21/90

Comments: LaAlO3 resistance 300-600Ω - Standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST703	1:1.38:1.51	-	860	30.0	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 1/3/91

Comments: MgO blotchy, 5-50kΩ resistance
PRD coated black blotchy - Standard run

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST704	1:1.38:1.5	-	865	30.2	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 1/7/91

Comments: Sample holder pushed to back of furnace when vacuum pulled to fast

Anneal: Backfill and cool with pure O2

Date: 1/10/91

Comments: Standard run - FD badly coated frayed, coated in clumps
MgO uniform, silver and shiny, resistance 150-250Ω

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST708	1:1.38:1.51	-	420	31.7	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 1/11/91

Comments: 23~525degrees - MgO coating thin, no resistance(infinity)
PRW-166 and 7p fibers included

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST709	1:1.38:1.51	-	465	29.6	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 1/11/91

Comments: (23)~550 degrees - FP and PRD-166 fibers look good

Date: 1/18/91

Comments: Standard run - LaAlO3 coating blotchy and granular
Resistance 400-800Ω

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST713	1:1.38:1.51	-	855	31.3	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 1/18/91

Comments: Standard run - Al2O3 granular - resistance 400-2000Ω - multi-colored

Run	(Y:Ba:Cu) Reagent wt Ratio	Powder Fed (grams)	Temp (°C)	Pres	Feed Rate
ST714	1:1.38:1.51	-	860	31.7	0.25

Flowrates
Ar: 5 ℓ/min
O_2: 1 ℓ/min

Anneal: Backfill and cool with pure O2

Date: 1/21/91

Comments: Standard run - FP badly frayed - coated balckand grey
Resistance 3-5kΩ

REFERENCES

1. Tachikawa, K. and Togeno, K., "Potential Methods for the Fabrication of High-T_c Superconductors for Wires and Cables," Proceedings of the IEEE, 17(8), 1124 (1989).

2. Leu, H. J., Singh, J. P., Dorris, S. E., and Poeppel, R. B., "Fabrication of a superconducting coil for solenoid applications," Supercond. Sci. Technol., 2, 311 (1989).

3. Alford, N. McN., Button, T. W., and Birchall, J. D., "Processing, properties and devices in high-T_c superconductors," Supercond. Sci. Technol., 1 (1989).

4. Yamashita, T., Era, M., Noge, S., Irie, A., Yamane, H., Hirai, T., Kurosawa, H., and Matsui, T., "Josephson Current in Microbridges of $YBa_2Cu_3O_{7-d}$ Thin Films Prepared by CVD," Jap. J. of Appl. Phys., 29(1), 74 (1990).

5. Personal communications, HOLOX Corp., (1990).

6. Chu, C. W., Hor, D. H., Meng, R. L., Gao, L. Huang, Z. J., Wang, Y. Z., Phys. Rev. Lett., 58, 405, (1987).

7. Phillips, J. C., **Physics of High-T_c Superconductors**, Academic Press, Inc., San Diego, CA (1989).

8. Wong-Ng, W., Roth, R. S., Swartzendruber, L. J., Bennett, L. H., Chiang, C. K., Beech, F., and Hubbard, C. R., "X-ray Powder Characterization of $Ba_2YCu_3O_{7-x}$," Advanced Ceramic Materials, 2(3), 565 (1987).

9. Clarke, D. R., Shaw, T. M., and Dimos, D., "Issues in the Processing of Cuprate Ceramic Superconductors," J. Am. Ceram. Soc., 72(7), 1103 (1989).

10. Ullman, J. E., McCallum, R. W., and Verhoeven, J. D., "Effect of atmosphere and rare earth on liquidus relations in RE-Ba-Cu-Oxides," J. Mater. Res., 4(4), 752 (1989).

11. Wong-Ng, W., Cook, L. P., Chiang, L. J., Swartzendruber, L. J., Bennett, L. H., Blendell, J., and Minor, D., "Structural phase transition study of $Ba_2YCu_3O_{6+x}$ in air," J. Mater. Res., 3(5), 832 (1988).

12. Hirai, T., Yamane, H., Kurosawa, H., Watanabe, K., Kobayashi, N., Iwasaki, H., and Muto, Y., "Preparation of High-J_c Y-Ba-Cu-O Films By CVD," International Superconductivity Electronics Conference (ISEC '89), June 12-13, Tokyo, Japan (1989).

13. Dimos, D., Chaudhari, P., Mannhart, J., and LeGoues, F. K., "Orientation Dependence of Grain-Boundary Critical Currents in $YBa_2Cu_3^{7-d}$ Bicrystals," Physical Review Letters, 61(2), 219 (1988).

14. McAlford, N. McN.,Birchall, J. D., Clegg, W. J., Harmer, M. A., Kendall, K., and Jones, D. H., "Physical and mechanical properties of $YBa_2Cu_3O^{7-d}$ superconductors," J. of Mat. Sci., 23(3), 161 (1988).

15. Lackey, W. J., Stinton, D. P., Cerny, G. A., Fehrenbacher, L. L., and Schaffhauser, A. C., "Ceramic Coatings for Heat Engine Materials - Status and Future Needs, ORNL/TM-8959, Oak Ridge National Laboratory, Oak Ridge, TN.

16. Lynch, C. T., CRC Handbook of Materials Science, CRC Press, Inc., Boca Raton, FL (1975).

17. Hashimoto, T. Fueki, K., Kishi, A., Azumi, T., and Koinuma, H., "Thermal Expansion Coefficients of High-T_c Superconductors," Jap. J. of Applied Physics, 27(2), 1214 (1988).

18. A. Berry, D. Gaskill, R. Holm, E. Cukauskas, R. Kaplan, and R. Henry, "Formation of High T_c Superconducting Films by Organometallic Chemical Vapor Deposition," Appl. Phys. Lett., 52, 1743-1745 (1988).

19. H. Yamane, H. Kurosawa, and T. Hirai, "Preparation of $YBa_2Cu_3O_{7-x}$ Films by Chemical Vapor Deposition," Chemistry Lett., Issue No. 6, 939-940 (1988).

20. C. Gonzalez, O. Schachner, H. Tippmann, and F. Trojer, "Superconducting Thin Films of Y-Ba-Cu-Oxide by Sol Gel and CVD Methods," Physica C, 153-155, 1042-1043 (1988).

21. T. Nakamori, H. Abe, T. Kanamori, and S. Shibata, "Superconducting Y-Ba-Cu-O Oxide Films by OMCVD," Jap. J. of Appl. Phys., 27, L1265-1267 (1988).

22. A. Panson, R. Charles, D. Schmidt, J. Szedon, G. Machiko, and A. Braginski, "Chemical Vapor Deposition of $YBa_2Cu_3O_7$ Using Metal-Organic Chelate Precursors," Appl. Phys. Lett., 53, 1756-1758 (1988).

23. K. Shinohara, F. Munakata, and M. Yamanaka, "Preparation of Y-Ba-Cu-O Superconducting Thin Film by Chemical Vapor Deposition," Jap. J. Appl. Phys., 27, L1683-1685 (1988).

24. J. Zhao, K. Dahmen, H. Marcy, L. Tonge, T. Marks, B. Wessels, and C. Kannewurf, "Organometallic Chemical Vapor Deposition of High T_c Superconducting Films Using a Volatile Fluorocarbon-Based Precursor," Appl. Phys. Lett., 53, 1750-1752 (1988).

25. H. Yamane, H. Masumoto, T. Hirai, H. Iwasaki, K. Watanabe, N. Kobayashi, and Y. Muto, "Y-Ba-Cu-O Superconducting Films Prepared on $SrTiO_3$ Substrates by Chemical Vapor Deposition," Appl. Phys. Lett., 53, 1548-1550 (1988).

26. K. Zhang, B. Kwak, E. Boyd, A. Wright, and A. Erbil, "C-axis Oriented $YBa_2Cu_3O_{7-x}$ Superconducting films by Metalorganic Chemical Vapor Deposition," Appl. Phys. Lett., 54, 380-382 (1989).

27. K. Watanabe, H. Yamane, H. Kurosawa, T. Hirai, N. Kobayashi, H. Iwasaki, K. Noto, and Y. Muto, "Critical Currents at 77.3K Under Magnetic Fields up to 27 T for an Y-Ba-Cu-O Film Prepared by Chemical Vapor Deposition," Appl. Phys. Lett., 54, 575-577 (1989).

28. S. Oda, H. Zama, T. Ohtsuka, K. Sugiyama, and T. Hattori, "Epitaxial Growth of YBaCuO Films on Sapphire at 500°C by Metalorganic Chemical Vapor Deposition," Jap. J. of Appl. Phys., 28, L427-429 (1989).

29. T. Tsuruoka, H. Takahashi, R. Kawasaki, and T. Kanamori, "Characteristics of Quenched Y-Ba-Cu-O Thin Films on $SrTiO_3$ (100),(110) Grown by Organometallic Chemical Vapor Deposition," Appl. Phys. Lett., 54, 1808-1809 (1989).

30. M. Ottosson, T. Andersson, J. Carlsson, A. Harsta, U. Jansson, P. Norling, K. Niskanen, and P. Nordblad, "Chemical Vapor Deposition of the Superconducting $YBa_2Cu_3O_{7-x}$ Phase Using Halides as Metal Sources," Appl. Phys. Lett., 54, 2476-2478 (1989).

31. H. Yamane, H. Kurosawa, and T. Hirai, "Preparation of Superconducting - Oxide Films by CVD and Their Properties," Proc. 7th European Conf. on CVD, Colloque De Physique, C-5, 131-140 (1989).

32. F. Schmaderer and G. Wahl, "CVD of Superconductive $YBa_2Cu_3O_{7-x}$," Proc. 7th European Conf. on CVD, Colloque De Physique, C-5, 119-129 (1989).

33. P. Dickenson, T. Geballe, A. Sanjurjo, D. Hildenbrand, G. Craig, M. Zisk, J. Collman, S. Banning, and R. Sievers, "Chemical Vapor Deposition of $YBa_2Cu_3O_{7-x}$ Superconducting Films," J. Appl. Phys., 66, 444-447 (1989).

34. Yamane, H., Kurosawa, H., Hirai, T., Watanabe, K., Iwasaki, H., Kobayashi, N., and Muto, Y., "High Critical Current Density of Y-Ba-Cu-O Superconducting Films Prepared by CVD," submitted to Superconductor Science and Technology.

35. F. Radpour, R. Singh, S. Sinha, A. Tulpule, P. Chou, R. Thakur, M. Rahmati, N. Hsu, and A. Kumar, "Preparation of Y-Ba-Cu-O Superconducting Thin Films Using BaF_2 as a Buffer Layer," Appl. Phys. Lett., 54(24), 2479-2480 (1989).

36. T. Hirai, H. Yamane, H. Kurosawa, K. Watanabe, N. Kobayashi, H. Iwasaki, and Y. Muto, 1989 International Superconductivity Electronics Conference (ISEC '89), June 12-13, 1989, Tokyo, Japan

37. K. Kanehori, N. Sugii, and K. Miyauchi, Journal of Solid State Chemistry, **82**, 103-108 (1989)

38. Singh, R., Sinha, S., Hsu, N. J., and Chou, P., "In situ deposition of BaF_2 as a buffer layer and the superconducting thin films of Y-Ba-Cu-O on silicon substrates by metalorganic chemical vapor deposition," J. Appl. Phys., 67(8), 3764 (1990).

39. Singh, R., Sinha, S., Hsu, N. J., and Chou, P., "Superconducting thin films of Y-Ba-Cu-O prepared by metalorganic chemical vapor deposition," J. Appl. Phys., 67(3), 1562 (1990).

40. Yamaguchi, T., Aski, S., Sadakata, N., Kohno, O., and Osanai, H., "Superconduting properties of $Ba_2YCu_3O_{7-x}$ thin films prepared by chemical vapor deposition on $SrTiO_3$ and a metal substrate," Appl. Phys. Lett., 55(15), 1581 (1989).

41. Matsuno, S., Uchikawa, F., and Yoshizaki, K., "Y-Ba-Cu-O Superconducting Films with High J_c Values by MOCVD using Ba-Addition Products," Jap. J. of Appl. Phys., 29(6), 1947 (1990).

42. Kanchori, K., Sugii, N., and Miyauchi, K., "Low Temperature Growth of Superconducting $YBa_2Cu_3O7-_X$ Thin Films By Metalorganic Chemical Vapor Deposition," MRS Proceedings, Boston, MA, Nov. 1990.

43. Radpour, F. Singh, R. Sinha, S., Chou, P., Hsu, N. J., and Rahmati, M., "Metal Organic Chemical Vapor Deposition of High-Temperature Superconducting Y-Ba-Cu-O Films," J. Electrochem. Soc., 137(8), 2462 (1990).

44. Tsuruoka, T. Kawasaki, R., Abe, H., and Shibata, S., "Low Temperature Preparation of Y-Ba-Cu-O Films By OMCVD,"

45. Rao, Y. K. and Kim, S. S., "Chemical Vapor Deposition of the Superconductor $YBa_2Cu_3O_{7-X}$ (s) from the Gas Phase," J. of Superconductivity, 2(3), 395 (1989).

46. Tsuroka, T., Kawasaki, R., and Abe, H., "Y-Ba-Cu-O Film Growth by OMCVD Using N_2O," Jap. J. of Appl. Phys., 28(10), 11800 (1989).

47. Abe, H., Tsuruoka, T., and Nakamori, T., "YBa_2Cu_3O7-x Film Formation by an OM-CVD Method," Jap. J. of Appl. Phys., 27(8), 11473 (1988).

48. Muto, Y., Watanabe, K., Kobayashi, N., Kawabe, H., Yamane, H., Kurosawa, H., and Hirai, T., "Transport Critical Current and Morphology For Y-Ba-Cu-O Films Prepared by Chemical Vapor Deposition," Physica C: Superconductivity, 162-164(1), 105 (1989).

49. Watanabe, K., Kobayashi, N., Yamane, H., Kurosawa, H., Hirai, T., Kawabe, H., and Muto, Y., "Critical Current Criterion in High T_c Superconducting Films," Jap. J. of Appl. Phys., 28(8), 1417 (1989).

50. Kurosawa, H., Yamane, H., Matsumoto, H., and Hirai, T., "Preparation of the c-axis Oriented Films of $YBa_2Cu_3O_{7-X}$ by Chemical Vapor Deposition.

51. Watanabe, K., Matsushita, T., Kobayashi, N., Kawabe, H., Aoyagi, F., Hiraga, K., Yamane, H., Kurosawa, H., Hirai, T., and Muto, Y., "Strong flux pinning centers in Y-Ba-Cu-O films prepared by chemical vapor deposition," Appl. Phys. Lett., 56,(15), 1490 (1990).

52. Zhang, K., Kwak, B. S., Boyd, E. P., Wright, A. C., and Erbil, A., "Metalorganic Chemical Vapor Deposition of Highly Textured Superconducting $YBa_2Cu_3O_{7-X}$ Films," Proceedings of the Conference of Science and Technology of Thin-Film Superconductors, Colorado, Nov. 1988.

53. Erbil, A., Zhang, K., Kwak, B. S., and Boyd, E. P., "A Review of Metalorganic Chemical Vapor Deposition of High-Temperature Superconducting Thin Films," SPIE Vol. 1187 Processing of Films for High T_c Superconducting Electronics, 104 (1989).

54. Yamane, H., Kurosawa, H., Hirai, T., Iwasaki, H., Kobayashi, N., and Muto, Y., "Temperature Dependence of Electrical Resistivity for Y-Ba-Cu-O Films Prepared by CVD," NSKGR, 96(7), 799 (1988).

55. Ohnishi, H., Kusakabe, Y., Kobayashi, M., Hoshinouchi, S., Harima, H., and Tachibana, K., Preparation and Characterization of Superconducting Y-Ba-Cu-O Films by the MOCVD Technique," Jap. J. of Appl. Phys., 29(6), 1070 (1990).

56. Yamane, H., Kurosawa, H., Hirai, T., Watanabe, K., Kobayashi, N., and Muto, Y., "Y-Ba-Cu-O Superconducting Films Prepared on $SrTiO_3$ (100) By CVD and Their High J_c In Magnetic Fields," Proceedings of the 11th International CVD Conference, Seattle, WA 1990.

57. Schmaderer, F., Huber, R., Oetzmann, H., and Wahl, G., "CVD of High Tc Superconductors," Proceedings of the 11th Int. Conf. on CVD, Seattle, WA (1990).

58. Zhang, J. M., Wessels, B. W., Tonge, L. M., Marks, T. J., "Formation of oriented high T_c superconducting Bi-Sr-Ca-Cu-O thin films on silver by organometallic chemical vapor deposition," Appl. Phys. Lett., 56(10), 976 (1990).

59. Endo, K., Hayashida, S., Ishiai, J., Matsuki, Y., Ikado, Y., Misawa, S., and Yoshida, S., "Preparation of As-Deposited Bi-Sr-Ca-Cu-O Films with High T_c Superconducting Phase by Metalorganic Chemical Vapor Deposition," Jap. J. of Appl. Phys., 29(2), L294 (1990).

60. Nemoto, M. and Yamanaka, M., "Chemical Vapor Deposition of superconducting Bi-Sr-Ca-Cu-O films using fluorocarbon-based precursors," J. Mater. Res., 5(1), 1 (1990).

61. Natori, K. Yoshizawa, S., Yoshino, J., and Kukimoto, H., "Preparation of Pb-Doped Bi-Sr-Ca-Cu-O Superconducting Thin Films Grown by Chemical Vapor Deposition," Jap. J. of Appl. Phys., 1930 (1990).

62. Yamane, H., Kurosawa, H., Hirai, T., Iwasaki, H., Kobayashi, N., and Muto, Y., "Formation of Bismuth Strontium Calcium Copper Oxide Superconducting Films by Chemical Vapor Depostion.

63. Hamaguchi, N., Gardiner, R., Kirlin, P. S., Dye, R., Hubbard, K. M., and Muenchausen, R. E., "Metalorganic chemical vapor deposition of $Tl_2Ba_2CaCu_2O_x$ superconducting thin films using fluorinated b-diketonate source reagents," Appl. Phys. Lett., 57(20), 2136 (1990).

64. Richeson, D. S., Tonge, L. M., Wang, X. K., Marcy, H. O., Marks, T. J., Ketterson, J. B., Chang, R. P. H., and Kannewurf, C. R., "New hybrid physcial vapor deposition/organometallic chemical vapor deposition route to high T_c Tl-Ba-Cu-Ca-O thin films," Appl. Phys. Lett., 55(26), 2778 (1989).

65. Richeson, D. S., Tonge, L. M., Zhao, J., Zhang, J., Marcy, H. O., Marka, T. J., Wessels, B. W., and Kannewurf, C. R., "Organometallic chemical vapor deposition routes to high T_c superconducting Tl-Ba-Ca-Cu-O films," Appl. Phys. Lett., 54(21), 2154 (1989).

66. Zhang, K. Boyd, E. P., Kwak, B. S., Wright, A. C., and Erbil, A., "Metalorganic chemical vapor deposition of $Tl_2CaBa_2Cu_2O_y$ superconducting thin films on sapphire," Appl. Phys. Lett., 55(12), 1258 (1989).

67. Chew, N. G., Goodyear, S. W., Edwards, J. A., Satchell, J. S., Blenkinsap, S. E., and Humphreys, R. G., "The Effect of Small Changes in Composition on the Electrical and Structural Properties of $YBa_2Cu_3O_7$ Thin Films," Appl. Phys. Lett.

68. Mogro-Campero, A., and Turner, L. G., "Lower temperature postannealing of thin films of YBa2Cu3O7 at lower oxygen partial pressure," submitted to Appl. Phys. Lett., 8/90.

69. Feenstra, R., Lindemer, T. B., Budai, J. D., and Galloway, M. D., "Effect of Oxygen Pressure on the Synthesis of YBa2Cu3O7-X Thin Films By Post Deposition Annealing," submitted to J. of Appl. Phys., 9/90.

70. Lackey, W. J., Carter, W. B., Hanigofsky, J. A., Hill, D. N., Barefield, E. K., Neumeier, G., O'Brien, D. F., Shapiro, M. J., Thompson, J. R., Green, A. J., and Moss, T. S., "Rapid Chemical Vapor Deposition of superconducting $YBa_2Cu_3O_x$," Appl. Phys. Lett., 56(12), 1175 (1990).

71. Lackey, W. J., Hanigofsky, J. A., Shapiro, M. J., Carter, W. B., Hill, D. N., Barefield, E. K., Judson, E. A., O'Brien, D. F., Chung, Y. S., Moss, T. S., and More, K. L., "Preparation of superconducting wire by deposition of $YBa_2Cu_3O_x$ onto fibers," Proceedings of the 11th International CVD Conference, Seattle, WA, Nov. 1990.

72. Fitzer, E. and Gadow, R., "Fiber-Reinforced Silicon Carbide," Am. Ceram. Soc. Bull., 65(2), 326 (1986).

73. P.H. Higgs, R.L. Finicle, R.J. Bobka, E.J. Seldin, and K.J. Zeitsch, "Research and Development on Advanced Graphite Materials," Report No. WADD TR 61-72, May 1964.

74. K. Brennfleck, M. Dietrich, E. Fitzer, and D. Kehr, "Chemical Vapor Deposition of Superconducting Niobium Carbonitride Films on Carbon Fibers," pp. 300-14 in Proceedings on the Seventh International Conference on Chemical Vapor Deposition, The Electrochemical Society, Inc., Princeton, NJ, 1979.

75. K. Brennfleck and E. Fitzer, "SiC-Coatings as Protective Layers for Carbon Fibers and Adhesion Promoters for Superconducting Niobium Carbonitride," ibid., pp. 370-75.

76. K. Brennfleck, E. Fitzer, G. Schoch, and M. Dietrich, "CVD of SiC-Interlayers and Their Interaction with Carbon Fibers and with Multi-Layered NbN Coatings," pp. 649-62 in Proceedings of the Ninth International Conference on Chemical Vapor Deposition, The Electrochemical Society, Inc., Pennington, NJ, 1984.

77. H.E. DeBolt, V.J. Krukokonis, R.M. Neff, F.E. Wawner, SAMPE, "Chemical Vapor Deposition of Boron on a Carbon Monofilament Substrate," Materials Review for '72, Vol. 17, pp. II-B-Two-1 - II-B-Two-10.

78. H.E. DeBolt, V.J. Krukonis, and F.E. Wawner, Jr., "High Strength, High Modulus Silicon Carbide Filament via Chemical Vapor Deposition," ibid., pp. 168-75.

79. F.S. Galasso, R.D. Veltri, and D.A. Scola, Study of High Resistance Inorganic Coatings on Graphite Fibers, United Technologies Research Center, East Hartford, CT, NASA-CR-159078, June, 1979.

80. J.A. Cornie, "Characterization, Shaping, and Joining of SiC/Superalloy Sheet for Exhaust System Components," Electric Corp., Report No. NASA CR-135301, July, 1977.

81. L. Aggour, E. Fitzer, E. Ignotowitz, and M. Sahebkar, "Chemical Vapour Deposition of Pyro-Carbon, SiC, TiC, TiN, Si and Ta on Different Types of Carbon Fibers," Carbon 12, 358-62 (1974).

82. L.R. Newkirk, R.E. Riley, H. Sheinberg, F.A. Valencia, and T.C. Wallace, "Preparation of Unidirectional Fiber Reinforced Tantalum Carbide Composites," pp. 488-98 in Proceedings of the Seventh International Conference on Chemical Vapor Deposition, The Electrochemical Society, Inc., Princeton, NJ, 1979.

83. Lackey, W. J., Hanigofsky, J. A., Groves, M. T., and Heaney, J. A., "Continuous Fiber Coating System," Ceram. Eng. Sci. Proc., 12(7-8), 1048-63 (1991).

84. Cheung, C. T., and Ruckenstein, E., "Superconductor-substrate interations of the Y-Ba-Cu oxide," J. Mater. Res., 4(1), 1 (1989).

85. Shapiro, M. J., More, K. L., Lackey, W. J., Hanigofsky, J. A., Hill, D. N., Carter, W. B., Barefield, E. K., Judson, E. A., O'Brien, D. F., Patrick, R., Chung, Y. S., and Moss, T. S., "Interaction of Chemically Vapor Deposited $YBa_2Cu_3O_x$ With Yttria Stabilized Zirconia Substrates," J. Am. Ceram. Soc., 74(8), 2021-24, (1991).

86. Shapiro, M. J., Ph.D dissertation, "Chemical Vapor Deposition of Silver Films for Superconducting Wire Applications," Georgia Institute of Technology, Atlanta, GA, March, 1991.

87. Kevex Quantex Software Reference Manual Version VI, Kevex Instruments, San Carlos, CA, Revised in 1989.

88. Judson, E. A., M.S. Thesis, "An Analysis of Preferred Orientation in $YBa_2Cu_3O_x$ Superconducting Films Deposited by CVD on Single and Polycrystalline Substrates," Georgia Institute of Technology, Atlanta, GA, June, 1991.

89. Smits, F. M., "Measurement of Sheet Resistivities with the 4-point Probe," Bell System Technical Journal, Vol. 37, p. 711, 1958.

90. Hollabough, C. M., Hahem, L. A., Reiswig, R. D., White, R. W., and Wagner, P., Nucl. Technol., 35, p. 537. 1977.

91. Newkirk, L. R. and Valencia, F. A., U.S. Patent No. 4,202,931, May 13, 1980.

92. Shapiro, M. J., Lackey, W. J., Hanigofsky, J. A., Hill, D. N., Carter, W. B., Barefield, E. K., "Chemical Vapor Deposition of Silver Films for Superconducting Wire Applications," submitted to the J. of Less-Common Metals, July, 1991.

93. O'Brien, D. F., M.S. Thesis, "Annealing Study of $YBa_2Cu_3O_x$ Deposited by CVD on Ceramic Fiber Tows," Georgia Institute of Technology, Atlanta, GA, June, 1991.

94. Hseuh, C. H., Becher, P. F., and Lackey, W. J., "Thermal and Mechanical Stresses in Superconducting Coatings on Fibers," J. Appl. Phys., 70(3), p. 1337-44, August, 1991.

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