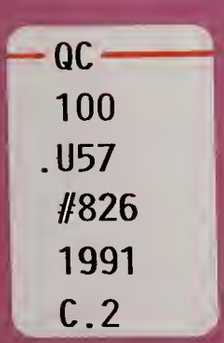
United States Department of Commerce
National Institute of Standards and Technology

NIST Special Publication 826

High-Temperature Superconductivity: Abstracts of NIST Publications, 1987–1991

Mary E. DeWeese, Editor



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Edited by
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Boulder, CO 80303

Supersedes NIST Special Publication 759

November 1991

U.S. Department of Commerce
Robert A. Mosbacher, Secretary

National Institute of Standards and Technology
John W. Lyons, Director

National Institute of Standards
and Technology
Special Publication 826
Natl. Inst. Stand. Technol.
Spec. Publ. 826
90 pages (Nov. 1991)
CODEN: NSPUE2

U.S. Government Printing Office
Washington: 1991

For sale by the Superintendent
of Documents
U.S. Government Printing Office
Washington, DC 20402

CONTENTS

ABBREVIATIONS

CAC = Center for Analytical Chemistry,
National Measurement Laboratory

CBS = Center for Basic Standards,
National Measurement Laboratory

CCP = Center for Chemical Physics,
National Measurement Laboratory

CEEE = Center for Electronics and Electrical Engineering,
National Engineering Laboratory

CRR = Center for Radiation Research,
National Measurement Laboratory

EEEL = Electronics and Electrical Engineering Laboratory
(formerly CEEE)

IMSE = Institute for Materials Science and Engineering

MSEL = Materials Science and Engineering Laboratory

NML = National Measurement Laboratory

FOREWORD

Robert A. Kamper

The National Institute of Standards and Technology (formerly the National Bureau of Standards) has been involved with research in superconductivity since soon after Kammerlingh Onnes's original discovery in 1911. Our first contribution to the literature was the Silsbee hypothesis to account for the critical current of a type-I superconductor, published by Francis Silsbee in 1916. The discoveries of the high-field superconductors in the late fifties and the Josephson effect in the early sixties introduced the possibility of serious engineering with superconductors and were followed by significant expansion of the program at NBS. A formal program to promote the application of superconductivity to physical standards and measurement techniques was established in 1969 and has been an active contributor to the development of superconducting electronics ever since. In the early seventies a program to define and establish techniques to measure the engineering characteristics of practical high-field superconductors was established. This has contributed to the development of reliable conductors and to the success of many large-magnet projects.

The discovery of the high-temperature superconductors introduced the need to extend research from metals to ceramics, for which NBS was also well prepared, with an established program in ceramics and an extensive array of facilities to analyze and map the composition, structure, and electronic energy spectrum of materials in a wide range of scales.

This collection of abstracts represents the first results of applying all these resources to high-temperature superconductors. Many of the papers have authors from several institutions. The National Institute of Standards and Technology encourages collaborative research projects to maximize the benefit of its resources to the U.S. research and development community. Anyone wishing to discuss the possibility of a collaborative research project should feel free to make contact with any of the NIST authors represented here.

HIGH-TEMPERATURE SUPERCONDUCTIVITY: ABSTRACTS OF NIST PUBLICATIONS, 1987-1991

Mary E. DeWeese, Editor

This is a collection of abstracts from 243 papers published between March 1987 and August 1991 covering various aspects of superconductivity research. The work of nine divisions of the National Institute of Standards and Technology (formerly the National Bureau of Standards) in both Boulder, Colorado, and Gaithersburg, Maryland, is represented.

Key words: bismuth superconductors; critical current; critical field; critical temperature; high T_c; high-temperature superconductors; low-temperature superconductors; superconducting ceramics; superconductors; thallium superconductors; YBCO; yttrium superconductors

1. COMPOSITIONAL ANALYSIS

STANDARD X-RAY DIFFRACTION POWER PATTERNS OF FIFTEEN CERAMIC PHASES, Winnie Wong-Ng, Howard F. McMurdie, Boris Paretzkin, Camden R. Hubbard, Alan L. Dragoo, and James M. Stewart,* Powder Diffraction, Vol. 2, No. 2, pp. 106-117 (June 1987). See pp. 191-201 for accelerated publication with additional coauthors, Yuming Zhang* and Katherine L. Davis. [IMSE/Ceramics Division]
*University of Maryland

See following abstract.

STANDARD X-RAY DIFFRACTION POWDER PATTERNS OF SIXTEEN CERAMIC PHASES, Winnie Wong-Ng, Howard F. McMurdie, Boris Paretzkin, Yuming Zhang,* Katherine L. Davis, Camden R. Hubbard, Alan L. Dragoo, and James M. Stewart,* Powder Diffraction, Vol. 2, No. 3, pp. 191-201 (September 1987). [IMSE/Ceramics Division]
*University of Maryland

The following sixteen reference patterns of boride, silicide, nitride, and oxide ceramics represent the second group of reference patterns of ceramic phases measured at the National Bureau of Standards under the project "High Quality Reference Patterns and Total Digital Powder Patterns of Technologically Important Ceramic Phases." Included in the sixteen reference patterns are reports for two high T_c superconducting oxide phases ($CuLa_{1.8}Sr_{0.2}O_4$ and $Ba_2Cu_3YO_7$) plus one related phase ($BaCuY_2O_5$). In addition to these new phases, five other patterns represent phases previously not contained in the PDF and eight represent major corrections to data in the file. The general methods of producing these X-ray powder diffraction reference patterns are described in this journal, Vol. 1, No. 1, p. 40 (1986).

X-RAY POWDER CHARACTERIZATION OF $Ba_2YCu_3O_{7-x}$, W. Wong-Ng, R.S. Roth, L.J. Swartzendruber, L.H. Bennett, C.K. Chiang, F. Beech, and C.R. Hubbard, Advanced Ceramic Materials: Ceramic Superconductors, Vol. 2, No. 3B, pp. 565-576 (July 15, 1987). [IMSE/Ceramics Division]

The X-ray powder diffraction technique was used in order to characterize the high T_c superconductor phase $Ba_2YCu_3O_{7-x}$ prepared under different conditions. High quality reference powder patterns for three compositions are presented. An attempt is made to correlate the differences of lattice parameters with oxygen content and superconductivity.

MEASUREMENT OF SUPERCONDUCTOR STOICHIOMETRY BY PROMPT AND DELAYED NEUTRON ACTIVATION, Richard M. Lindstrom, Transactions, American Nuclear Society, Nuclear Activation Analysis/Applications-I, Vol. 56, pp. 231 (1988). [CAC/Inorganic Analytical Research Division]

Prompt and delayed neutron activation analysis have been applied to the nondestructive measurement of the major metals in small samples of $YBa_2Cu_3O_7$ superconductors, starting materials, and intermediate products. The small activation cross section and low gamma ray

energies of Y-90 m make the determination of yttrium imprecise in the presence of the large quantities of Cu-64 radioactivity. The physical parameters for neutron-capture prompt gamma-ray activation analysis (PGAA) are more attractive than delayed counting for stoichiometry measurements. The PGAA spectra of Y, Ba, and Cu show three, four, and six analytically useful gamma ray lines respectively, with nicely balanced intensities for the composition range of interest. A 100 mg sample gives a Poisson precision for yttrium (the weakest element) of 2% after only a 2 hour irradiation. Since the concentrations and mole ratios Y/Cu and Ba/Cu may be calculated immediately after the irradiation ends, the results can be returned within a day.

APPLICATION OF WAVELENGTH DISPERSIVE DIGITAL COMPOSITIONAL MAPPING TO HIGH-TEMPERATURE SUPERCONDUCTORS, R.B. Marinenko, D.E. Newbury, D.S. Bright, R.L. Myklebust, and J.E. Blendell, Microbeam Analysis—1988, D.E. Newbury, Editor, San Francisco Press, pp. 37-40 (1988). [CAC/Gas and Particulate Science Division, and IMSE/Ceramics Division]

The new high-T_c superconductors are advanced ceramic materials prepared from oxide powders which are pressed and sintered. Their physical properties depend on the microstructure, which in turn depends strongly on the processing sequence used in their production. We have used wavelength-dispersive (WDS) x-ray compositional mapping to study the microstructure of the $Ba_2YCu_3O_{6+x}$ ceramics being produced at the National Bureau of Standards so as to understand better the processing procedures and the resulting physical properties.

MEASUREMENT OF SUPERCONDUCTOR STOICHIOMETRY BY PROMPT AND DELAYED NEUTRON ACTIVATION, Richard M. Lindstrom, Transactions, American Nuclear Society, Nuclear Activation Analysis/Applications-I, Vol. 56, pp. 231 (1988). [CAC/Inorganic Analytical Research Division]

Prompt and delayed neutron activation analysis have been applied to the nondestructive measurement of the major metals in small samples of $YBa_2Cu_3O_7$ superconductors, starting materials, and intermediate products. The small activation cross section and low gamma ray energies of Y-90 m make the determination of yttrium imprecise in the presence of the large quantities of Cu-64 radioactivity. The physical parameters for neutron-capture prompt gamma-ray activation analysis (PGAA) are more attractive than delayed counting for stoichiometry measurements. The PGAA spectra of Y, Ba, and Cu show three, four, and six analytically useful gamma ray lines respectively, with nicely balanced intensities for the composition range of interest. A 100 mg sample gives a Poisson precision for yttrium (the weakest element) of 2% after only a 2 hour irradiation. Since the concentrations and mole ratios Y/Cu and Ba/Cu may be calculated immediately after the irradiation ends, the results can be returned within a day.

INITIAL STAGES OF DEGRADATION OF SUPERCONDUCTOR SURFACES: 0_2, H_2, CO_2, AND CO CHEMISORPTION IN La_2-xSr_xCuO_4, Richard L. Kurtz, Roger Stockbauer, Theodore E. Madey, Donald Mueller,* Arnold Shih,* and Louis Toth,* Physical Review B, Vol. 37, No. 13, pp. 7936-7939 (May 1, 1988). [CCP/Surface Science Division]

*Naval Research Laboratory

The initial stages of degradation of high-T_c superconductor surfaces by interaction with atmospheric gases have been studied using ultraviolet photoelectron spectroscopy. O_2 has little effect, while H_2O adsorbs dissociatively with an initial sticking coefficient $S_0 \sim 1$, and forms a surface hydroxide. CO_2 adsorbs forming a carbonatelike surface species with an initial sticking coefficient of ~ 0.3. CO is observed to result in similar adsorbed surface species with a much lower sticking coefficient of $\sim 3 \times 10^{-4}$.

APPLICATION OF WAVELENGTH DISPERSIVE DIGITAL COMPOSITIONAL MAPPING TO HIGH-TEMPERATURE SUPERCONDUCTORS, R.B. Marinenko, D.E. Newbury, D.S. Bright, R.L. Myklebust, and J.E. Blendell, Microbeam Analysis—1988, D.E. Newbury, Editor, San Francisco Press, pp. 37-40 (1988). [CAC/Gas and Particulate Science Division, and IMSE/Ceramics Division]

The new high-T_c superconductors are advanced ceramic materials prepared from oxide powders which are pressed and sintered. Their physical properties depend on the microstructure, which in turn depends strongly on the processing sequence used in their production. We have used wavelength-dispersive (WDS) x-ray compositional mapping to study the microstructure of the $_{B}a_2YCu_3O_{6+x}$ ceramics being produced at the National Bureau of Standards so as to understand better the processing procedures and the resulting physical properties.

STANDARD X-RAY DIFFRACTION POWDER PATTERNS OF FOURTEEN CERAMIC PHASES, Winnie Wong-Ng, Howard F. McMurdie, Boris Paretzkin, Alan L. Dragoo, Powder Diffraction, Vol. 3, No. 2, pp. 113-121 (June 1988). [IMSE/Ceramics Division]

The following fourteen reference patterns of carbide, nitride, telluride, and oxide ceramics are reported. Included in the fourteen reference patterns are data for three high T_c superconducting oxide and related phases (Ba_2CuO_3, $CuSrO_2$, and $Ba_2Cu_3YO_6$). The general methods of producing these X-ray powder diffraction reference patterns are described in this journal, Vol. 1, No. 1, p. 40 (1986).

STANDARD X-RAY DIFFRACTION POWER PATTERNS OF FIFTEEN CERAMIC PHASES, Winnie Wong-Ng, Howard F. McMurdie, Boris Paretzkin, M.A. Kuchinski, and Alan L. Dragoo, Powder Diffraction, Vol. 4, No. 2, pp. 106-121 (June 1989). [IMSE/Ceramics Division]

Fifteen reference patterns of oxide ceramics are reported. Included in the fifteen reference patterns are data for nine high critical temperature superconducting oxide and related phases $Ba_2Cu_3ErO_7$, $Ba_{3.2}Cu_{1.7}Er_{0.8}O_{6.1} \cdot xCO_2$, $Ba_2Cu_3HoO_7$, $Ba_2Cu_3(Pr_{0.5}Y_{0.5})O_7$, $Ba_3Dy_4O_9$, $Ba_3Yb_4O_9$, $(Ba_{0.6}Sr_{0.4})_2Cu_3YO_7$, $(Ba_{0.8}Sr_{0.2})_2Cu_3YO_7$, and $CuNd_2O_4$. The general methods of producing these X-ray powder diffraction reference patterns were described previously in this journal (Vol. 1, No. 1, pg. 40 (1986)). The symbols used in this article are defined in the PDF cards.

STANDARD X-RAY DIFFRACTION POWER PATTERNS OF FOURTEEN CERAMIC PHASES, Winnie Wong-Ng, Howard F. McMurdie, Boris Paretzkin, M.A. Kuchinski, and Alan L. Dragoo, Powder Diffraction, Vol. 4, No. 1, pp. 40-54 (1989). [IMSE/Ceramics Division]

Fourteen reference patterns of oxide ceramics are reported. Included in the fourteen reference patterns are data for nine high critical temperature superconducting oxide related phases: ($BaCuEr_2O_5$, $Ba_2Cu_3EuO_7$, $BaCuGd_2O_5$, $Ba_2Cu_3GdO_7$, $Ba_{2.4}Cu_{1.8}La_{3.6}O_{9.6}$, $BaCu_5La_4O_{13}$, $Ba_{1.9}Cu_3La_{1.1}O_7$, $Ba_2Cu_3SmO_7$ and $(Ba_{0.4}Sr_{0.6})_2Cu_3YO_7$. The general methods of producing these X-ray powder diffraction reference patterns were described previously in this journal (Vol. 1, No. 1, pg. 40 (1986)). The symbols used in this article are defined in the PDF cards.

2. CRITICAL CURRENT AND CRITICAL TEMPERATURE

TRANSPORT CRITICAL-CURRENT CHARACTERISTICS OF $Y_1Ba_2Cu_3O_x$, J.W. Ekin, A.J. Panson,* A.I. Braginski,* M.A. Janocko,* M. Hong,† J. Kwo,† S.H. Liou,† D.W. Capone, II,‡ and B. Flandermeyer,‡ Materials Research Society, Vol. EA-11, pp. 223-226 (June 1987). [CEEE/Electromagnetic Technology Division]

*Westinghouse Research & Development Center
†AT&T Bell Laboratories, Murray Hill
‡Argonne National Laboratory

Voltage versus current (V-I) characteristics were measured at magnetic fields up to 24 T at a temperature of 77 K in several $Y_1Ba_2Cu_3O_x$ samples fabricated at different laboratories. Critical temperatures, T_c, measured by resistivity were about 93 K. All samples showed linear V-I characteristics at current levels much greater than the critical current, I_c. However, the slope was significantly less than the normal resistance at T_c. The slope increased with magnetic field and reached the normal resistance value only at fields greater than 24 T. Values of the transport critical-current density J_c near zero magnetic field were generally low and variable (about 1 to 200 A/cm^2). The transport J_c fell sharply when magnetic field was applied, decreasing by about an order of magnitude between 10^{-3} T and 1 T. This leads to an effective upper critical field for transport critical currents in $Y_1Ba_2Cu_3O_x$ that is significantly less than the upper critical field defined by the field at which the resistance increases to the normal-state value. The transport J_c appears to be significantly less than J_c calculated from magnetization data on similar samples. These results are consistent with the transport critical-current in the $Y_1Ba_2Cu_3O_x$ system being dominated by a "weak-link" region between high-J_c grains.

STUDIES OF NbTi STRANDS FROM CORELESS RUTHERFORD CABLES, L.F. Goodrich, E.S. Pittman, J.W. Ekin, and R.M. Scanlan, IEEE Transactions on Magnetics, Vol. MAG-23, No. 2, pp. 1642-1645 (March 1987). [CEEE/Electromagnetic Technology Division]

The electromechanical properties of NbTi strands extracted from coreless Rutherford cables were studied to clarify the relative effects of strand location and field angle on current degradation that occurs in cables that have been compacted into a keystone shape. Detailed critical-current measurements were made on two samples which were fabricated under controlled conditions. These are prototype cables for high energy physics applications. Specific factors that are addressed are the nature, location, and amount of degradation. This information is intended to lead to methods for reducing the amount of critical-current degradation in cable manufacture.

RELATIONSHIPS BETWEEN CRITICAL CURRENT AND STRESS IN NbTi, J.W. Ekin, IEEE Transactions on Magnetics, Vol. MAG-23, No. 2, pp. 1634-1637 (March 1987). [CEEE/Electromagnetic Technology Division]

The effects of various types of stress on the critical current of a multifilamentary NbTi superconductor are reported. Degradation of critical current due to axial tension applied at 4 K, transverse compression applied at 4 K, and hairpin bending strain applied at room temperature has been measured. The degradation from axial tension is much greater than from transverse compression in many practical cases because the soft copper matrix limits the build-up of

transverse compression. The degradation from typical levels of transverse compression is only about 4% at 8 T, for example. For axial tension, on the other hand, higher stresses can occur that will degrade the critical current by 24%, for example, at 7 T and 2.7% strain. Both the axial-tensile and the transverse-compressive stress effects are about 98% reversible; thus the degradation will be seen only when the conductor is under operational stress. The results indicate that a primary origin of the critical current degradation in NbTi is a stress-induced reversible decrease in the upper critical field.

EVIDENCE FOR WEAK LINK AND ANISOTROPY LIMITATIONS ON THE TRANSPORT CRITICAL CURRENT IN BULK POLYCRYSTALLINE $Y_1Ba_2Cu_3O_x$, J.W. Ekin, A.I. Braginski,* A.J. Panson,* M.A. Janocko,* D.W. Capone, II,†
N.J. Zaluzec,† B. Flandermeyer,† O.F. de Lima,† M. Hong,‡ J. Kwo,‡ and S.H. Liou,‡ Journal of Applied Physics, Vol. 62, No. 12, pp. 4821-4828 (December 15, 1987). [CEEE/Electromagnetic Technology Division]

*Westinghouse Research & Development Center
†Argonne National Laboratory
‡AT&T Bell Laboratories, Murray Hill

Measurements of the transport critical-current density (J_c), magnetization J_c, and magnetoresistance in a number of bulk sintered samples of $Y_1Ba_2Cu_3O_x$ from several different laboratories indicate that the transport J_c is limited by weak-link regions between high J_c regions. The weak-link J_c has a Josephson character, decreasing by two orders of magnitude as the magnetic field is increased from 0.1 to 10 mT at 77 K. An examination of the grain-boundary region in $Y_1Ba_2Cu_3O_x$ shows no observable impurities or second phases to the scale of the [001] lattice planes ($\sim$ 12 Å). The effect of intrinsic conduction anisotropy is discussed. A current-transfer model is proposed in which weak conduction along the c axis plays a role in limiting J_c at grain boundaries. Orienting the grains in the powder state during processing may result in enhanced transport J_c in bulk conductors.

TRANSPORT CRITICAL CURRENT CHARACTERISTICS OF $Y_1Ba_2Cu_3O_x$ AND POSSIBILITIES FOR ITS ENHANCEMENT, J.W. Ekin Advanced Ceramic Materials: Ceramic Superconductors, Vol. 2, No. 3B, pp. 586-592 (July 1987). [CEEE/Electromagnetic Technology Division]

Several general processing methods for increasing the critical current density, J_c, in bulk sintered $Y_1Ba_2Cu_3O_x$ superconductor are outlined. Data indicate that the transport J_c in bulk polycrystalline specimens is dominated by a weak-link region between high-J_c grains and that potentially much higher J_c may be possible. Two possible causes of such a weak-link phenomenon are considered: low-T_c phases or impurities localized in the grain boundary region, and anisotropy of the superconducting properties. Several methods for minimizing the weak-link effects to increase the J_c are discussed.

JOSEPHSON JUNCTION MODEL OF CRITICAL CURRENT IN GRANULAR $Y_1Ba_2Cu_3O_{7-\delta}$ SUPERCONDUCTORS, Robert L. Peterson and Jack W. Ekin, Physical Review B Rapid Communications, Vol. 37, No. 16, pp. 9848-9851 (June 1, 1988). [CEEE/Electromagnetic Technology Division]

We calculate the transport critical current density in a granular superconductor in magnetic fields below about 5×10^{-3} T. The field dependence in this region is assumed to be controlled by intragranular or intergranular Josephson junctions. Various model calculations are fit to transport critical current bulk $Y_1Ba_2Cu_3O_{7-\delta}$ ceramic superconductors, whose average grain size somewhat exceeds 10 μm. The results yield an average junction cross-sectional area (thickness $\times$ length) of 4-6 μm^2. If the junctions are at the grain boundaries, a London penetration depth of about 150-300 nm is inferred, consistent with other estimates. We conclude that Josephson junctions are limiting the transport critical current in these samples and that they lie at the grain boundaries. The parameters of the fit are not consistent with Josephson junctions at twinning boundaries.

MAGNETIC-FIELD-MODULATED MICROWAVE-ABSORPTION DETECTION IN A Bi-Sr-Ca-Cu-O SUPERCONDUCTOR, J. Bohandy,† F.J. Adrian,† B.F. Kim,† K. Moorjani,† R.D. Shull, L.J. Swartzendruber, L.H. Bennett, and J.S. Wallace, Journal of Superconductivity, Vol. 1, No. 2, pp. 191-196 (1988). [IMSE/Metallurgy Division]

†The Johns Hopkins University, Applied Physics Laboratory

Superconducting transitions are observed at 110 K, 100 K, and 72 K in a nominally $BiSrCaCu_2O_x$ ceramic using the novel technique of magnetic-field-modulated microwave-absorption detection. The response of the $BiSrCaCu_2O_x$ ceramic to an external magnetic field differs markedly from that of YBa_2Cu_3O, and, in particular, $-dT_c/dH$ is much greater in the bismuth sample.

IRREGULARITY IN Nb-Ti FILAMENT AREA AND ELECTRIC FIELD *VERSUS* CURRENT CHARACTERISTICS, J.W. Ekin, Cryogenics 1987, Vol. 27, pp. 603-608 (November 1987). [CEEE/Electromagnetic Technology Division]

There is a correlation between irregularity in filament area ("sausaging") and the shape of a superconductor's electric field (E) versus current (I) relationship. The shape of the E-I characteristic is quantified in terms of the resistive transition parameter, n, defined by $E \propto I^n$. Low values of n less than about 20 correlate with a wide filament diameter distribution, while n values over 50 correspond to a distribution more than 2.5 times smaller. It is proposed that the low-field (constant) value of n be used as an index of filament quality in evaluating different superconductors for practical applications. A model is also suggested to explain this effect in terms of a locally depressed filament critical current, which forces current to transfer across the normal matrix material into neighboring filaments. The relationship between n and the statistical distribution of filament diameters may be useful as an easy method of estimating the extent of sausaging in practical multifilamentary Nb-Ti superconductors from measurements of n.

TRANSVERSE STRESS EFFECT ON MULTIFILAMENTARY Nb_3Sn SUPERCONDUCTOR, J.W. Ekin, Advances in Cryogenic Engineering Materials, Vol. 34, pp. 547-552 (1988). [CEEE/Electromagnetic Technology Division]

A large reversible degradation of the critical current of multifilamentary Nb_3Sn superconductors has been observed under the application of uniaxial compressive stress applied transverse to the conductor axis at 4 K. In bronze-process multifilamentary Nb_3Sn, the onset of significant

degradation occurs at about 50 MPa. The intrinsic effect of transverse stress on the upper critical field is about ten times greater than for axial stress. Although transverse stress on Nb_3Sn filaments is less than axial stress in most applications, it will need to be considered in the internal stress design of large magnets because of the greater sensitivity of Nb_3Sn to transverse stress. The effect scales with conductor thickness and this will place limits on conductor dimensions and the spacing between distributed reinforcement in large magnets.

DEVELOPMENT OF STANDARDS FOR SUPERCONDUCTORS, INTERIM REPORT, JANUARY 1986-DECEMBER 1987, L.F. Goodrich, ed., National Bureau of Standards NBSIR 88-3088, 75 p. (February 1988). [CEEE/Electromagnetic Technology Division]

A cooperative program with the Department of Energy, the National Bureau of Standards, other national laboratories, and private industry is in progress to develop standard measurement practices for use in large scale applications of superconductivity. This report describes research for the period January 1986 through December 1987. It contains the results of critical current studies on the effect of power-supply current ripple, measurements on cable strands, an interlaboratory comparison (round robin) on a large NbTi monolithic conductor, and a Nb_3Sn round robin. Several useful current supply circuits have been developed. The reduction in coupling losses in multifilamentary NbTi conductors has been addressed by a study of the magnetic properties of matrix material consisting of dilute alloys of Mn in Cu. In addition, vibrating-sample magnetometry is shown to be adaptable to the measurement of coupling losses, in addition to hysteresis losses, in multifilamentary conductors.

CURRENT RIPPLE EFFECT ON SUPERCONDUCTIVE DC CRITICAL CURRENT MEASUREMENT, L.F. Goodrich, S.L. Bray, and A.F. Clark, Advances in Cryogenic Engineering Materials, Vol. 34, pp. 1019-1026 (1988). [CEEE/Electromagnetic Technology Division]

The effect of sample-current power-supply ripple on the measurement of dc critical current is reported. Measurements were made on multifilamentary NbTi superconductor. In general, ripple in a current supply becomes more significant above 500 A because effective filtering is difficult. The presence of current ripple reduces the measured dc critical current. Ripple also causes noise at the input to the voltmeter used for the measurements. The quantitative effect of current ripple was studied using a battery current supply modified to allow the creation of ripple current with variable frequency and amplitude. Problems common to all large-conductor critical-current measurements are discussed.

EFFECT OF TRANSVERSE COMPRESSIVE STRESS ON THE CRITICAL CURRENT AND UPPER CRITICAL FIELD OF Nb_3Sn, J.W. Ekin, Journal of Applied Physics, Vol. 62, No. 12, pp. 4829-4834 (December 15, 1987). [CEEE/Electromagnetic Technology Division]

A large reversible degradation of the critical current of multifilamentary Nb_3Sn superconductors has been observed when uniaxial compressive stress is applied transverse to the conductor at 4 K. In bronze-process multifilamentary Nb_3Sn, the onset of significant degradation occurs at about 50 MPa. In an applied field of 10 T, the magnitude of the effect is about seven times larger for transverse stress than for stress applied along the conductor axis. The transverse stress effect

increases with magnetic field and is associated with a reversible degradation of the upper critical field. The intrinsic effect of transverse stress on the upper critical field is about 10 times greater than for axial stress. Although axial stresses on the Nb_3Sn filaments are greater than transverse stresses in most applications, the transverse stress effect will need to be considered in the internal design of large magnets because of the greater sensitivity of Nb_3Sn to transverse stress. It is shown that the transverse stress from the Lorentz force on the conductor is proportional to conductor thickness. This will place limits on conductor dimensions and the spacing between distributed reinforcement in large magnets. The effect may be particularly significant in cabled conductors where large transverse stress concentrations can occur at strand crossover points.

BATTERY POWERED CURRENT SUPPLY FOR SUPERCONDUCTOR MEASUREMENTS, S.L. Bray, L.F. Goodrich, W.P. Dube, Review of Scientific Instruments, Vol. 60, No. 2, pp. 261-264 (February 1989). [CEEE/Electromagnetic Technology Division]

To measure the critical current of superconductors, a high output current supply is required. In addition to high current capability, the supply should be designed to reduce ground loop problems, respond linearly to an input control signal, and minimize output noise. A current supply with these qualifications has been constructed and tested. Although the supply was originally designed for testing conventional superconductors at high current levels, it has also been successfully used in measurements on the high-critical-temperature ceramic superconductors where the maximum current output was less than 1 A. The supply can produce 1000 A output current with a noise level of approximately 0.05 A peak-to-peak. The specifics of the current supply's designs and performance are given.

CRYOGENIC BATHYSPHERE FOR RAPID-VARIABLE-TEMPERATURE CHARACTERIZATION OF HIGH-T_c SUPERCONDUCTORS, John Moreland, Y. Li,* R. Folsom, and T.E. Capobianco, Review of Scientific Instruments, Vol. 59, No. 12, pp. 2535-2538 (December 1988). [CEEE/Electromagnetic Technology Division]
*Chinese Academy of Physics, Beijing, China

A bathysphere consisting of an inverted Dewar flask for submersible operation in cryogenic fluids is used to measure the resistance of superconductors, including high-T_c superconducting copper oxides, as a function of temperature from 4 K to 300 K. We describe the cryostat incorporating the bathysphere and present data on NbTi (44% Ti) and $YBa_2Cu_3O_{7-\delta}$ with respective superconducting transition temperatures of 9.5 K and 91.5 K. There are several advantages to the bathysphere method. The cryostat is of simple compact design easily adapted to high-field applications where magnet bore size is a limiting factor. The sample and thermometer are thermolyzed in the dry vapor trapped at the top of the bathysphere. Temperature can be varied rapidly from 300 K to 4 K at a rate of 1 K/min with less than 0.1 K thermal lag between the sample and thermometer.

CRITICAL CURRENTS OF HIGH-T_c SUPERCONDUCTORS: PINNING, WEAK LINKS, CONDUCTION ANISOTROPY, AND CONTACT RESISTIVITIES, J.W. Ekin, R.L. Peterson, and S.L. Bray, Proceedings of the 1988 Materials Research Society International Meeting on Advanced

Materials, Tokyo, Japan, May 30-June 3, 1988, Vol. 6, pp. 135-144 (1989. [CEEE/Electromagnetic Technology Division]

The coherence length ξ of high-T_c superconductors in the c-direction is much smaller than for conventional superconductors and the c-axis lattice spacing is several times larger. This intrinsic difference has significant consequences for transport properties. Because the pinning effectiveness of defects of average size $<D>$ is greatest for $<D>/\xi = 1$ and decreases rapidly for $<D>/\xi < 1$, defects on the scale of the atomic lattice are not effective as pinning sites in conventional superconductors (where $<D> << \xi$), but can play an important role in high-T_c superconductors (where $<D> \sim \xi$, depending on crystallographic direction). Unfortunately, the size of the atomic lattice spacing relative to ξ can also create weak-link effects at lattice defects. From the practical standpoint of producing high transport critical-current density J_c in high-T_c superconductors, the conventional problem of pinning-force enhancement is replaced by weak-link minimization. The rapid decrease of J_c at low magnetic fields ($\leq 10^{-2}$ T in YBCO) is described well by a Josephson-junction weak-link model. The transport J_c is also nearly independent of the angle between the applied magnetic field and suggesting highly convoluted percolation paths in these materials. Crystallographic conduction anisotropy is a secondary limitation on J_c, but emerges as a prime determinant of transport J_c in the absence of weak-link effects. The effect of conduction anisotropy on transport J_c is described in terms of a current-transfer model. A summary is given of a method to achieve very low resistivity contacts ($\sim 10^{-10}$ Ω-cm^2) with oxide superconductors. Low resistivity contacts have been fabricated with both yttrium and thallium based compounds.

RESISTANCE MEASUREMENTS OF HIGH Tc SUPERCONDUCTORS USING A NOVEL "BATHYSPHERE" CRYOSTAT, John Moreland, Y. Li,* R.M. Folsom, and T.E. Capobianco, IEEE Transactions on Magnetics, Vol. 25, No. 2, pp. 2560-2562 (March 2, 1989). [CEEE/Electromagnetic Technology Division]
*Chinese Academy of Physics, Beijing

We have developed a novel cryostat for variable temperature testing of high temperature superconductors. The cryostat is a bathysphere consisting of an overturned stainless steel Dewar suspended in liquid helium. A sample-heater-thermometer assembly is located at the top of the encapsulated (and thermally insulated) vapor space inside of the Dewar. The sample can be rapidly cycled from 300 K to 4 K at an average rate of 1 K/min with a thermal hysteresis of less than 0.1 K. Helium vapor flows through a plug in the bottom of the bathysphere so that pressure of the vapor is roughly ambient. This provides ample heat transfer to and from the sample to maintain thermal equilibrium in the vapor space. Results for resistance versus temperature of some high temperature superconductors in a magnetic field are presented. Also various definitions for thermodynamic and practical T_c's derived from transport resistivity measurements are suggested and discussed.

Nb_3Sn CRITICAL-CURRENT MEASUREMENTS USING TUBULAR FIBERGLASS-EPOXY MANDRELS, L.F. Goodrich, S.L. Bray, and T.C. Stauffer, IEEE Transactions on Magnetics, Vol. MAG-25, No. 2, pp. 2375-2378 (March 1989). [CEEE/Electromagnetic Technology Division]

A systematic study of the effect of sample mounting techniques on the superconducting critical-current measurement was made in conjunction with the VAMAS (Versailles Agreement on Advanced Materials and Standards) interlaboratory comparison (round robin) measurements. A seemingly small change in mandrel geometry can result in a 40% change in the measured critical current of a Nb_3Sn sample at 12 T. This is a result of a change in the conductor pre-strain at 4 K caused by variation in thermal contraction between thick- and thin-walled fiberglass-epoxy composite (G-10) tubes. An approximate measure of the variations in thermal contraction (from room to liquid nitrogen temperature) indicates a 0.2% greater contraction for the thick-walled tube. This difference, combined with strain sensitivity measurements, is consistent with the observed decrease in critical current. Previous publications on the thermal contraction of G-10 have addressed the plate geometry, but not the tube geometry. The contraction of a G-10 plate is highly anisotropic. The radial contraction of a tube is different than the contraction of a plate, however, because the circumferential fiberglass is put into hoop compression by the epoxy, and the resulting contraction is a competition between the two structural components. This appears to be the source of the variation in thermal contraction with tube wall thickness.

CURRENT CAPACITY DEGRADATION IN SUPERCONDUCTING CABLE STRANDS, L.F. Goodrich and S.L. Bray, IEEE Transactions on Magnetics, Vol. MAG-25, No. 2, pp. 1949-1952 (March 1989). [CEEE/Electromagnetic Technology Division]

The electromagnetic properties of NbTi strands extracted from Rutherford cables were studied to clarify the effect of mechanical deformation, caused by the cabling process, on the current capacity of the strands. Three different cables were studied, all of which are prototypes for the Superconducting Super Collider's dipole magnets. The extracted cable strands were instrumented to allow measurement of the voltage across several key regions of mechanical deformation as a function of current and the orientation of the applied magnetic field. The resulting data are presented in terms of the strand's voltage profile as well as its critical current in order to more thoroughly characterize the conductor's electromagnetic properties. The cable strands show very localized reductions in current capacity that are well correlated with the regions of high mechanical deformation. For example, at a particular field orientation, the voltage across a portion of the strand that is only 3% of the total strand length contributes 92% of the total strand voltage. Two applied magnetic field orientations, parallel and perpendicular to the cable's width, have pronounced effects on the electrical properties of the strand. Both of these magnetic field orientations will arise in applications.

CRITICAL-CURRENT MEASUREMENTS OF Nb_3Sn SUPERCONDUCTORS: NBS CONTRIBUTION TO THE VAMAS INTERLABORATORY COMPARISON, L.F. Goodrich and S.L. Bray, Cryogenics, Vol. 29, pp. 699-709 (July 1989). [CEEE/Electromagnetic Technology Division]

Critical-current measurements on several Nb_3Sn superconductors were made as part of an interlaboratory comparison (round robin). These measurements were made in conjunction with 24 laboratories from the European economic community, Japan, and the USA as part of the Versailles Agreement on Advanced Materials and Standards (VAMAS). The results of the NBS measurements, including the effect of sample mounting techniques on the measured critical current, are given. A systematic study of the effect of measurement mandrel (tubular sample-holder made from G-10 fiberglass-epoxy composite) geometry revealed that a seemingly small

change in that geometry can result in a 40% change in the measured critical current at a magnetic field of 12 T. Specifically, the radial thermal contraction of the measurement mandrel depends on its wall thickness and, thus, so does the conductor prestrain (at 4 K) and, ultimately, the measured critical current. Techniques for reducing variation in the measured critical current are suggested.

EFFECT OF ROOM TEMPERATURE STRESS ON THE CRITICAL CURRENT OF NbTi, S.L. Bray and J.W. Ekin, Journal of Applied Physics, Vol. 65, No. 2, pp. 684-687 (January 15, 1989). [CEEE/Electromagnetic Technology Division]

The effect of axial tensile stress, applied at room temperature, on the critical current of NbTi superconducting wire was measured and compared with the effect of tensile stress applied at liquid-helium temperature (~4 K). The results of these measurements indicate that the effect on the critical current is independent of the temperature at which the stress is applied. Thus, the existing 4-K data base can be used to determine I_c degradation from room-temperature fabrication stress, cool-down stress introduced by differential contraction, as well as 4-K stress generated by the Lorentz force when the magnet is energized. To generalize these results for arbitrary matrix-to-superconductor volume ratios, the data are presented in terms of the stress on the NbTi portion of the composite conductor. Methods for determining the stress on the NbTi from the total composite load are presented.

OXYGEN ISOTOPE EFFECT IN THE SUPERCONDUCTING Bi-Sr-Ca-Cu-O SYSTEM, H. Katayama-Yoshida,* T. Hirooka,* A. Oyamada,* Y. Okabe,* T. Takahashi,* T. Sasaki,* A. Ochiai,* T. Suzuki,* A.J. Mascarenhas, J.I. Pankove, T.F. Ciszek, S.K. Deb,† R.B. Goldfarb, and Yongkang Li,‡ Physica C, Vol. 156, pp. 481-484 (1988). [CEEE/Electromagnetic Technology Division]
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An oxygen isotope effect is observed in mixed-phase Bi-Sr-Ca-Cu-O superconductors when ^{18}O is substituted for ^{16}O. The superconducting transition temperature T_c measured by electrical resistivity and magnetic susceptibility, is lowered by about 0.32 K for the higher-T_c (110 K) phase and by about 0.34 K for the lower-T_c (75 K) phase. These results suggest a measurable contribution to the superconductivity from phonons.

AIRY PATTERN, WEAK-LINK MODELING OF CRITICAL CURRENTS IN HIGH-T_c SUPERCONDUCTORS, R.L. Peterson and J.W. Ekin, Physica C, Vol. 1577, No. 2, pp. 325-333 (1989). [CEEE/Electromagnetic Technology Division]

We have measured the transport critical current density at very low magnetic fields in samples of superconducting bulk sintered $Y_1Ba_2Cu_3O_x$, $Y_1Ba_2Cu_4O_x$, and $Ho_1Ba_2Cu_3O_x$ obtained from several sources. The results are analyzed at low fields ($\leq$10 mT) with a statistical model which assumes that the current is limited by Josephson weak links (SNS or SIS Josephson junctions or microbridges) whose locations are to be determined. Each weak link is assumed to be described by an Airy current-field pattern rather than a Fraunhofer pattern. The former has a better

theoretical foundation and is in better agreement with the data, varying as $H^{-3/2}$ upon averaging. The fitting procedure yields the average cross sectional area of the weak links. By assuming the link thickness to be twice the London penetration depth at 77 K, we find that the average linear dimensions of the links are in all cases comparable to the grain dimensions. The quantitative analysis also confirms the percolation concept, in which a subset of weakest links controls the transport current.

MODELING OF CRITICAL CURRENTS IN GRANULAR HIGH-T_c SUPERCONDUCTORS, R.L. Peterson and J.W. Ekin, Proceedings of High T_c Superconductors: Magnetic Interactions of High T_c Superconductors, Gaithersburg, MD, October 11-13, 1988, Progress in High Temperature Superconductivity, Vol. 17, pp. 190-195 (1989). [CEEE/Electromagnetic Technology Division]

The transport critical current density of several samples of bulk sintered high-T_c superconductors was measured at very low magnetic fields and fitted to a model which assumes that the impediments to current at such fields are Josephson weak links. A sample of particular interest was $Y_1Ba_2Cu_3O_x$ made from hydroxycarbonate precursors; the final bulk sintered sample was very fine-grained, having an average grain size of about 1.8 μm as determined by a linear intercept analysis. The fit to the model is excellent if the average linear dimension of the weak links is chosen to be 2.0 μm. We conclude that this sample as well as others has Josephson weak links at its grain boundaries, and that any intragrain defects which may be responsible for flux pinning are not the primary weak links limiting the transport J_c of bulk samples at very low magnetic fields.

CURRENT RIPPLE EFFECT ON SUPERCONDUCTIVE DC CRITICAL CURRENT MEASUREMENTS, L.F. Goodrich and S.L. Bray, Cryogenics, Vol. 28, pp. 737-743 (1988). [CEEE/Electromagnetic Technology Division]

The effect of current ripple or noise on d.c. critical current measurements was systematically studied. Measurements were made on multifilamentary NbTi superconductor. A low-noise, battery-powered current supply was required in this study in order to make the pure d.c. critical current measurements. Also, an electronic circuit that stimulates a superconductor's general current-voltage characteristic was developed and used as an analysis tool. In order to make critical current measurements in which current ripple was present, the battery supply was modified to allow the introduction of controlled amounts of a.c. ripple. In general, ripple in a current supply becomes more significant in current supplies rated above 500 A because effective filtering is difficult. The effect of current ripple is a reduction in the measured d.c. critical current; however, ripple of sufficient amplitude can result in arbitrary measurement results. The results of this work are general and quantitatively applicable to the evaluation of critical current data and measurement systems. An unexpected benefit of this work was a more precise method for general critical current data acquisition. Problems common to all large-conductor critical-current measurements are discussed.

VAMAS INTERCOMPARISON OF CRITICAL CURRENT MEASUREMENT IN Nb_3Sn WIRES, K. Tachikawa,* K. Itoh,† H. Wada,† D. Gould,‡ H. Jones,§ C.R. Walters,‖ L.F. Goodrich, J.W. Ekin,

and S.L. Bray, IEEE Transactions on Magnetics, Vol. 25, No. 2, pp. 2368-2369 (March 1989). [CEEE/Electromagnetic Technology Division]

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The VAMAS technical working party in the area of superconducting and cryogenic structural materials has recently carried out the first world-wide intercomparison of critical current, I_c, measurement on multifilamentary Nb_3Sn wires. Three sample wires were supplied from each of EC (European Communities), Japan and USA. The total number of participant labs were 24 (EC 11, Japan 8 and USA 5). There were few restrictions for the I_c measurement at participant labs. The standard deviations of the I_c values reported from these labs varied among test samples, and were 6-21% of averaged I_c's at 12 Tesla.

INTEGRITY TESTS FOR HIGH-T_c AND CONVENTIONAL CRITICAL-CURRENT MEASUREMENT SYSTEMS, L.F. Goodrich and S.L. Bray, Advances in Cryogenic Engineering Materials, Vol. 36, Part A, pp. 43-50 (1989). [CEEE/Electromagnetic Technology Division]

Critical-current measurement systems must be extremely sensitive to the small differential voltage that is present across the test specimen as it changes from the zero resistance state to the flux-flow resistance state. Consequently, these measurement systems are also sensitive to interfering voltages. Specific methods for testing the sensitivity of critical-current measurement systems and for detecting the presence of interfering voltages are discussed. These include a simple procedure that simulates the zero resistance state and the use of an electronic circuit that simulates the flux-flow resistance state.

THERMAL CONTRACTION OF FIBERGLASS-EPOXY SAMPLE HOLDERS USED FOR Nb_3Sn CRITICAL-CURRENT MEASUREMENTS, L.F. Goodrich, S.L. Bray, and T.C. Stauffer, Advances in Cryogenic Engineering Materials, Vol. 36A, pp. 117-124 (1989). [CEEE/Electromagnetic Technology Division]

It is typical for Nb_3Sn-Cu superconductor specimens to be wound into coils on tubular specimen holders for critical-current measurements. If the thermal contraction of the holder is different than that of the specimen, axial strain may be applied to the specimen upon cooling from room to liquid-helium temperature. This strain can affect the measured critical current. The thermal contraction was measured for three different Nb_3Sn-Cu superconductors. Also, the thermal contraction was measured for several different specimen holders, all of which were made from fiberglass-epoxy composites. The specimen holder measurements were made using an electrical-resistance strain-gage technique, and they were confirmed by direct mechanical measurements. The tubes varied in diameter, wall thickness, and fabrication technique. Some of the tubes were made directly from tube stock, an others were machined from plate stock. The results of these measurements show that the thermal contraction of tube stock is strongly dependent on the ratio of its wall thickness to its radius, while the contraction of tubes machined from plate stock is relatively independent of these dimensions. Critical-current measurements of Nb_3Sn-Cu

specimens mounted on these various holders show that the presence of differential thermal contraction between the specimen and its holder can significantly affect the measured critical current.

S-N-S BEHAVIOR OF GRAIN BOUNDARIES IN POLYCRYSTALLINE $La_{1.85}Sr_{0.15}CuO_{4-y}$, A. Roshko, J.S. Moodera,* Y.-M. Chiang,* to appear in the Proceedings of the International M^2S-HTSC Conference, Stanford, California, July 1989, Physica C, Vol. 162-164, pp. 1625-1626 (1989). [CEEE/Electromagnetic Technology Division]

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The transport critical current density in polycrystalline $La_{1.85}Sr_{0.15}CuO_{4-y}$ samples in which the microstructure has been well characterized and with densities greater than 95% of theoretical, has been measured in fields from 4 x 10^{-5} to 15 T, at temperatures varied from 13 K to just below T_c. The dependence of critical current on temperature and magnetic field in zero and low fields, close to T_c, is consistent with S-N-S behavior, as expected from analyses of grain boundary composition.

DOUBLE-STEP BEHAVIOR OF CRITICAL CURRENT VS. MAGNETIC FIELD IN Y-, Bi- AND Tl-BASED BULK HIGH-T_c SUPERCONDUCTORS, J.W. Ekin, T.M. Larson, A.M. Hermann,* Z.Z. Sheng,* K. Togano,† and H. Kumakura,† Physica C 160, pp. 489-496 (1989). [CEEE/Electromagnetic Technology Division]

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A double-step characteristic is observed at 76 K in the transport critical current as a function of magnetic field (10^{-4} T to 10 T) in bulk sintered Y-, Bi- and Tl-based high-T_c superconducting materials. The low-field, step-like drop in the critical current density J_c commences at magnetic fields B between about 0.3 and 2 mT. This is followed by a plateau region of relatively constant critical current extending from about 30 to 300 mT, and then a second drop at fields between about 0.3 and 10 T. These features occur for all three superconductor materials and are interpreted respectively as a self-field/weak-link regime, a remnant percolation path regime and a flux-flow/upper-critical-field regime. The sharpness of the transition of the voltage-current (V-I) characteristic, represented by the transition parameter n (i.e., $V\chi I^n$), has a similar double-step shape as a function of magnetic field directly corresponding to the features of the $J_c(B)$ characteristic.

ELECTROMECHANICAL PROPERTIES OF SUPERCONDUCTORS FOR HIGH-ENERGY PHYSICS APPLICATIONS, Part II, J.W. Ekin, L.F. Goodrich, S.L. Bray, N.F. Bergren, R.B. Goldfarb, National Institute of Standards and Technology, NISTIR 89-3912, 154 p. (November 1989). [CEEE/Electromagnetic Technology Division]

This report presents data on superconductor performance under mechanical load. The data are needed for setting mechanical design constraints and measuring the electromechanical performance of NbTi superconductors for DOE high-energy physics magnet applications. The effect of axial tensile stress, applied at room temperature, on the critical current of NbTi

superconductor strands has been measured. The data show a simple result that the effect on the critical current is independent of the temperature at which the stress is applied; this allows the existing 4 K data base to be used to determine critical current degradation from room temperature fabrication stress, from cool-down stress introduced by differential contraction, and from 4 K stress generated by the Lorentz force when the magnet is energized. A study of the critical-current variations along NbTi strands extracted from a Rutherford cable has been made also. The results show that the principal mechanical degradation is extremely localized at the regions where the NbTi strand is bent around the edge of the cable. For example, only 3% of the total strand length can contribute 92% of the total strand voltage. A further study has been made of the effects of bending strain on the critical current of NbTi conductors. The degradation of the critical current from bending strain is much greater at low values of electric field than at high, suggesting that irregularity of the filament cross-sectional area introduced by bending may be the source of the I_c degradation. The bend tolerance of an NbTi conductor can be enhanced by increasing the local copper-to-superconductor area ratio. Measurements of the permeability, saturation magnetization, and intrinsic coercivity of several high-permeability steel alloys were made. The overall differences of saturation magnetization and intrinsic coercivity between cold-rolled steel samples were not significant.

NOVEL PROCEDURE FOR MAPPING THE J_c-H_{c2}-T_c SURFACE AND ITS APPLICATION TO HIGH TEMPERATURE SUPERCONDUCTORS, John Moreland, Y.K. Li,* L.F. Goodrich, A. Roshko, and R.H. Ono, Proceedings of the Conference on the Science and Technology of Thin-Film Superconductors, April 30-May 4, 1990, Denver, CO, Science and Technology of Thin-Film Superconductors 2, R.D. McConnell and R. Noufi, editors, New York: Plenum Press, pp. 429-438 (1990). [CEEE/Electromagnetic Technology Division]

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We have used an ac lock-in method for measuring the dynamic resistance versus current (dV/dI-I) curves to determine $J_c(T,H)$. The sample current consisted of a small constant oscillating current added to a variable dc current. The dc current was either slowly ramped for dV/dI-I measurements or controlled keeping the dV/dI-I constant while ramping temperature or field. In this way, it was possible to measure $J_c(T)$ at constant H. The temperature was controlled between 4 and 300 K using a bathysphere cryostat. The bathysphere cryostat was inserted into a high field magnet for measurements at fields up to 7 T. We have measured several high temperature superconductors including $Y_1Ba_2Cu_3O_{7-\delta}$ thin films. In addition, we have measured the dV/dI-I curve of a simulator with a V-I curve having the form $V = 10\ \mu V \times (I/1.4\ A)^{13}$. Upon numerical integration, our data for the simulator are consistent with those obtained by a dc method using an analog dc nanovoltmeter to measure the V-I curves directly.

HIGH T_c SUPERCONDUCTORS AND CRITICAL CURRENT MEASUREMENT, L.F. Goodrich and S.L. Bray, Cryogenics, Vol. 30, pp. 667-678 (August 1990). [CEEE/Electromagnetic Technology Division]

With the introduction of high T_c superconductors, a number of problems associated with critical current, I_c, measurement have arisen. The existing I_c measurement practices were developed and proved for low T_c superconductors. There are substantial differences between the two classes of materials. When the I_c concept was casually extended to the high T_c conductors, measurement

inconsistency, ambiguity and, in some cases, invalidity followed. A discussion of the underlying philosophy of I_c measurement is presented and a number of measurement variables that can influence the measured I_c are discussed. Many of the problems stem from inadequate reporting practices, and recommendations are given for improving measurement reports.

DEVELOPMENT OF STANDARDS FOR SUPERCONDUCTORS, FINAL REPORT, JANUARY 1988-DECEMBER 1989, L.F. Goodrich, R.B. Goldfarb, S.L. Bray, National Institute of Standards and Technology, NISTIR 90-3935, 148 p. (January 1990). [CEEE/Electromagnetic Technology Division]

A cooperative program with the Department of Energy, the National Institute of Standards and Technology, other national laboratories, and private industry is in progress to develop standard measurement practices for use in large scale applications of superconductivity. This report describes research for the period January 1988 through December 1989. It contains the results of critical current studies on a large conductor Reference Material (RM), the effect of power-supply current ripple, the measurements of large conductors, and an interlaboratory comparison (round robin) of Nb_3Sn wires. Short-range variations in magnetic hysteresis loss in multifilamentary Nb_3Sn were studied. The results of participation in several interlaboratory comparisons are described.

CURRENT SUPPLY FOR HIGH-T_c SUPERCONDUCTOR TESTING, S.L. Bray and L.F. Goodrich, Measurement Science Technology, Vol. 1, pp. 491-494 (1990). [CEEE/Electromagnetic Technology Division]

Precise and accurate measurements of the DC critical current of high-T_c superconductors often require a current supply that has high input-to-output isolation, high isolation from ground and low output ripple. Also, to ensure precise current control, the supply should have low current drift and its input-output characteristic should be linear. A design for a simple and inexpensive current supply that has these characteristics is presented. The primary power source is a 12 C wet-cell battery and the typical operating range is from 10 mA to 10 A. The supply's current ripple is <0.4 mA RMS over this operating range. At an output current of 5 A the current drift is $<2\%$ h^{-1} without a warm-up period. The maximum variation of the supply's output from linearity, over its full operating range, is $<5\%$.

TRANSPORT CRITICAL CURRENT OF ALIGNED POLYCRYSTALLINE $Y_1Ba_2Cu_3O_{7-\delta}$ AND EVIDENCE FOR A NONWEAK-LINKED COMPONENT OF INTERGRANULAR CURRENT CONDUCTION, J.W. Ekin, H.R. Hart, Jr.,* and A.R. Gaddipati,* Journal of Applied Physics, Vol. 68, No. 5, pp. 2285-2295 (September 1, 1990). [CEEE/Electromagnetic Technology Division]
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A study of grain alignment and its effect on the dc transport critical current in fine-grained bulk $Y_1Ba_2Cu_3O_{7-\delta}$ is reported in magnetic fields from 10^{-4} T to 26 T. Two features distinguish the critical current density J_c of aligned bulk $Y_1Ba_2Cu_3O_{7-\delta}$ from unaligned material. First, the effective critical field where the intergranular J_c approaches zero is about four times higher (30 T) for aligned samples with field parallel to the *a*, *b* planes, than it is for polycrystalline unaligned samples (7 T). Second, the nearly field independent plateau value of J_c between 10 mT and 1 T

is one to two orders of magnitude higher than typical plateau values of J_c in unaligned bulk-sintered $Y_1Ba_2Cu_3O_{7-\delta}$, for field parallel to the *a*, *b* planes. A low-field (< 10 mT) weak-link decrease in J_c with magnetic field is still observed, but it is much smaller than for unaligned material. These data clearly demonstrate that alignment alone significantly reduces the *weak-link* problem in fine-grained polycrystalline samples with low-aspect-ratio (4:1) grains (unlike melt-grown samples where there has been some ambiguity as to the relative importance of alignment versus large grain growth). Furthermore, the results provide strong evidence that there are two parallel components of *intergranular* current conduction, one consisting of weak-linked material, the other behaving like intrinsic intragranular material that is not weak-linked. A comparison with unaligned $Y_1Ba_2Cu_3O_{7-\delta}$ indicates that the volume fraction of such nonweak-linked material is significantly enhanced by grain alignment, but still only 0.01%-0.1% of the grain boundary area. Field-cooled and force-free, J_c data are also presented, along with detailed measurements of the shapes of the voltage-current characteristics.

SWITCHING IN HIGH-T_c SUPERCONDUCTOR CURRENT TRANSPORT MEASUREMENTS, L.F. Goodrich, John Moreland, and A. Roshko, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 1194-1197 (March 1991). [CEEE/Electromagnetic Technology Division]

Switching voltages can occur in four-wire current transport measurements of sintered high-T_c superconductors. These switching voltages are irreversible shifts in the voltage-current characteristic of the superconductor that result in multiple branches. The voltage along these branches can be very nonlinear as a function of current and can be positive or negative in polarity relative to the current direction. These voltages can interfere with the correct determination of resistivity and critical current density. Experimental data on unaligned sintered $Y_1Ba_2Cu_3O_{7-\delta}$ which illustrate the complex nature of the voltages and the confusion they can create are presented. Models based on weak links and H_{c1}, and on other effects, are discussed as are observations on NbTi and Nb_3Sn based superconductors.

SOFTWARE TECHNIQUES TO IMPROVE DATA RELIABILITY IN SUPERCONDUCTOR AND LOW-RESISTANCE MEASUREMENTS, L.F. Goodrich and A.N. Srivastava, NIST Journal of Research, Vol 95, No. 5, pp. 575-589 (September-October 1990). [CEEE/Electromagnetic Technology Division]

Software techniques have been developed to take low-amplitude data in various patterns, assign a figure of merit to a set of data readings, edit data for erroneous readings (or other experimental variations), and to alert the experimenter if the detected errors are beyond the scope of the software. Erroneous voltage readings from digital voltmeters, intermittent electrical connections, and an array of similar variations in data have been detected through the use of a data editor. The fixed limit data editor removes readings that are inconsistent with the distribution of the majority of the data readings. The frequency of erroneous readings from a particular digital voltmeter ranges from 1 error per 100 000 readings to 1 error per 100 readings. The magnitude of the error can be as large as 3 percent of full scale with a zero volt input to the voltmeter. It may be necessary to have multiple meters measuring voltages in the same circuit in order to generate these erroneous readings. A systematic study was performed on the occurrence of the internally generated erroneous voltmeter readings, and it was determined that the amount that a

reading was in error scaled with one of a few parameters. The software techniques described here have been used in a variety of measurements, such as resistance-versus-temperature measurements made on cryoconductors or superconductors, and voltage-versus-current measurements made on superconductors to determine the critical current.

CRITICAL-CURRENT DIFFRACTION PATTERNS OF GRAIN-BOUNDARY JOSEPHSON WEAK LINKS, R.L. Peterson and J.W. Ekin, Physical Review B, Vol. 42, No. 13, pp. 8014-8018 (November 1, 1990). [CEEE/Electromagnetic Technology Division]

We discuss the diffraction patterns and other characteristics of the critical current as a function of magnetic field in grain-boundary Josephson barriers. Diffraction patterns occur not just for SIS junctions but for all types of Josephson links, including SNS junctions, which may be present at grain boundaries in high-T_c superconductors. We discuss the generality of the Airy diffraction pattern, which is expected to characterize grain-boundary barriers in bulk material more accurately than the Fraunhofer pattern. The transport critical-current density in many bulk, granular high-T_c superconductors has a power-law dependence on very low magnetic fields, characteristic of averaged diffraction patterns, and cannot be fitted by an exponential magnetic-field dependence, which may result from the material properties of the barriers.

ENHANCED FLUX CREEP IN Nb-Ti SUPERCONDUCTORS AFTER AN INCREASE IN TEMPERATURE, R.W. Cross and R.B. Goldfarb, Applied Physics Letters, Vol. 58, No. 4, pp. 415-416 (January 28, 1991). [CEEE/Electromagnetic Technology Division]

The magnetic fields of Superconducting Super Collider (SSC) dipole magnets change with time when the magnets are operated at constant current. The decay of the field is though to be a consequence of flux creep in the Nb-Ti filaments in the superconducting cables. However, measured magnetic relaxation of small samples of SSC cable as a function of time is unlike the large decays that are observed in the fields of the actual magnets. We have made relaxation measurements on sample SSC conductors at 3.5 and 4.0 K after field cycling. The decay at both temperatures was 2.8% in 50 min. However, the relaxation measured after a temperature increase from 3.5 to 4.0 K was 4.8% in 50 min. A likely reason for the greater magnetization decay is that, after an increase in temperature, the Nb-Ti is in a supercritical state, with shielding currents flowing at a density greater than the new critical current density. This causes enhanced flux creep. We suggest that a small temperature rise during the operation of SSC magnets may contribute to the unexpectedly large magnetic field decay.

EFFECT OF MECHANICAL DEFORMATION ON Nb-Ti FILAMENT PROXIMITY-EFFECT COUPLING AT THE EDGES OF SSC CABLES, T.W. Petersen and R.B. Goldfarb, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 1809-1810 (March 1991). [CEEE/Electromagnetic Technology Division]

Magnetization as a function of transverse magnetic field and time was measured for short strands extracted from the centers and edges of five Nb-Ti Rutherford cables designed for use in Superconducting Super Collider dipole magnets. The multifilamentary strands all had 6-μm diameter filaments. Edge samples, which had severe mechanical deformation, showed small magnetic coupling losses at low fields, compared to no coupling losses for undeformed center strands. Sharp strand bends at cable edges decreases the interfilament spacing to the order of the coherence length in the normal matrix material which increases the effective filament diameter and hysteresis loss at low fields. Microscopic studies of the cables' cross sections confirmed smaller interfilament separations in these samples. Flux creep measurements, represented by the time dependence of magnetization, showed little difference between edge and center samples. This indicates that the proximity-coupled matrix in edge samples is not a significant source of flux creep.

CURRENT RECORD IN SUPERCONDUCTORS, J.W. Ekin, K. Salama,* and V. Selvamanickam,* Nature, Vol. 350, p. 26 (March 7, 1991). [CEEE/Electromagnetic Technology Division]
*University of Houston, Texas

Critical currents in excess of 200 A at 30 T have been measured at 4.2 K in melt grown $YBa_2Cu_3O_7$. Critical currents of about 10 A were obtained at 30 T at 77 K. The results demonstrate the possibility of obtaining high supercurrents at both liquid helium and liquid nitrogen temperatures for applications at magnetic fields of 30 T or more.

WEAK-LINK-FREE BEHAVIOUR OF HIGH-ANGLE $YBa_2Cu_3O_{7-\delta}$ GRAIN BOUNDARIES IN HIGH MAGNETIC FIELDS, S.E. Babcock,* X.Y. Cai,* D.L. Kaiser, and D.C. Larbalestier, Nature, Vol. 347, No. 6289, pp. 167-169 (September 13, 1990). [IMSE/Metallurgy Division]
*University of Wisconsin

A crucial issue in both the physics and the application of high-temperature superconductors is the low transport critical current density (J_{ct}) of polycrystalline materials. Much thinking about this issue has been defined by the thin-film $YBa_2Cu_3O_{7-\delta}$ bicrystal experiments of Dimos et al., which clearly showed greatly reduced values of J_{ct} at high-angle grain boundaries (mis-orientation angle $\geq 10°$). The reproduciblity of this result for a wide range of misorientation relationships led them to conclude that all high-angle grain boundaries act intrinsically as Josephson junctions. By contrast, we describe here two high-angle grain boundaries with superconducting properties that clearly lack the weak-link signatures characteristic of Josephson junctions. Our bulk-scale bicrystal results provide direct evidence that not all high-angle grain boundaries are alike in their superconducting properties, and that at least some high-angle boundaries can carry significant supercurrents at 77 K in high magnetic fields.

CRITICAL CURRENT BEHAVIOR OF Ag-COATED $YBa_2Cu_3O_{7-x}$, R.H. Ono, J.A. Beall, T. Harvey, C. Reintsema, M. Johansson, M. Cromar, L.F. Goodrich, J. Moreland, A. Roshko, and T.C. Stauffer, IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 1471-1474 (March 1991). [EEEL/Electromagnetic Technology Division]

We have studied the behavior of high-quality $YBa_2Cu_3O_{7-x}$ (YBCO) thin films with Ag overlayers. We chose to study Ag in detail because of its widespread use as contact metalization and our earlier studies of proximity effects in YBCO. The details of transport critical current measurements are presented. The Ag coatings can reduce normal state resistance while not degrading the critical current density, J_c.

3. CRYSTAL STRUCTURE

X-RAY STUDIES OF HELIUM-QUENCHED $Ba_2YCu_3O_{7-x}$, Winnie Wong-Ng and Lawrence P. Cook, Advanced Ceramic Materials: Ceramic Superconductors, Vol. 2, No. 3B, pp. 624-631 (July 15, 1987). [IMSE/Ceramics Division]

A series of 11 samples of $Ba_2YCu_3O_{7-x}$ ($x \simeq 0$ to 1) was prepared from single phase orthorhombic material by annealing at temperatures from 400 to 1000°C, followed by rapid quenching. All quenchings were performed using a liquid nitrogen-cooled copper cold well through which helium gas was passed. Lattice parameters were obtained for these materials by X-ray powder diffraction methods to obtain dependence of cell volumes and crystal symmetry on annealing temperature.

CRYSTAL STRUCTURE OF THE HIGH-TEMPERATURE SUPERCONDUCTOR $La_{1.85}Sr_{0.15}CuO_4$ ABOVE AND BELOW T_c, R.J. Cava,* A. Santoro, D.W. Johnson, Jr.,* and W.W. Rhodes,* Physical Review B, Vol. 35, No. 13, pp. 6716-6720 (May 1, 1987). [IMSE/Reactor Radiation Division]
*AT&T Bell Laboratories, Murray Hill

The crystal structure of $La_{1.85}Sr_{0.15}CuO_4$ has been determined at 300, 60, and 10 K by neutron-diffraction powder profile analysis. The structure is of the tetragonal K_2NiF_4 type at ambient temperature, but undergoes an orthorhombic distortion near 200 K which buckles the copper-oxygen planes. The crystal structure of the orthorhombic phase is not significantly different above and below T_c. The copper-oxygen coordination polyhedron changes shape in a subtle manner at the tetragonal-to-orthorhombic phase transition, but is unchanged at T_c.

NEUTRON STUDY OF THE CRYSTAL STRUCTURE AND VACANCY DISTRIBUTION IN THE SUPERCONDUCTOR $Ba_2YCu_3O_{9-\delta}$, F. Beech, S. Miraglia, A. Santoro, and R.S. Roth, Physical Review B, Vol. 35, No. 16, pp. 8778-8781 (June 1, 1987). [IMSE/Reactor Radiation Division]

Two samples of the high-temperature superconductor $Ba_2YCu_3O_{9-\delta}$ with $\delta = 2.0$ and 2.2 have been studied at room temperature and at 10 K, with the neutron powder diffraction method and profile analysis. The structure of the compound is orthorhombic. The oxygen atoms are located on four sets of sites and not on five sets as reported earlier in x-ray-diffraction studies. Of the two sets of copper atoms, one is surrounded by four oxygen atoms at distances 1.9299(4) and 1.9607(4) Å. This arrangement forms a two-dimensional framework of Cu-O atoms that extends indefinitely along the *a* and *b* axis. A fifth oxygen atom is located at 2.295(3) Å from the copper atom, giving a pyramidal configuration. In the other set, the Cu atoms are also surrounded by four oxygen atoms, with distances 1.9429(1) and 1.846(2) Å. In this case, however, the oxygen atoms form rectangles connected by vertices, and resulting in chains along the *b* axis. In the compound with $\delta = 2.0$ all oxygen sites are fully occupied. When $\delta = 2.2$ there are oxygen vacancies, but these are confined to one set of positions only, specifically to the oxygen atoms of the chains, located on the *b* axis. No detectable change of the structure has been observed between room temperature and low temperature.

THE STRUCTURE AND PROPERTIES OF $Ba_2YCu_3O_6$, A. Santoro, S. Miraglia, and F. Beech, S.A. Sunshine,* D.W. Murphy,* L.F. Schneemeyer,* and J.V. Waszczak,* Materials Research Bulletin, Vol. 22, pp. 1007-1113 (1987). [IMSE/Reactor Radiation Division]
*AT&T Bell Laboratories, Murray Hill

The structure of $Ba_2YCu_3O_6$ has been determined by neutron diffraction powder profile analysis. The cell is tetragonal P4/mmm with a = 3.8570(1) Å and c = 11.8194(3) Å. The cations are in a perovskite type arrangement, with Ba and Y ordered on the A site to give a cell tripled along c. The oxygens occupy only 2/3 of the perovskite anion sites and are ordered such that 1/3 of the Cu is two-fold coordinated and 2/3 is five-fold coordinated. This configuration can be derived from that of the superconductor $Ba_2YCu_3O_7$ by removing the oxygen atoms along the b axis at the (0,1/2,0) position. A pressed pellet exhibited semiconductivity with a band gap of 0.21 eV from 125-300 K. The compound is described as $Ba_2YCu^{1+}Cu_2^{2+}O_6$.

X-RAY POWDER STUDY OF 2BaO:CuO, W. Wong-Ng, K.L. Davis, and R.S. Roth, Journal of the American Ceramic Society, Vol. 71, No. 2, pp. C-64 to C-67 (1987). [IMSE/Ceramics Division]

A compound of composition 2BaO:CuO was synthesized during the phase equilibria study of the $BaO-Y_2O_3-CuO_x$ system. Phase characterization has been carried out by using X-ray powder diffraction. The crystal symmetry was found to be the same as that of Ca_2CuO_3 and Sr_2CuO_3. It is orthorhombic with space group Immm and lattice parameters a = 12.9655(14), b = 4.1007(3), c = 3.9069(5) Å, V = 207.72(3) Å. The experimental pattern shows good agreement, in general, with intensity values calculated by assuming Ba_2CuO_3 to have a structure similar to that of Sr_2CuO_3 and Ca_2CuO_3. Intensity discrepancy for the h00 reflections might be due to preferred orientation.

NEUTRON POWDER DIFFRACTION STUDY OF ORTHORHOMBIC $Ba_2YCu_3O_{6.5}$, S. Miraglia, F. Beech, A. Santoro, D. Tran Qui, S.A. Sunshine,* and D.W. Murphy,* Materials Research Bulletin, Research Bulletin, Vol. 22, pp. 1733-1740 (December 1987). [IMSE/Reactor Radiation Division]
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The structure of orthorhombic $Ba_2YCu_3O_{6.5}$ has been refined with neutron diffraction profile analysis. The space group of the compound is Pmmm, and the lattice parameters are a = 3.8468(1), b = 3.8747(1), and c = 11.7466(5) Å. The oxygen sites at (0,0,z), 1/2,0,z), and (0,1/2,z) are fully occupied, while the sites at (0,1/2,0) are 50% full and the sites at (1/2,0,0) are vacant. A comparison of bond distances in this sample and in other compositions in the system $Ba_2YCu_3O_\delta$ ($6.0 \leq \delta \leq 7.0$) shows that the environment of the Ba atoms and of the Cu atoms at (0,0,0) and (0,0,z) change significantly with the amount of oxygen in the unit cell.

THE X-RAY POWDER DIFFRACTION PROFILE STUDIES ON $YBa_2Cu_3O_{7.0}$ AND $YBa_2Cu_3O_{6.8}$, Y. Zhang,* W. Wong-Ng, B. Morosin,† C.R. Hubbard, J.M. Stewart,* and S.W. Freiman, Physica C, Vol. 152, pp. 130-132 (1988). [IMSE/Ceramics Division]
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†Sandia National Laboratories

Two barium yttrium cuprate compositions having different oxygen stoichiometries, $YBa_2Cu_3O_{7.0}$ and $YBa_2Cu_3O_{6.8}$, were prepared from a single phase material under different processing conditions. A comparison of the X-ray powder diffraction (XRD) patterns of these two samples showed a significant difference in diffraction line breadth. X-ray diffraction line profile analysis has been employed to study the profile differences.

POSSIBLE EVIDENCE FOR SUPERCONDUCTING LAYERS IN SINGLE CRYSTAL $Y_Ba_2Cu_3O_7$-x BY FIELD ION MICROSCOPY, A.J. Melmed, R.D. Shull, C.K. Chiang, and H.A. Fowler, Science, Vol. 239, pp. 176-178 (January 8, 1988). [IMSE/Ceramics Division and Metallurgy Division]

The high-transition-temperature superconducting ceramic material $YBa_2Cu_3O_{7-x}$ ($0 < x < 0.5$) has been examined by field ion microscopy. Specimens from nominally superconducting and nonsuperconducting samples (determined by magnetic susceptibility measurements) were studied by field ion microscopy and significant differences were found. Preferential imaging of atomic or molecular layers, due to preferential field evaporation, field ionization, or both, was found in the superconducting phase below the transition temperature and is interpreted as possible evidence for the occurrence of relatively highly conducting layers in the $YBa_2Cu_3O_{7-x}$ unit cell perpendicular to the orthorhombic c-axis. Similar results were obtained for $YbBa_2Cu_3O_{7-x}$.

SIGNIFICANCE OF PLANE VERSUS CHAIN SITES IN HIGH-TEMPERATURE OXIDE SUPERCONDUCTORS, Gang Xiao,* M.Z. Cieplak,* D. Musser,* A. Gavrin,* F.H. Streitz,* C.L. Chien,* J.J. Rhyne, and J.A. Gotaas, Nature, Vol. 332, No. 6161, pp. 238-240 (March 17, 1988). [IMSE/Reactor Radiation Division]
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One of the outstanding questions concerning the high-temperature superconductor $YBa_2Cu_3O_7$ is the relative importance of the CuO_2 planes (Cu(2) site) and the CuO chains (Cu(1) site). Many theories have stressed the importance of the 3*d* holes at the Cu sites, which provide an antiferromagnetic correlated background necessary for the coupling of the superconducting electrons. We have substituted Zn($3d^{10}4s^2$) and Ga($3d^{10}4s^24p^1$) in the Cu(2) and Cu(1) sites, respectively, while maintaining the oxygen stoichiometry at 7. In the valence states of 2+ and 3+ respectively, the 3*d* bands of both elements are completely full, without the complication of a magnetic moment. Here we discuss the structural changes that accompany Zn and Ga substitution, as deduced from X-ray and neutron diffraction, and correlate these changes with the superconducting properties. We show that the integrity of the planes is much more important than that of the chains in sustaining high-T_c superconductivity.

STRUCTURAL PHASE TRANSITIONS OF $Ba_2RCu_3O_{6+x}$ SUPERCONDUCTORS, W. Wong-Ng, L.P. Cook, C.K. Chiang, L.J. Swartzendruber, and L.H. Bennett, Adv. Cer. Materials, pp. 27-42 (May 1988). [IMSE/Ceramics and Metallurgy Divisions]

In order to gain more insight into the effect of oxygen stoichiometry on superconductivity and the correlation between the size of the rare-earth elements and superconductivity, the phase transitions between the orthorhombic and tetragonal structures of several high T_c superconductors

$Ba_2RCu_3O_{6+x}$ (where R = Sm, Y, Gd and Er; x = 0 to 1) have been investigated as a function of oxygen content achieved by quenching in air.

Various measurements including x-ray diffraction, thermogravimetric analysis, and determination of Meissner effects were carried out in order to correlate the nature of the phase transition with crystallographic data, superconductivity and annealing temperature.

Phase transformations from orthorhombic $Ba_2RCu_3O_7$ to tetragonal $Ba_2RCu_3O_6$ are apparently second order and may involve two orthorhombic regions which correspond to two T_c plateaus, region (A), T_c of 92 K with cell parameters $a < b \approx c/3$ and region (B), T_c of from 52 to 60 K with $a < b < c/3$. The transformations from orthorhombic to tetragonal for all four compounds takes place in a temperature range of 625 to 770°C. The temperature range of the region B plateaus also varies with different R. Both structural phase transition temperatures and characteristics of the T_c plateaus appear to follow a trend anticipated from the dependence of the ionic radius on the f electron count.

BISMUTH VALENCE ORDER-DISORDER STUDY IN $BaBiO_3$ BY POWDER NEUTRON DIFFRACTION, C. Chaillout, A. Santoro, J.P. Remeika,* A.S. Cooper,* G.P. Espinosa,* and M. Marezio,† Solid State Communications, Vol. 65, No. 11, pp. 1363-1369 (1988). [IMSE/Reactor Radiation Division]

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Two structural arrangements have been shown to exist for the perovskite-like compound $BaBi^{3+}_{0.5}Bi^{5+}_{0.5}$ by powder neutron diffraction data. The first is characterized by a partial (75%) order of the Bi^{3+} and Bi^{5+} cations on the two crystallographically independent sites, while in the second arrangement the two cations are almost 100% disordered. The structure of eleven differently-prepared $BaBiO_3$ samples showed that the deciding factor for obtaining one or the other arrangement is the temperature at which the sample is prepared or subsequently heat-treated. On the contrary, the cooling rate and the atmosphere (air or oxygen) do not seem to be important parameters. DTA measurements indicated that $BaBiO_3$ undergoes an additional phase transition at 860°C on heating and 801°C on cooling. This transition probably corresponds to the disproportionation of the Bi into Bi^{3+} and Bi^{5+} cations. This conjecture explains why the heat-treatment at 800°C yields compounds containing partial cation ordering. Since the symmetry is cubic above and below the 801°C transition, the 75% ordered $BaBiO_3$ stable between 801°C and 480°C would contain a trigonal dynamical distortion which would be the precursor of the static trigonal distortion which takes place at 480°C. In every compound the partial ordering is always 75%. This could be interpreted as an indication that an additional ordering of Au_3Cu type exists on the two Bi sublattices.

NEUTRON AND ELECTRON DIFFRACTION STUDY OF $YBa_2Cu2_2Cu1_{.77}Fe_{.23}O_{7.13}$, P. Bordet,* J.L. Hodeau,* P. Strobel,* M. Marezio,* and A. Santoro, Solid State Communications, Vol. 66, No. 4, pp. 435-439 (1988). [IMSE/Reactor Radiation Division]

*Laboratoire de Cristallographie du CNRS

The compound of formula $YB_2Cu_{2.7}Fe_{0.3}O_{7.13}$ has been analyzed by neutron and electron diffraction techniques. The material is tetragonal with lattice parameters a = b = 3.8674(1), c = 11.6687(2) Å and space group P4/mmm. The Fe cations substitute only the Cu cations located on the basal plane of the structure and can adopt three different types of coordination (tetrahedral, pyrimidal and octahedral) depending upon the content and distribution of the extra oxygen atoms on the plane. Calculations of the effective valence of iron cations seem to indicate that Fe^{3+} is present in tetrahedral coordination and Fe^{4+} in pyramidal and octahedral coordination, while values of $Cu^{2.2+}$ and $Cu^{2.47+}$ were found for the copper cations located at (00z) and (000), respectively. The electron diffraction experiments show diffuse scattering planes parallel to (110)* and $(1\bar{1}0)$*. Crosses of strong intensity are visible at reciprocal nodes located between the reciprocal lattice layers. This diffuse scattering is interpreted in terms of linear clusters of iron cations extending along the [110] and $[1\bar{1}0]$ directions having a width of a few cations. The clusters are separated by domains of orthorhombic $YBa_2Cu_3O_{6+x}$ having the same orientation or rotated of 90° one with respect to the other.

SUPERCONDUCTIVITY IN LANTHANUM BARIUM CUPRATE PEROVSKITES, S.A. Sunshine,* L.F. Schneemeyer,* J.V. Waszczak,* D.W. Murphy,* S. Miraglia, A. Santoro, and F. Beech, Journal of Crystal Growth, Vol. 85, pp. 632-638 (1987). [IMSE/Reactor Radiation Division]

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Samples in the system $Ba_{2-x}La_{1+x}Cu_3O_{7-\delta}$ ($0.0 \leq x \leq 0.5$; $0 < \delta < 0.25$) have been prepared and characterized by X-ray powder diffraction, magnetic susceptibility, electrical resistivity, and for x = 0.5 powder neutron diffraction. This neutron diffraction study on $Ba_{1.5}La_{1.5}Cu_3O_{7.25}$ indicates that the structure is similar to the 92 K superconductor $Ba_2YCu_3O_7$ with two exceptions. First, the "Ba site" contains 75% Ba and 25% La and second, the symmetry is tetragonal. At x = 0.5 the material is a semiconductor, but as x decreases metallic behavior and superconductivity are observed. Compositions with $x < 0.2$ contain traces of $BaCuO_2$, but exhibit $T_c^{onset} \approx 90$ K and R = O near 80 K. For x = 0.2-0.3, $T_c^{onset} \approx 60$ K and R = O near 50 K are observed. For $x \leq 0.1$ orthorhombic symmetry is observed. These results provide further evidence that oxygen stoichiometry and details of the oxygen ordering are important to superconductivity.

ANTIFERROMAGNETIC ORDER IN $YBa_2Cu_{3-x}Co_xO_{6+y}$, P.F. Miceli,* J.M. Tarascon,* L.H. Greene,* P. Barboux,* M. Giroud,* D.A. Neumann, J.J. Rhyne, L.F. Schneemeyer,† and J.V. Waszczak,† Physical Review B, Vol. 38, No. 13, pp. 9209-9212 (November 1, 1988). [IMSE/Reactor Radiation Division]

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We present a neutron scattering study of oxygen-deficient $YBa_2Cu_{3-x}Co_xO_{6+y}$ where, with increasing Co concentration, long-range antiferromagnetic order develops on the chain sites. For x = 0.2, two magnetic transitions are found: Below $T_{N2} = 211$ K, there is an ordered moment on both Cu sites, while for $T_{N2} < T < T_{N1} = 415$ K only a "plane" moment is present. For x = 0.8, an ordered moment appears on both Cu sites with a single transition at 435 K. Evidence for magnetic ordering was also found by magnetic susceptibility measurements. These

results imply that Co substitution enhances antiferromagnetic coupling between the "plane" and "chain" sites.

CRYSTAL CHEMISTRY OF SUPERCONDUCTORS: A GUIDE TO THE TAILORING OF NEW COMPOUNDS, A. Santoro, F. Beech, M. Marezio,* and R.J. Cava,* Physica C, Vol. 156, pp. 693-700 (1988). [IMSE/Reactor Radiation Division]
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The crystal structures of the known superconducting copper oxides can be described in terms of two basic structural types. The series $La_2Ca_{n-1}Cu_nO_{2n+2}$, $(Tl,Bi)_2(Ba,Sr)_2Ca_{n-1}Cu_nO_{2n+4}$ and $TlBa_2Ca_{n-1}Cu_nO_{2n+3}$ can be viewed as made of alternating slices having the rock salt and perovskite structure. The compounds $Ba_2YCu_4O_8$ and $Ba_4Y_2Cu_7O_{14+x}$, on the other hand, comprise of perovskite blocks alternating with blocks in which a crystallographic shear is present. The effect of this shear is that of forming double chains of edge sharing squares with oxygen atoms at the corners and copper atoms at the center. The superconductor $Ba_2YCu_3O_7$ can be described in terms of both structural types and may be considered as an intermediate type between the other two. The basic building blocks of these superconducting materials can be further broken down into constituent nets (or meshes). This description allows one to envisage new structures built from these meshes containing the key structural elements present in the currently known superconductors. As such, the structural schemes used in this description may be used as a guide in the preparation of new materials with interesting electronic properties.

NEUTRON POWDER DIFFRACTION STUDY OF $Pb_2Sr_2YCu_3O_8$, THE PROTOTYPE OF A NEW FAMILY OF SUPERCONDUCTORS, R.J. Cava,* M. Marezio,* J.J. Krajewski,* W.F. Peck, Jr.,* A. Santoro, and F. Beech, Physica C, Vol. 157, pp. 272-278 (1989). [IMSE/Reactor Radiation Division]
*AT&T Bell Laboratories, Murray Hill

The structure of $Pb_2Sr_2YCu_3O_8$ has been analyzed with powder neutron diffraction techniques and profile analysis. The results of this study confirm the general structural features determined by X-ray single-crystal methods. The material has pseudo tetragonal symmetry, but is orthorhombic, space group Cmmm with lattice parameters $a = 5.3933(2)$, $b = 5.4311(2)$, and $c = 15.7334(6)$ Å. The orthorhombic distortion is caused by the disordering in the a lane of the oxygen atoms of the PbO layers over the general position of the space group, with $x = 0.275(5)$ and $y = 0.402(5)$ Å. The structure of this compound can be derived from that of $YBa_2Cu_3O_6$ by substituting the Cu layers with blocks of ...PbO.Cu.PbO... . Of the two Cu atoms in the asymmetric unit, one has two fold coordination while the other has five fold pyramidal coordination with the apex elongated along the c-axis.

MAGNETIC TRANSITIONS IN THE SYSTEM $YBa_2Cu_{2.8}Co_{0.2}O_{6+y}$, P.F. Miceli,* J.M. Tarascon,* P. Barboux,* L.H. Greene,* B.G. Bagley,* G.W. Hull,* M. Giroud,* J.J. Rhyne, and D.A. Neumann, Physical Review B, Vol. 39, p. 12375 (1989). [IMSE/Reactor Radiation Division]
*Bell Communications Research

We have studied the oxygen dependence of the two magnetic transitions (antiferromagnetic ordering of chains and planes) in $YBa_2Cu_{2.8}Co_{0.2}O_{6+y}$ using neutron scattering. It is found that

both transition temperatures increase with decreasing oxygen concentration. At $y \approx 0.37$ ($\equiv y_0$) the two transition temperatures are equal, so that chains and planes order at a single transition temperature for $y \leq y_0$. For $y = 1$ the compound is superconducting at 60 K. Therefore, this system qualitatively exhibits the magnetic and superconducting properties of pure $YBa_2Cu_3O_{6+y}$ while providing an important insight on the oxygen dependence of chain site magnetic ordering. A discussion is presented which also includes results on Ni and Al substitutions.

STRUCTURE AND MAGNETIC PROPERTIES OF NONSUPERCONDUCTING DOPED Co AND Fe-$Bi_2Sr_2Cu_{1-x}M_xO_y$, J.M. Tarascon,* P.F. Miceli,* P. Barboux,* D.M. Hwang,* G.W. Hull,* M. Giroud,* L.H. Greene,* Yvon Le Page,† W.R. McKinnon,† E. Tselepis,† G. Pleizier,† M. Eibschutz,‡ D.A. Neumann, and J.J. Rhyne, Physical Review B, Vol. 39, p. 11587 (1989). [IMSE/Reactor Radiation Division]

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The structure and magnetic properties of the $Bi_2Sr_2Cu_{1-x}M_xO_y$ (M = Co and Fe) materials were studied. The limits of solid solution formation are at $x = 0.5$ for the Fe system and $x = 1$ for the Co system. Crystals of the new $Bi_2Sr_2CoO_y$ phase were grown and the structure established by x-ray crystallography. The subcell is the same as that of the 10 K superconductor, $Bi_2Sr_2CuO_y$, but the superstructure is different, as it exhibits a commensurate modulation of periodicity 4 instead of 5. Extra oxygen is accommodated in the Bi layers, as in $Bi_2Sr_3Fe_2O_y$, and the structure of the Bi-O layers can be described as 50% rocksalt-type and 50% oxygen-deficient perovskite for $x = 1$, but with disorder at the oxygen positions. The formal valence of Co in this compound is about 2.5 ± 0.2 as deduced from structural and chemical measurements, whereas Fe adopts the oxidation state +3 as deduced by Mössbauer measurements. $Bi_2Sr_2CuO_y$ is an antiferromagnetic insulator with the spins lying within the CoO_2 sheets and the antiferromagnetic ordering temperature (T_N) is sensitive to processing conditions and composition changes. The high anisotropy of the susceptibility suggests that $Bi_2Sr_2CuO_y$ may be an Ising or xy antiferromagnet.

CRYSTALLOGRAPHY, Anthony Santoro, Chapter 4 in High T_c Superconductivity, New York: Springer-Verlag, pp. 84-121 (1990). [IMSE/Reactor Radiation Division]

The structures of the superconducting compounds belonging to the systems $BaPb_{1-x}Bi_xO_3$, $La_{2-x}M_xCuO_{4-y}$, $Ba_2YCu_3O_x$, $Ba_{2-x}La_{1+x}Cu_3O_x$, and $Bi_2CaSr_2Cu_2O_x$ are described in detail in this chapter, and their relationship with the structure of perovskite is illustrated from a structural point of view. The role of neutron diffraction to locate light atom in the presence of heavy metals is underlined, and a brief description of the Rietveld method is given. Compounds with crystallographic shear, such as $Ba_2YCu_4O_8$ and $Ba_4Y_2Cu_7O_{14+x}$, are briefly described at the end of the chapter.

X-RAY DIFFRACTION STUDY OF A THERMOMECHANICALLY DETWINNED SINGLE CRYSTAL OF $YBa_2Cu_3O_{6+x}$, Winnie Wong-Ng, Frank W. Gayle, Debra L. Kaiser, Steven F. Watkins,*

and Frank R. Fronczek,* Physical Review B, Vol. 41, No. 7, pp. 4220-4223 (March 1, 1990). [IMSE/Ceramics Division]
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A twin-free single crystal of $YBa_2Cu_3O_{6+x}$ (YBC) has been studied at room temperature and 115 K by x-ray diffraction analysis. The crystal was grown from a Y-Ba-Cu-O melt in a gold crucible, leading to a 2% substitution of Au for Cu. Twins were removed from the as-grown crystal by a novel thermomechanical process, resulting in an orthorhombic (Pmmm), single-domain crystal. Oxygen positions and occupancies in the Cu-O basal plane have been refined, showing that the 0(5) site is completely vacant, and the 0(4) atoms are offset from the crystallographic mirror plane positions by 0.15 Å in a zig-zag fashion. Gold, which is a common impurity in crystals grown by the present technique, was found to occupy Cu(1) sites only. Weak superlattice reflections suggest a possible three-dimensional ordering of O and/or Au. To our knowledge, this paper reports the first high resolution x-ray study of an intentionally detwinned, superconducting YBC single crystal.

STRUCTURAL PHASE TRANSFORMATION STUDIES OF THE HIGH T_c SUPERCONDUCTING MATERIALS $Ba_2RCu_3O_{6+x}$, IN AIR,, W. Wong-Ng, L.P. Cook, C.K. Chiang, M.D. Vaudin, D.L. Kaiser, F. Beech, L.J. Swartzendruber, L.H. Bennett, and E.R. Fuller, Jr., Journal of Material Research, Vol. 3, No. 5, pp. 832-839 (September/October 1988). [IMSE/Ceramics Division]

To understand the crystal chemistry and to gain more insight into the effect of oxygen stoichiometry on superconductivity, we have investigated the phase transformations between the orthorhombic and tetragonal structures of several high T_c superconductors, $Ba_2RCu_3O_{6+x}$, where R = Sm, Gd, Y, Ho and Er, and x = 0 to 1. Various techniques including X-ray diffraction, thermogravimetric analysis, neutron scattering, transmission electron microscopy, and determination of Meissner effect have been used to study the nature of the phase transition.

AC magnetic susceptibility measurements suggest that the transformation from the oxygen-rich orthorhombic phase to the oxygen-deficient tetragonal phase involves two orthorhombic phases, designated here as O(A) and O(B). A present there is no clear evidence of where the phase boundary between O(A) and O(B) is. Electron diffraction studies performed on an yttrium material quenched from 670°C show both a splitting of the strong diffraction spots as a result of twinning and also extra, weak, diffuse reflections indicating that the a-axis cell dimension is doubled. Neutron diffraction results show the absence of long-range ordering of oxygen; the electron diffraction results correspond to short-range oxygen ordering.

The structural phase transition temperatures, oxygen stoichiometry and characteristics of the T_c plateaus appear to follow a trend anticipated from the dependence of the ionic radius on the number of f electrons as R progresses across the lanthanide series. Rare earth elements with a smaller ionic radius stabilize the orthorhombic phase to higher temperatures and lower oxygen content. Also, the superconducting properties, viz., the T_c values, are less sensitive to the oxygen content for materials with smaller ionic radius.

CRYSTAL STRUCTURE, ATOMIC ORDERING AND CHARGE LOCALIZATION IN $Pb_2Sr_2Y_{1-x}Ca_xCu_3O_{8+\delta}$ ($x = 0$, $\delta = 1.47$), M. Marezio,*† A. Santoro, J.J. Caponi,† E.A. Hewat,‡ R.J. Cava,* and F. Beech, Physica C, Vol. 169, pp. 401-412 (1990). [IMSE/Reactor Radiation Division]

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Neutron, X-ray and electron diffraction measurements have been carried out on $Pb_2Sr_2Y_{1-x}Ca_xCu_3O_{8+\delta}$ samples. The oxygen incorporated in the structure during the oxidation is located on the (Cu) planes sandwiched between the two (PbO) layers. A theoretical composition of $\delta = 2$ is possible, although in practice only a stoichiometry corresponding to $\delta = 1.9$ has been achieved so far. The extra oxygen present for $\delta > 0$ forms ordered structures in which the Cu cations of the (CuO_δ) planes have square planar coordination in $Pb_2Sr_2YCu_3O_9$, either square planar, pyramidal, and octahedral, or only pyramidal coordination in $Pb_2Sr_2YCu_3O_{9.5}$, and exclusively octahedral coordination in $Pb_2Sr_2YCu_3O_{10}$. In the range of composition $0 \leq \delta \leq 1$, mixtures of two phases are obtained, one with $\delta = 0$ stoichiometry and the other with $\delta = 1$, and whose relative quantities depend on the total amount of oxygen incorporated by the sample. The positive charges induced in $Pb_2Sr_2YCu_3O_{8+\delta}$ by oxygen incorporation oxidize the Cu^{1+} cations to 2+ and some of the Pb^{2+} cations to 4+. An order between Pb^{2+} and Pb^{4+} is established and this localization hinders the charge transfer to the conducting (CuO_2) planes and, for this reason, no superconductivity is present in oxidized samples. The cation valences are estimated from the coordination numbers and from the bond length-bond strength relationship.

$Pb_2Sr_2YCu_3O_8$ becomes superconducting at ≈ 80 K when some of the trivalent Y cations are replaced by divalent Ca. In this case the extra positive charges oxidize the Cu^{2+} cations in the CuO_2 planes instead of Pb^{2+} to 4+ and Cu^{1+} to 2+, as does the incorporation of oxygen. This different behavior can be explained as a concentration effect which changes the oxidation/reduction potentials. When heat treated at 500°C in O_2, $Pb_2Sr_2YCu_3O_8$ behaves similarly to the undoped compound. The oxygen uptake suppresses the superconducting transition which is re-established by heat treating the sample at the same temperature in N_2.

INCORPORATION OF GOLD INTO $YBa_2Cu_3O_7$: STRUCTURE AND T_c ENHANCEMENT, Marta Z. Cieplak,*† Gang Xiao,* C.L. Chien,* A. Bakhshai,* D. Artymowicz,* W. Bryden,* J.K. Stalick, and J.J. Rhyne, Physical Review B, Vol. 42, No. 10, pp. 6200-6208 (October 1, 1990). [IMSE/Reactor Radiation Division]

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The structural and superconducting properties of gold-doped $YBa_2Cu_3O_7$ compounds have been studied with x-ray and neutron diffraction, optical microscopy, weight-loss analysis, magnetization, and resistivity measurements. The solubility of Au in $YBa_2Cu_3O_7$ is close to 10 at. %, below which all samples are thermally stable up to the processing temperature (950°). Metallic Au grains precipitate out when the Au content exceeds 10 at. %. Analysis of the x-ray and neutron-scattering intensities shows that Au substitutes for the Cu(1) chain site exclusively and has a probable valence state of Au^{3+}. The main effect of Au doping is a substantial uniaxial lattice expansion, while the orthorhombic structure is preserved. We present a detailed set of structural data including the Cu-O bond lengths and interplanar distances in a Au-doped sample.

Unlike all other known dopants on Cu sites, the Au dopant is not detrimental to the superconducting properties. On the contrary, T_c is slightly enhanced over that of $YBa_2Cu_3O_7$. Such an enhancement, observed for the first time for a Cu site dopant, is intrinsic and reproducible. We discuss the possible causes of the T_c increase and the features of Au dopant useful in the investigation of high-T_c superconductivity.

NEUTRON DIFFRACTION STUDY OF THE "BROWN PHASE" $BaNd_2CuO_5$, Judith K. Stalick and Winnie Wong-Ng, Materials Letters, Vol. 9, No. 10, pp. 401-404 (June 1990). [MSEL/Ceramics Division]

A neutron Rietveld refinement study of the "brown phase" $BaNd_2CuO_5$ shows that this material is isostructural with the La analog, but adopts a totally different structure from that of the "green phase" BaR_2CuO_5 formed for R = Y and most lanthanide elements. $BaNd_2CuO_5$ is tetragonal, space group P4/mbm(126), with Z = 2 and refined cell parameters a = 6.7015(1) and c = 5.8211(1) Å. The $BaNd_2CuO_5$ framework is built from edge- and face-sharing BaO_{10} and NdO_8 polyhedra, while in the green phase structure the smaller R^{3+} ion is surrounded by seven oxygen atoms. The square-planar CuO_4 groups are isolated in the structure.

4. ELASTIC CONSTANTS AND PHONON SPECTRA

THE BULK MODULUS AND YOUNG'S MODULUS OF THE SUPERCONDUCTOR $Ba_2Cu_3YO_7$, S. Block, G.J. Piermarini, R.G. Munro, and W. Wong-Ng, Advanced Ceramic Materials: Ceramic Superconductors, Vol. 2, No. 3B, p. 601 (July 15, 1987). [IMSE/Ceramics Division]

The isothermal equation of state of the high temperature superconducting ceramic material $Ba_2YCu_3O_7$ has been determined by measurements in a diamond anvil pressure cell using an energy dispersive x-ray diffraction method. The orthorhombic unit cell lattice parameters (a = 3.8856 Å, b = 11.6804 Å, c = 3.8185 Å) were found to have compressions of (2.0%, 2.3%, 1.1%) respectively over the pressure range from one atmosphere to 10.6 GPa at room temperature. Subsequent equation of state analysis of the approximately linear compression of the volume determined that the isothermal bulk modulus was 196 $\pm$ 17 GPa. Young's modulus was estimated to be 235 $\pm$ 20 GPa assuming that the Poisson's ratio for $Ba_2YCu_3O_7$ was 0.3 which is typical of many ceramics.

PHONON DENSITY OF STATES OF SUPERCONDUCTING $YBa_2Cu_3O_7$ AND THE NONSUPERCONDUCTING ANALOG $YBa_2Cu_3O_6$, J.J. Rhyne, D.A. Neumann, J.A. Gotaas, F. Beech, L. Toth,* S. Lawrence,* S. Wolf,* M. Osofsky,* and D.U. Gubser,* Physical Review B, Vol. 36, No. 4, pp. 2294-2297 (August 1, 1987). [IMSE/Reactor Radiation Division]
*Naval Research Laboratory

Neutron scattering has been used to study the vibrational density of states and the atomic structure of the high-temperature superconductor $YBa_2Cu_3O_7$ and the analogous nonsuperconducting compound $YBa_2Cu_3O_6$. The density of states of the superconductor shows a strong double peak at about 20 meV and a second major maximum near 70 meV with additional less-intense features present at intermediate energies. The $YBa_2Cu_3O_6$ material shows a similar energy spectrum above 45 meV; however, below this energy there are significant differences associated with the O vacancies in the linear b-axis chain site.

ELASTIC CONSTANTS AND DEBYE TEMPERATURE OF POLYCRYSTALLINE $Y_1Ba_2Cu_3O_{7-x}$, H.M. Ledbetter, M.W. Austin, S.A. Kim, and Ming Lei, Journal of Materials Research, Vol. 2, No. 6, pp. 786-789 (November-December 1987). [IMSE/Fracture and Deformation Division]

Using ultrasonic methods, the quasi-isotropic elastic stiffness of void-containing $Y_1Ba_2Cu_3O_{7-x}$ were determined. By a composite-material model, these were corrected to the void-free state. From these, the Debye characteristic temperature was calculated. All the elastic stiffnesses fall well below those of polycrystalline $BaTiO_3$, an approximate crystal-structural building block of $Y_1Ba_2Cu_3O_{7-x}$. The low apparent stiffness may result from oxygen vacancies, which soften interionic forces. Also, it may result from microcracks, which reduce elastic stiffness without lowering mass density.

SHEAR-MODULUS CHANGE BELOW T_c IN $YBa_2Cu_3O_{7-x}$, H.M. Ledbetter, M.W. Austin, S.A. Kim, T. Datta,* C.E. Violet,† Journal of Materials Research, Vol. 2, No. 6, pp. 790-792 (November-December 1987). [IMSE/Fracture and Deformation Division]

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The ultrasonic transverse-wave velocity in the high-T_c metal-oxide superconductor $YBa_2Cu_3O_{7-x}$ between 275 and 4 K was measured. Above T_c the velocity shows normal behavior: during cooling, it displays a monotonic increase with decreasing slope. During cooling below T_c the shear modulus departs from normal behavior, increasing as $\Delta G/G = b[1 - (T/T_c)^n]$, where $n = 3$. These results depart dramatically from those expected for a simple second-order normal-superconducting phase transition.

POISSON-RATIO ANOMALIES IN THE $Y_1Ba_2Cu_3O_{7-x}$ SUPERCONDUCTOR, H.M. Ledbetter, S.A. Kim, T. Datta,* J. Estrada,* and C.E. Violet,† Physical Review Letters, October 1987. [IMSE/Fracture and Deformation Division]
*University of South Carolina
†Lawrence Livermore National Laboratory

When cooled from 295 to 4 K, polycrystalline $Y_1Ba_2Cu_3O_{7-x}$ shows anomalous Poisson-ratio behavior. Instead of an expected slow and steady decrease, with a minimum at zero temperature, the superconductor shows two additional minima and maxima, the latter centered at 190 K and 70 K. Magnetic-susceptibility transition begins at 80 K. Thus, the lower maximum relates to the superconducting phase transition. Because the Poisson ratio reveals interatomic-bonding changes, we ascribe the upper maximum to small electronic changes, possibly precursors to the superconducting transition.

LOW-TEMPERATURE ELASTIC CONSTANTS OF A $Y_1Ba_2Cu_3O_{7-x}$ SUPERCONDUCTOR, H.M. Ledbetter, S.A. Kim, M.W. Austin, T. Datta,* J. Estrada,* and C.E. Violet,† Physical Review B, October 1987. [IMSE/Fracture and Deformation Division]
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†Lawrence Livermore National Laboratory

Using ultrasonic methods, from 295 to 4 K, we determined the elastic constants of the high-T_c superconductor $Y_1Ba_2Cu_3O_{7-x}$. We report the Poisson ratio and three elastic-stiffness moduli: bulk, shear, Young. During cooling, the stiffnesses increase smoothly and monotonically until T_c, where they increase abruptly. Above T_c, the shear modulus increases normally; but the bulk modulus shows a slightly abnormal increase. Especially, the Poisson ratio behaves abnormally versus temperature.

REENTRANT SOFTENING IN PEROVSKITE SUPERCONDUCTORS, T. Datta,* C. Almasan,* J. Estrada,* C.E. Violet,† and H.M. Ledbetter, Physical Review Letters, Vol. 37, No. 13, pp. 7502-7505 (May 1, 1988). [IMSE/Fracture and Deformation Division]
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We suggest a model—reentrant elastic softening—that achieves three useful results. First, and principally, it reconciles existing sound-velocity-elastic-constant measurements with

thermodynamics. Second, it leads to Debye characteristic temperatures that agree with those from specific-heat and phonon density-of-states determinations. Third, it links elastic-constant-temperature behavior in Y-Ba-Cu-O and La-Sr-Cu-O. The model predicts a superconducting-state elastic stiffness lower than the normal state.

MEASUREMENT OF SUPERCONDUCTOR STOICHIOMETRY BY PROMPT AND DELAYED NEUTRON ACTIVATION, Richard M. Lindstrom, Transactions, American Nuclear Society, Nuclear Activation Analysis/Applications-I, Vol. 56, pp. 231 (1988). [CAC/Inorganic Analytical Research Division]

Prompt and delayed neutron activation analysis have been applied to the nondestructive measurement of the major metals in small samples of $YBa_2Cu_3O_7$ superconductors, starting materials, and intermediate products. The small activation cross section and low gamma ray energies of Y-90 m make the determination of yttrium imprecise in the presence of the large quantities of Cu-64 radioactivity. The physical parameters for neutron-capture prompt gamma-ray activation analysis (PGAA) are more attractive than delayed counting for stoichiometry measurements. The PGAA spectra of Y, Ba, and Cu show three, four, and six analytically useful gamma ray lines respectively, with nicely balanced intensities for the composition range of interest. A 100 mg sample gives a Poisson precision for yttrium (the weakest element) of 2% after only a 2 hour irradiation. Since the concentrations and mole ratios Y/Cu and Ba/Cu may be calculated immediately after the irradiation ends, the results can be returned within a day.

APPLICATION OF WAVELENGTH DISPERSIVE DIGITAL COMPOSITIONAL MAPPING TO HIGH-TEMPERATURE SUPERCONDUCTORS, R.B. Marinenko, D.E. Newbury, D.S. Bright, R.L. Myklebust, and J.E. Blendell, Microbeam Analysis——1988, D.E. Newbury, Editor, San Francisco Press, pp. 37-40 (1988). [CAC/Gas and Particulate Science Division, and IMSE/Ceramics Division]

The new high-T_c superconductors are advanced ceramic materials prepared from oxide powders which are pressed and sintered. Their physical properties depend on the microstructure, which in turn depends strongly on the processing sequence used in their production. We have used wavelength-dispersive (WDS) x-ray compositional mapping to study the microstructure of the $Ba_2YCu_3O_{6+x}$ ceramics being produced at the National Bureau of Standards so as to understand better the processing procedures and the resulting physical properties.

ELASTIC PROPERTIES OF METAL-OXIDE SUPERCONDUCTORS, Hassel Ledbetter, Journal of Metals, Vol. 40, No. 1, pp. 24-30 (January 1988). [IMSE/ Fracture and Deformation Division]

This article reviews the elastic properties of the new perovskite-related superconductors, especially $Y_1Ba_2Cu_3O_{7-x}$. Considered are such related matters as atomic size, crystal structure, microstructure, twins, and microscopic mechanism. Also examined are related physical properties such as Debye characteristic temperature, thermal expansivity, specific heat, ultrasonic attenuation, and phonon density-of-states.

REENTRANT SOFTENING IN COPPER-OXIDE SUPERCONDUCTORS, C.E. Violet,* T. Datta,† H.M. Ledbetter, C. Almasan,† and J. Estrada,† in High Temperature Superconductors, Materials Research Society Symposium Series, Vol. 99, pp. 375-378 (1988). [IMSE/Fracture and Deformation Division]

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†University of South Carolina

We suggest a model—reentrant elastic softening—that achieves three useful results. First, and principally, it reconciles existing sound-velocity−elastic-constant measurements with thermodynamics. Second, it leads to Debye characteristic temperatures that agree with those from specific heat and phonon density-of-states. Third, it links elastic-constant−temperature behavior in Y-Ba-Cu-O and La-Ba-Cu-O. The model predicts a lower superconducting-state elastic stiffness.

ELASTIC CONSTANTS OF POLYCRYSTALLINE $Y_1Ba_2Cu_3O_{7-x}$, H.M. Ledbetter, S.A. Kim, and D.W. Capone,* in High-Temperature Superconductors II, Materials Research Society Symposium Series, pp. 293-296 (1988). [IMSE/Fracture and Deformation Division]

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We report the elastic constants of a polycrystalline $Y_1Ba_2Cu_3O_{7-x}$ superconductor between 295 and 4 K. Roughly speaking, the elastic constants behave regularly. However, small departures from regular behavior provide much interest. We draw the following conclusions from the measurements: (1) Elastic constants show irregularities below and above T_c. During cooling, all the elastic constants show an irregularity near 200 K. (2) Within measurement error (about 1 part in 1000), none of the elastic stiffnesses shows an abrupt change at T_c, 91 K. (3) Below T_c, all the elastic constants show regular behavior, except υ (Poisson ratio). (4) During cooling, between 160 and 70 K, the material behaves like it undergoes a sluggish phase transformation. (5) The large (4 percent) decrease in Poisson ratio is unexpected. It indicates large interatomic-force changes. (6) The unusual flatness of $\upsilon(T)$ near 295 K suggests unusual material-property changes above ambient temperatures.

HYSTERETIC PHASE TRANSITION IN $Y_1Ba_2Cu_3O_{7-x}$ SUPERCONDUCTORS, H.M. Ledbetter and S.A. Kim, Physical Review B, Vol. 38, No. 16, pp. 11857-11860 (December 1, 1988). [IMSE/Fracture and Deformation Division]

We studied ultrasonic-wave velocities, both longitudinal and shear, in $Y_1Ba_2Cu_3O_{7-x}$, between 5 and 295 K, during both cooling and warming. Both waves, especially the longitudinal, show thermal hysteresis. The results suggest a hysteretic phase change that occurs between 160 and 170 K during cooling and between 170 and 260 K during warming. This phase-change hypothesis explains anomalies in several physical properties. The phase change agrees with thermodynamic-instability predictions. We confirmed the hysteresis in Ho-Ba-Cu-O, where it is smaller than in Y-Ba-Cu-O, and in Eu-Ba-Cu-O, where it is larger. In a companion perovskite, $BaTiO_3$, we observed zero hysteresis. At T_c, 91 K, sound velocities show no measurable change in either magnitude or slope. This continuity disputes the current popular view that, contrary to thermodynamics, elastic stiffness increases upon cooling through T_c into the superconducting

state. We believe that stiffening results from the usual thermal effects after a phase transformation from a stiffer phase.

ELASTIC CONSTANTS OF THE POLYCRYSTALLINE Bi-Pb-Sr-Ca-Cu-O SUPERCONDUCTOR, H.M. Ledbetter, S.A. Kim, R.B. Goldfarb, and K. Togano,* Physical Review B, Vol. 39, No. 13, pp. 9689-9692 (May 1, 1989). [IMSE/Fracture and Deformation Division and CEEE/Electromagnetic Technology Division]

*National Research Institute for Metals, Japan

For polycrystalline $(Bi\text{-}Pb)_2Sr_2Ca_2Cu_3O_{10}$, we report the 5 - 295-K elastic constants: shear modulus (G), Young modulus (E), bulk modulus (B), and Poisson ratio (ν). Both G and E show nearly normal temperature variation. During cooling, B softens between 215 and 65 K. The Poisson ratio decreases abruptly at 215 K and decreases almost five percent in the 295 - 5-K region. In its temperature dependence, this compound resembles $YBa_2Cu_3O_7$. However, it shows much lower elastic stiffness. Corrected to the void-free state, we calculate a Debye temperature of 312 K, versus 436 K for $YBa_2Cu_3O_7$. The Poisson ratio, 0.20, agrees well with that of $YBa_2Cu_3O_7$, 0.21. We use Kresin's model to interrelate Debye temperature, critical temperature, and electron-phonon interaction parameter for four classes of copper-oxide superconductors.

MADELUNG POTENTIALS AND VALENCES IN THE $Y_1Ba_2Cu_3O_7$ SUPERCONDUCTOR, H. Ledbetter and M. Lei,* Physica C, Vol. 166, pp. 483-485 (1990). [IMSE/Fracture and Deformation Division]

*Institute for Metals Research, PRC

Using Ewald's method, we calculated the ion-site potentials and Madelung energy of $Y_1Ba_2Cu_3O_7$ (orthorhombic, Pmmm, No. 47). We considered the effects of copper-ion and oxygen-ion valences. Among seven suggested copper-oxygen ion-charge configurations, only two gave a low electrostatic bonding energy to agree with oxygen vacancies on 01 sites. Only one configuration gives a realistic bulk modulus. We perturbed this configuration by equalizing the Cu1-Cu2 valences and introducing a hole into the CuO_2 plane at the oxygen sites. This configuration also agrees well with observation.

GRÜNEISEN PARAMETER OF $Y_1Ba_2Cu_3O_7$, Hassel Ledbetter, Physica C, Vol. 159, pp. 488-490 (1989). [IMSE/Fracture and Deformation Division]

Contrary to reports that the Grüneisen parameter of $Y_1Ba_2Cu_3O_7$ is approximately 3.0, I argue that this parameter is approximately 1.5, a value consistent with metal oxides. I offer three arguments. One depends on a lower bulk modulus (B), near 101 GPa, than found in high-pressure X-ray diffraction studies, which yield bulk-modulus values up to 200 GPa. The second depends on an ionic-crystal-model calculation of the Grüneisen parameter. The third depends on the Anderson-Grüneisen parameter determined by measuring dB/dT.

IS $Y_1Ba_2Cu_3O_7$ STIFF OR SOFT?, H. Ledbetter and M. Lei,* Journal of Materials Research, Vol. 5, No. 2, pp. 1-4 (February 1990). [IMSE/Fracture and Deformation Division]
*Institute for Metals Research, PRC

Using several measured and calculated physical properties, we argue that the high-T_c metal-oxide superconductor $Y_1Ba_2Cu_3O_7$ is elastically soft compared with $BaTiO_3$ or $SrTiO_3$. We conclude that the bulk modulus equals approximately 107 GPa, despite several high-pressure-x-ray diffraction studies that report values up to approximately 200 GPa. Part of the argument uses an ionic-crystal-model calculation of the bulk modulus.

ELASTIC CONSTANTS, DEBYE TEMPERATURES, AND ELECTRON-PHONON PARAMETERS OF SUPERCONDUCTING CUPRATES AND RELATED OXIDES, Hassel Ledbetter, Ming Lei,* and Sudook Kim, Phase Transitions, Vol. 23, pp. 61-70 (1990). [IMSE/Fracture and Deformation Division]
*Institute for Metals Research, PRC

Using both measurements and modeling, we studied the cohesive and related properties of several oxides. Superconducting oxides include $La_{1.85}Sr_{0.15}CuO_4$, $Y_1Ba_2Cu_3O_7$, and $(Bi\text{-}Pb)_2Sr_2Ca_2Cu_3O_{10}$. Related nonsuperconducting oxides include $SrTiO_3$, $BaTiO_3$, and La_2CuO_4. For these materials, we give the complete quasiisotropic elastic constants corrected to the void-free state. From elastic constants and atomic volume, we calculated Debye characteristic temperatures, ΘD. Using Kresin's model, valid for all values of the electron-phonon parameter, λ, we estimated λ from T_c and ΘD. For the superconducting cuprates, λ ranges from 0.92 (La---0) to 7.96 (T1---0). Except perhaps for T1---0, these parameters fall within a range predicted by theory. For all the above materials, we show the 295--5-K variation of ΘD. We support our elastic-constant measurements with Born-model calculations of the bulk modulus.

SPECIFIC HEAT OF THE HIGH-T_c SUPERCONDUCTOR $(Bi_{1.66}Pb_{0.34})Ca_2Sr_2Cu_3O_{10}$, R.A. Fisher,* S. Kim,* Y. Wu,* N.E. Phillips,* H.M. Ledbetter, and K. Togano,† Physica C, Vol. 162-164, pp. 502-503 (1989). [IMSE/Fracture and Deformation Division]
*Lawrence Berkeley Laboratory
†National Research Institute for Metals, Japan

The specific heat (C) was measured for H=0 and 7T in the ranges 0.4 to 20K and 65 to 125K. The coefficient of the low-temperature linear term in C was 0 ± 0.5 mJ/K^2•mole. On initial cooling, an anomaly in C was observed at T_c, but there were dramatic temperature hysteresis effects.

LOW-TEMPERATURE ELASTIC CONSTANTS OF POLYCRYSTALLINE La_2CuO_4 AND $La_{1.85}Sr_{0.15}CuO_4$, Hassel Ledbetter, S.A. Kim, C.E. Violet,* and J.D. Thompson,† Physica C, Vol. 162-164, pp. 460-461 (1989). [IMSE/Fracture and Deformation Division]

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Using ultrasonic methods, we measured the 295--4-K elastic constants of superconductive $La_{1.85}Sr_{0.15}CuO_4$ and nonsuperconductive La_2CuO_4. These materials show two elastic-constant similarities: nearly the same ambient-temperature elastic constants and an elastic-stiffness minimum in the 20--40-K region. Their principal difference is that La2CuO4 softens 3% during cooling, while $La_{1.85}Sr_{0.15}CuO_4$ softens 30%. This reversible wide-temperature-range softening resembles a magnetic phase transition. Perhaps it relates to the large drop in spin-excitation intensity found by Shirane and coworkers using inelastic neutron scattering.

5. ELECTRICAL CONTACTS

METHOD FOR MAKING LOW RESISTIVITY CONTACTS TO HIGH-T_c SUPERCONDUCTORS, J.W. Ekin, A.J. Panson,* and B.A. Blankenship,* Applied Physics Letters, Vol. 52, No. 4, pp. 331-333 (January 25, 1988). [CEEE/Electromagnetic Technology Division]
*Westinghouse Research & Development Center

A method for making low-resistivity contacts to high T_c superconductors has been developed, which has achieved contact surface resistivities less than 10 $\mu\Omega$ cm^2 at 76 K and does not require sample heating above ~150°C. This is an upper limit for the contact resistivity obtained at high current densities up to 10^2-10^3 A/cm^2 across the contact interface. At lower measuring current densities the contact resistivities were lower and the voltage-current curve was nonlinear, having a superconducting transition character. On cooling from 295 to 76 K, the contact resistivity decreased several times, in contrast to indium solder contacts where the resistivity increased on cooling. The contacts showed consistently low resistivity and little degradation when exposed to dry air over a four-month period and when repeatedly cycled between room temperature and 76 K. The contacts are formed by sputter depositing a layer of a noble metal—silver and gold were used—on a clean superconductor surface to protect the surface and serve as a contact pad. External connections to the contact pads have been made using both solder and wire-bonding techniques.

HIGH-T_c SUPERCONDUCTOR/NOBLE-METAL CONTACTS WITH SURFACE RESISTIVITIES IN THE 10^{-10} Ω CM^2 RANGE, J.W. Ekin, T.M. Larson, N.F. Bergren, A.J. Nelson,* A.B. Swartzlander,* L.L. Kazmerski,* A.J. Panson,† and B.A. Blankenship,† Applied Physics Letters, Vol. 52, No. 21, pp. 1819-1821 (May 23, 1988). [CEEE/Electromagnetic Technology Division]
*Solar Energy Research Institute
†Westinghouse Research and Development Center

Contact surface resistivities (product of contact resistance and area) in the 10^{-10} Ω cm^2 range have been obtained for both silver and gold contacts to high T_c superconductors. This is a reduction by about eight orders of magnitude from the contact resistivity of indium solder connections. The contact resistivity is low enough to be considered for both on-chip and package interconnect applications. The contacts were formed by sputter depositing either silver or gold at low temperatures (< 100°C) on a clean surface of $Y_1Ba_2Cu_3O_{7-\delta}$ (YBCO) and later annealing the contacts in oxygen. Annealing temperature characteristics show that for bulk-sintered YBCO samples there is a sharp decrease in contact resistivity after annealing silver/YBCO contacts for 1 h above ~600°C. Oxygen annealing for longer times (8 h) did not reduce the contact resistivity of silver contacts as much as annealing for 1 h. Auger microprobe analysis shows that indium/YBCO contacts contain a significant concentration of oxygen in the indium layer adjacent to the YBCO interface. Silver and gold contacts, on the other hand, contain almost no oxygen and have favorable interfacial chemistry with low oxygen affinity. Silver also acts as a "switchable" passivation buffer, allowing oxygen to penetrate to the YBCO interface at elevated temperatures, but protecting the YBCO surface at room temperature.

EFFECT OF OXYGEN ANNEALING ON LOW-RESISTIVITY CONTACTS FOR HIGH-T_c SUPERCONDUCTORS, J.W. Ekin, A.J. Panson,* and B.A. Blankenship,* High Temperature Superconductors, M.B. Brodsky, H.L. Tuller, R.C. Dynes, and K. Kitazawa, eds., Proceedings, Materials Research Society, Vol. 99, High Temperature Superconductors, Pittsburgh, PA, pp. 283-286 (1988). [CEEE/Electromagnetic Technology Division]

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A method for making low resistivity contacts to high-T_c superconductors has been developed, consisting of depositing noble metal contact pads (silver or gold) on a clean superconductor surface at low temperatures ($<150°C$). After annealing the silver contact pads in oxygen at intermediate temperatures ($\leq 500°C$) for one hour, contact resistivities less than 2×10^{-8} Ω-cm^2 at 76 K are obtained, about six orders of magnitude less than for indium-solder contacts. Before annealing, the contact resistivities are still very low, in the 10^{-6} to 10^{-5} Ω-cm^2 range at 76 K, which would be useful when contacts with low fabrication temperatures are required. The voltage-current characteristics of the contacts are strongly nonlinear after annealing, having a superconducting transition character. This is ascribed to the critical current of the superconducting material being exceeded at the contact interface. External connections to the contact pads have been made using both solder and thermosonic wire-bonding techniques.

SILVER SCREEN CONTACTS TO SINTERED YBaCuOx POWDER FOR RAPID SUPERCONDUCTOR CHARACTERIZATION, John Moreland and L.F. Goodrich, Proceedings of the Applied Superconductivity Conference, IEEE Transactions on Magnetics, Vol. MAG-25, No. 2, pp. 2056-2059 (March 1989). [CEEE/Electromagnetic Technology Division]

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

OHMIC CONTACTS TO HIGH-TC SUPERCONDUCTORS, J.W. Ekin, Proceedings of the SPIE October 1989 Conference, Processing of Films for High Tc Superconducting Electronics, Vol. 1187, pp. 359-364 (1990). [CEEE/Electromagnetic Technology Division]

This note summarizes and gives references describing the details of a method for reducing high T_c contact surface resistivities, ρ_c, to the 10^{-10} Ω cm^2 range $\rho_c = RA$, where R is the contact resistance and A is the contact area). The reduction was obtained using both gold and silver contacts, and represents a decrease in contact resistivity by over eight orders of magnitude from that obtained using indium solder connections. We have obtained most of the results so far for $YBa_2Cu_3O_{7-\delta}$ (YBCO) but preliminary data for the Bi and Tl based compounds will also be summarized.

PROSPECTS FOR HIGH TEMPERATURE SUPERCONDUCTOR-SEMICONDUCTOR CONTACTS, John Moreland, J.W. Ekin, and T. Larson, Proceedings of the ASM International Third Electronic Materials and Processing Congress, August 20-23, 1990, San Francisco, CA, pp. 195-201 (1991). [CEEE/Electromagnetic Technology Division]

Development of high temperature superconductor-semiconductor contacts with very low contact resistivities is required for microelectronic applications such as circuit interconnects or superconductor-semiconductor-superconductor (S/Semi/S) proximity effect devices. Several thin-film deposition methods have been developed for forming very low resistance contacts to high temperature superconductors. For example, we have measured contact resistivities as low as 10^{-10} Ω-cm^2 for annealed Ag films on sintered $YBa_2Cu_3O_{7-\delta}$ samples. Such a contact could potentially be part of a superconductor-semiconductor contact that relies on an intervening normal metal, spanning superconductor and semiconductor interfaces. Presently we are surveying materials commonly used to make ohmic contacts to Si and GaAs for their compatibility with high-temperature superconductors. Prime material characteristics necessary for potential contact structures are (1) low chemical reactivity of the intervening materials with high-temperature superconductors, (2) limited interdiffusion of multilayer constituents, and (3) stable, low contact resistivity, ohmic I-V characteristics.

PROSPECTS FOR HIGH TEMPERATURE SUPERCONDUCTOR-SEMICONDUCTOR CONTACTS, John Moreland, J.W. Ekin, and T. Larson, New Technology and Electronic Packaging, B.R. Livesay and N.D. Nagarkan, editors, Materials Park, Ohio: ASM International, pp. 195-201 (1990). [CEEE/Electromagnetic Technology Division]

Development of high temperature superconductor-semiconductor contacts with very low contact resistivities is required for microelectronic applications such as circuit interconnects or superconductor-semiconductor-superconductor (S/Semi/S) proximity effect devices. Several thin-film deposition methods have been developed for forming very low resistance contacts to high temperature superconductors. For example, we have measured contact resistivities as low as 10^{-10} Ω-cm^2 for annealed Ag films on sintered $YBa_2Cu_3O_{7-\delta}$ samples. Such a contact could potentially be part of a superconductor-semiconductor contact that relies on an intervening normal metal, spanning superconductor and semiconductor interfaces. Presently we are surveying materials commonly used to make ohmic contacts to Si and GaAs for their compatibility with high-temperature superconductors. Prime material characteristics necessary for potential contact structures are (1) low chemical reactivity of the intervening materials with high-temperature superconductors, (2) limited interdiffusion of multilayer constituents, and (3) stable, low-contact resistivity, ohmic I-V characteristics.

6. ELECTRONIC STRUCTURE

RESONANT PHOTOEMISSION STUDY OF SUPERCONDUCTING Y-Ba-Cu-O, Richard L. Kurtz, Roger L. Stockbauer, Donald Mueller,* Arnold Shih,* Louis E. Toth,* Michael Osofsky,* and Stuart A. Wolf,* Physical Review B Rapid Communications, Vol. 35, No. 16, pp. 8818-8820 (June 1, 1987). [CCP/Surface Science Division]

*Naval Research Laboratory

Ultraviolet photoelectron spectra of 93-K superconducting compound, $YBa_2Cu_3O_7$, have been obtained using photon energies ranging from 60 to 106 eV. Resonant photoemission is used to identify the chemical origin of the features in the valence-band electronic structure.

SOFT X-RAY ABSORPTION AND EMISSION SPECTRA AND THE ELECTRONIC STRUCTURE OF THE $Ba_2YCu_3O_{7-x}$ SUPERCONDUCTOR, K.-L. Tsang,* C.H. Zhang,* T.A. Callcott,* L.R. Canfield, D.L. Ederer, J.E. Blendell, C.W. Clark, N. Wassdahl,† J.E. Rubensson,† G. Bray,† N. Mortensson,† J. Nordgren,† R. Nyholm,‡ and S. Cramm,§ Physical Review B, Vol. 37, No. 4, pp. 2293-2296, (February 1, 1988). [CRR/Radiation Physics Division, IMSE/Ceramics Division]

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We present ϒ-beam-excited soft-x-ray emission spectra and total-photoelectron-yield spectra in the 20-600 eV photon energy range for $Ba_2YCu_3O_{7-x}$. In soft-x-ray emission, the $N_{4,5}$ structure of Ba, the $M_{4,5}$ spectrum of Y, and the K spectrum of O provide a direct measure of the p-type partial density of states (p-PDOS) localized on the respective atomic sites. In each case the p-PDOS is very small at the Fermi energy ε_F. The maximum in the O K spectrum is 2.6 ± 0.5 eV below ε_F. This result provides direct confirmation that the shoulder previously found at ε_F - 2.3 eV in photoelectron emission spectra is associated with O p orbitals. No changes are observed between spectra taken above and below T_c.

SYNCHROTRON RADIATION STUDIES OF THE ELECTRONIC STRUCTURES OF HIGH-T_c SUPERCONDUCTORS, Richard L. Kurtz, AIP Proceedings No. 165, American Vacuum Society Series 3: Thin Film Processing and Characterization of High-T_c Superconductors, J.M.E. Harper, R.J. Colton, and L.C. Feldman, eds., p. 222 (1988). [CBS/Surface Science Division]

Experimental measurements of the electronic structure of the high-T_c superconductors $La_{2-x}Sr_xCuO_4$ and $YBa_2Cu_3O_{7-x}$ are reviewed and discussed in the context of determining the strengths of the interactions within the valence bands. It is concluded that the Cu $3d$ - O $2p$ valence bands are highly hybridized and there are strong electron-electron correlations. It is shown that the Cu^{3+} configuration ($3d^8$) is energetically unfavorable and the Cu in these systems is best thought of as having an approximate valence of +2. The influence of surface stoichiometry on the electronic structure is discussed and preliminary molecular adsorption studies are reported.

TOTAL DIELECTRIC FUNCTION: ALGEBRAIC SIGN, ELECTRON-LATTICE RESPONSE, AND SUPERCONDUCTIVITY, Philip B. Allen,* Marvin L. Cohen,† and David R. Penn, Physical Review B, Vol. 38, No. 4, pp. 2513-2525 (August 1, 1988). [CRR/Radiation Physics Division]

*Naval Research Laboratory and Department of Physics, State University of New York, Stony Brook
†Department of Physics, University of California, Berkeley

The interaction between two test charges in a solid can be described in terms of a total dielectric function that includes electronic and lattice polarization. Crystal stability requires the eigenvalues of ϵ^{-1} to be ≤ 1. Some implications for superconductivity are discussed. A total dielectric function for the electron-lattice system is derived in the mean field approximation and its inverse is explicitly constructed. The low-lying poles of ϵ^{-1} give the correct phonon frequencies as determined by the usual dynamical matrix.

A PHOTOEMISSION STUDY OF HIGH T_c OXIDES, Donald Mueller,* Arnold Shih,* Louis E. Toth,* Michael Osofsky,* Stuart A. Wolf,* Richard L. Kurtz, and Roger L. Stockbauer, Physical Review B, Rapid Communications, Vol. 37, pp. 7936 (1988). [CCP/Surface Science Division]

*Naval Research Laboratory

A number of ceramic compounds including $YBa_2Cu_3O_7$ have been reported to superconduct at temperatures in excess of 35 K. A critical feature in the processing of these compounds required to obtain good superconductive properties appears to be a final annealing stage in an oxygen containing atmosphere to increase the oxygen content of the materials. Without the oxygen anneal step, the superconducting transition temperature drops and the drop in the temperature versus resistance curve becomes less defined.

To aid in the development of a better understanding of the electronic structure in this class of materials, we have examined changes in the electronic density of states for $YBa_2Cu_3O_x$ with oxygen content via ultraviolet photoelectron spectroscopy. Samples with x = 6.95, 6.5 and 6.05 were examined.

X-RAY POWDER DIFFRACTION CHARACTERIZATION OF BaR_2CuO_5 (R=YTTRIUM AND THE LANTHANIDES) AND RELATED COMPOUNDS, W. Wong-Ng, M.A. Kuchinski, H.F. McMurdie, and B. Paretzkin, Powder Diffraction, Vol. 4, No. 1 (March 1989). [IMSE/Ceramics Division]

A series of $BaO:R_xO_y:CuO$ materials, where R=Y, La, Ce, Pr, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu, has been prepared and characterized by X-ray powder diffraction methods. All BaR_2CuO_5 phases, commonly referred to as the "green phases," are orthorhombic with space group PBcn and are isostructural with each other. These single phase materials could be prepared with most lanthanides, except for La, Ce, Pr, Nd, and Tb. Possible reasons for the exceptions are discussed. Both La and Nd tend to form a brown solid solution of $Ba_{2-2x}R_{4-2x}Cu_{2-x}O_{10-2x}$ with a tetragonal space group of P4/mbm. The major phases found in the Ce, Pr, and Tb compositions are the perovskite-related structures $BaRO_3$, and in the Pr case, $Ba_2PrCu_3O_{6+x}$ as well. The cell parameters of the green phase materials increase progressively from the Lu compound to the Sm compound: 'a' ranges from 7.0506(6) to 7.2754(4) Å, 'b' from 12.0534(8) to 12.4029(7) Å, 'c' from 5.6099(5) to 5.7602(3) Å, and the cell volume from 467.75(6) to 519.78(4) $Å^3$. A correlation of the crystallographic data with the size of the R elements is given.

RESONANT PHOTOEMISSION AND CHEMISORPTION STUDIES OF Tl-Ba-Ca-Cu-O, Roger L. Stockbauer, Steven W. Robey, Richard L. Kurtz, D. Mueller,* A. Shih,* A.K. Singh,* L. Toth,* and M. Osofsky,* AIP Proceedings No. 182, Hi T_c Superconducting Thin Films, Devices, and Characterization, G. Margaritondo, R. Joynt, and M. Onellion, eds., p. 276 (1989). [NML/Center for Chemical Physics]

*Naval Research Laboratory, Washington, DC

Resonant photoemission has been used to study the electronic states and electron-electron interactions in a bulk sample of Tl-Ba-Ca-Cu-O high temperature superconductor. The electronic structure, i.e., broad peaks in the valence band at 3.2 and 5.5 eV and satellites at 10 and 13 eV binding energy, is similar to that observed for the 1-2-3 materials. This indicates that the electronic states and interactions are similar in these different classes of superconductor.

The surface reactivity has been probed with photoemission using controlled exposures of atmospheric gases. Both H_2O and CO_2 react with a sticking probability of near 0.2, forming hydroxide and carbonate species, respectively. O_2 is non-reacting while CO reacts only slightly.

RESONANT EXCITATION OF AN OXYGEN VALENCE SATELLITE IN PHOTOEMISSION FROM HIGH-T_c SUPERCONDUCTORS, Richard L. Kurtz, Steven W. Robey, Roger L. Stockbauer, D. Mueller,* A. Shih,* and L. Toth,* Physics Review B, Vol. 39, No. 7, pp. 4768-4771 (March 1, 1989). [NML/Center for Chemical Physics]

*Naval Research Laboratory, Washington, DC.

A detailed analysis of the intensities of valence band photoelectron features of superconducting $YBa_2Cu_3O_{7-x}$ and semiconducting $La_{1.85}Sr_{0.05}CuO_4$ has revealed a resonance in the peak located at a binding energy of $\approx$9.5 eV for photon energies spanning the onset of O 2s excitations. This demonstrated conclusively that the feature is associated with oxygen excitations. The origin of the satellite is described and its disappearance on superconducting surfaces is explained.

ELECTRONIC STRUCTURE OF HIGH-T_c SUPERCONDUCTORS STUDIED USING PHOTOELECTRON SPECTROSCOPY, Richard L. Kurtz, Steven W. Robey, Roger L. Stockbauer, D. Mueller,* A. Shih,* L. Toth,* A.K. Singh,* and M. Osofsky,* Vacuum, Vol. 39, Nos. 7 & 8, pp. 611-615 (1989). [NML/Center for Chemical Physics]

*Naval Research Laboratories, Washington, DC.

Fundamental information about the structure of the valence band and the chemical valence states of the various constituents of the La-Sr-Cu, Y-Ba-Cu, Bi-Sr-Ca-Cu, and Tl-Ca-Ba-Cu oxides have been obtained using photoelectron spectroscopy. These results show that the one-electron theories do not adequately describe the electronic structure of these superconductors. The atomic origins of the features observed in the valence bands have been investigated by studying photoemission resonances and changes in excitation cross-sections with photon energy. Results to date suggest that these materials have varying densities of states at the Fermi level, valence bands composed of O *2p* and Cu *3d* states, and display no significant changes in the band structure associated with the superconducting behavior when the temperature is lowered below T_c. In addition, the complex surface chemistry of these oxides makes it essential to study the surface stoichiometry

and the interaction of simple molecules. O_2 and CO are found to interact only weakly with the surfaces of the materials studied to date, while H_2O and CO_2 react strongly, forming hydroxides and carbonates.

ELECTRONIC CORRELATIONS AND SATELLITES IN SUPERCONDUCTING OXIDES, K.J. Chang,* Marvin L. Cohen,† and David R. Penn, Physical Review B, Vol. 38, No. 13, pp. 8691-8696 (1988). [CRR/Radiation Physics Division]

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†Department of Physics, University of California

The satellite observed at binding energy of 12-13 eV below the Fermi level in the high-T_c oxide superconductors $La_{2-x}Sr_xCuO_4$ and $YBa_2Cu_3O_{7-\delta}$ is viewed as originating from a state with two holes bound at the same Cu site. As in the case of Ni metal, the satellite is caused by an intra-*d*-shell shakeup process into a Cu $3d^8$ final state and its intensity is enhanced at resonance because of a super-Koster-Kronig transition. Based on the *t*-matrix approach for the hole self-energy, we study the effect of electronic correlations on the one-electron band structure. We examine the position of the satellite and find a large Coulomb interaction energy of ~ 5 eV at the Cu site when the experimental satellite position is duplicated by the theory. Since this energy is comparable with the 3*d* bandwidth, the two-hole bound state is a high-energy excitation. This indicates that in the ground state a creation of two holes at the Cu site is unlikely and thus holes are formed at the O sites when Sr is substituted for La in La_2CuO_4 and when the oxidation is increased in $YBa_2Cu_3O_{7-\delta}$.

OXYGEN PARTIAL DENSITY OF STATES CHANGE IN THE $YBa_2Cu_3O_x$ COMPOUNDS FOR $x \approx 6$, 6.5, 7 MEASURED BY SOFT X-RAY EMISSION, C.H. Zhang,* T.A. Callcott,* K.-L. Tsang,* D.L. Ederer, J.E. Blendell, C.W. Clark, T. Scimeca,† and Y.-W. Liu,‡ Physical Review B, Vol. 39, No. 7, pp. 4796-4799 (March 1, 1989). [CRR/Radiation Physics Division, IMSE/Ceramics Division]

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We present oxygen *K* soft x-ray emission spectra for the $YBa_2Cu_3O_x$ compounds with *x* nominally equal to 6, 6.5, and 7 and compare them with x-ray emission spectra determined from recent band-structure calculations. The *K* emission spectrum of O provides a measure of the *p*-type local partial density of states (*p*-LPDOS) at the oxygen sites. As x decreases from 7-δ to about 6.5, a chemical shift of the entire spectrum to lower energy indicates that screening is modified for all oxygen sites. The integrated intensity of the spectra is nearly unchanged by oxygen removal, indicating an increase in *p*-LPDOS per oxygen site. These results and changes in the spectral shape suggest that itinerant electron density near the O atoms is reduced and bound electron density is increased as oxygen is removed.

ELECTRONIC DENSITY OF STATES OF HIGH TEMPERATURE SUPERCONDUCTORS FROM SOFT X-RAY SPECTROSCOPY, T.A. Callcott,* K.-L. Tsang,* C.H. Zhang,* D.L. Ederer, C.W. Clark, N. Wassdahl,† J.E. Rubensson,† G. Bray,† N. Mortensson,† J. Nordgren,† R. Nyholm,‡ and S. Cramm,§ Extended Abstracts, High-Temperature Superconductors II, April 5-9, 1988, Reno, Nevada (1988). [CRR/Radiation Physics Division, IMSE/Ceramics Division]

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We present electron and photon beam excited soft x-ray emission spectra for the $YBa_2Cu_3O_{7-x}$ superconductor. The soft x-ray emission spectra determine the p-type partial density of states (p-PDOS) localized on the Ba, Y, and O atomic sites. The p-PDOS are very small at the Fermi energy ε_F, and exhibit peaks at 3.5 to 4 eV below ε_F. K emission spectra of O confirm the interpretation that the structure observed in previous photoemission measurements is associated with the O 2p orbitals. The copper $L_{2,3}$ emission spectrum, which samples d-PDOS, and the O K emission spectrum have been modeled theoretically and are in excellent agreement with the soft x-ray emission spectrum. Differences between the model and the experiment are attributed to correlation effects.

SOFT X-RAY ABSORPTION AND EMISSION SPECTRA OF THE $YBa_2Cu_3O_{7-x}$ SUPER-CONDUCTOR, K.-L. Tsang,* C.H. Zhang,* T.A. Callcott,* L.R. Canfield, D.L. Ederer, J.E. Blendell, C.W. Clark, Drexel High T_c Conference Proceedings (July 1987). [CRR/Radiation Physics Division, IMSE/Ceramics Division]

*University of Tennessee

We present total photoelectron yield spectra and electron-beam excited soft x-ray emission spectra for the $YBa_2Cu_3O_{7-x}$ superconductor. The 2+ valencies of Cu and Ba in the compound are confirmed by total photoelectron yield measurements. Emission spectra determine the p-type partial density of states (p-PDOS) localized on the Ba, Y, and O atomic sites. The p-PDOS are very small at the Fermi energy ε_F, and exhibit peaks at 3.5 to 4 eV below ε_F. K emission spectra of O confirm the interpretation that the structure observed in previous photoemission measurements is associated with O p orbitals.

SOFT X-RAY ABSORPTION AND EMISSION SPECTRA AND THE ELECTRONIC STRUCTURE OF SOME EXOTIC MATERIALS, D.L. Ederer, L.R. Canfield, T.A. Callcott,* K.-L. Tsang,* C.H. Zhang,* E.T. Arakawa,† SPIE, Vol. 911 X-Ray and VUV Interaction Data Bases, Calculations, and Measurements, pp. 75-79 (1988). [CRR/Radiation Physics Division]

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The technique of soft x-ray fluorescence spectroscopy (SXE) is complementary to that of photoemission spectroscopy (PES). SXE probes the local partial density of states (PDOS), selects dipole allowed symmetries, and is not necessarily surface sensitive. PES on the other hand, averages over the DOS and can be used to measure the dispersion of the energy bands. PES is also very surface sensitive. We present measurements on the high T_c superconductors, the

quasicrystalline phase of AlMn, and the LiAl intermetallic alloy. These measurements provide insight for theoretical modeling. In the case of the high T_c compound and the intermetallic compound the measurements are in good agreement with the theory. However, for the quasicrystals the measurements provide new insights to challenge theory.

SOFT X-RAY ABSORPTION AND EMISSION SPECTRA AND THE ELECTRONIC STRUCTURE OF THE $Ba_2YCu_3O_{7-x}$ SUPERCONDUCTOR, K.-L. Tsang,* C.H. Zhang,* T.A. Callcott,* L.R. Canfield, D.L. Ederer, J.E. Blendell, C.W. Clark, N. Wassdahl,† J.E. Rubensson,† G. Bray,† N. Mortensson,† J. Nordgren,† R. Nyholm,‡ and S. Cramm,§ Journal de Physique, Colloque C9, supplément au n°12, Tome 48, C9-1193 - C9-1197 (décembre 1987). [CRR/Radiation Physics Division, IMSE/Ceramics Division]

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§Universität Hamburg, West Germany

We present e-beam excited soft x-ray emission spectra and total photoelectron yield spectra in the 20-600 eV photon energy range for the $Ba_2YCu_3O_{7-x}$ superconductor. We confirm the 2+ valency of Cu in the compound by total yield measurements. In soft-x-ray emission, the $N_{4,5}$ spectrum of Ba, the $M_{4,5}$ spectrum of Y, and the K spectrum of O provide measures of the p-type partial density of states (p-PDOS) localized on the respective atomic sites. In each case the p-PDOS is very small at the Fermi energy with the first peak in the p-PDOS lying 3.5 to 4 eV below the Fermi energy. The K spectra of O confirm the interpretation that the structures observed in the photoemission measurements are associated with the O $2p$ orbitals. Finally no changes are observed between spectra taken above and below T_c.

SOFT X-RAY EMISSION SPECTRA AND THE BONDING OF ALUMINUM, T.A. Callcott,* K.-L. Tsang,* C.H. Zhang,* D.L. Ederer, and E.T. Arakawa,† Journal de Physique, Colloque C9, supplément au n°12, Tome 48, C9-1053 - C9-1057 (décembre 1987). [CRR/Radiation Physics Division]

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The $L_{2,3}$ soft x-ray emission (SXE) spectra of Al in metallic Al, Al-Mn alloys, dilute Al-Mg alloys, the intermetallic compounds LiAl and Ni_3Al, the semiconducting alloy (Al-Ga)As and insulating Al_2O_3 are presented. The spectra provide a measure of the s-like partial density of states (PDOS) localized at the Al atoms and show prominent qualitative features that may be identified with each of the major types of bonding in solids, i.e., metallic, covalent and ionic.

The spectra of metallic Al and of the Al-Mn alloys have parabolic densities of states characteristic of nearly free electron PDOS's. Al in Mg alloys have metallic PDOS's with a strong enhancement of the low energy region of the spectrum which is associated with screening of the extra charge on the Al ion core.

In LiAl, the Al has tetragonal local symmetry, the spectrum is similar to that of silicon and the bonding is a combination of covalent and metallic. In $Al_xGa_{1-x}As$ alloys, the Al has tetragonal local symmetry, but the spectrum is strongly modified from the characteristic covalent form by the presence of a strong ionic component in the bonding. Finally, the spectrum of Al_2O_3 shows features characteristic of localize electronic orbitals and ionic bonding.

ELECTRONIC STRUCTURE OF THE ICOSAHEDRAL AND OTHER PHASES OF ALUMINUM-MANGANESE ALLOYS STUDIED BY SOFT-X-RAY EMISSION SPECTROSCOPY, D.L. Ederer, R. Schaefer, K.-L. Tsang,* C.H. Zhang,* T.A. Callcott,* and E.T. Arakawa,† Physical Review B, Vol. 37, No. 15, pp. 8594-8597 (May 15, 1988). [CRR/Radiation Physics Division]

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Aluminum $L_{2,3}$ soft-x-ray emission spectra have been observed for several aluminum-manganese alloys exhibiting icosahedral, decagonal, and the normal crystalline symmetries. The spectra of all the alloys are qualitatively similar and are slightly narrower ($\sim$0.2 eV) than the width observed for metallic aluminum. Compared to the sample with crystalline symmetry, the quasicrystals show no chemical shift and a decrease in the partial density of states of s and d symmetry at the Fermi energy, contradicting the predictions of several calculations.

WIGNER-SEITZ LOCAL-ENVIRONMENT STUDY OF THE HIGH-T_c SUPERCONDUCTORS, M. Melamud, L.H. Bennett, and R.E. Watson,* Physical Review B, Vol. 38, No. 7, pp. 4624-4631 (September 1, 1988). [IMSE/Ceramics Division]

*Brookhaven National Laboratory

The near-neighbor environments and the bonding of atoms in the high-T_c superconductors are studied using a Wigner-Steitz-cell construction. Assuming metallic radii for the atoms, it is shown that the Ba, Y, and La atoms have large coordination numbers, implying a three-dimensional bonding scheme. The La-Cu-O type ($\simeq$40 K) and the Y-Ba-Cu-O type ($\simeq$90 K) superconductors both display the same bonding characteristics.

MOSSBAUER STUDY OF THE EFFECT OF OXYGEN STOICHIOMETRY ON THE HIGH T_c SUPERCONDUCTOR $Y_1Ba_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$, L.J. Swartzendruber, L.H. Bennett, M.Z. Harford,* and Mark Rubinstein,* Journal of Superconductivity, Vol. 1, No. 2, pp. 219-230 (1988). [IMSE/Ceramics Division and Metallurgy Division]

*Naval Research Laboratory

The Mossbauer effect of oxygen-depleted $YBa_2Cu_3O_{7-x}$ and $PrBa_2Cu_3O_{7-x}$ doped with 3% ^{57}Fe has been investigated at room temperature. A manifold of quadrupole-split spectra has been found, whose parameters are in general agreement with those found by other workers. In addition, the Mossbauer spectra show that a fraction of the Fe sites develop magnetic order at room temperature when the oxygen content is reduced. It is demonstrated that the observed asymmetries in the Mossbauer spectra can be the result of a preferential alignment of the platelike crystallites that arises during the normal sample preparation process. The tendency to bond with

the oxygen atoms is presumed to be responsible for the discreteness of the Mossbauer spectra as a function of oxygen depletion.

MÖSSBAUER HYPERFINE FIELDS IN $RBa_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$ SUPERCONDUCTORS, Mark Rubinstein,* M.Z. Harford,* L.J. Swartzendruber, and L.H. Bennett, Journal de Physique, Colloque C8, Supplément au n° 12; Tome 49 (décembre 1988). [IMSE/Ceramics Division and Metallurgy Division]
*Naval Research Laboratory

Room temperature ^{57}Fe Mössbauer spectra of $RBa_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$ [R=Y, Pr, Er] were obtained from samples with varying x. A magnetically split hyperfine field spectrum was observed for the most oxygen-deficient Y sample, for all the Pr samples, and for none of the Er samples.

MAGNETIC PROPERTIES OF SOME HIGH T_c SUPERCONDUCTING COMPOUNDS, P.F. Miceli,* J.M. Tarascon,* B.G. Bagley,* L.H. Greene,* P. Barboux,* G.W. Hull,* M. Giroud,* J.J. Rhyne, and D.A. Neumann, in High Temperature Superconductors, Vol. 20, Tenth Winter Meeting on Low Temperature Physics, World Scientific Publishing, p. 89 (1989). [IMSE/Reactor Radiation Division]
*Bell Communications Research

The magnetic properties of two high temperature superconducting compounds having Co substitutions for Cu are studied by neutron scattering. The "123" system can exhibit two antiferromagnetic transitions or superconductivity depending upon the composition. In the "n=1" phase of the Bi based system, the Co substitution displays antiferromagnetic ordering with a small ferromagnetic canting of the spins.

THEORETICAL MODELS FOR HIGH-TEMPERATURE SUPERCONDUCTIVITY, R.C. Casella, Il Nuovo Cimento, Vol. 10 D, N. 12, pp. 1439-1448 (Dicembre 1988). [IMSE/Reactor Radiation Division]

A semi-phenomenological analysis is given of the effects of certain band structure features on the gap ratios $2\Delta/k_BT_c$ for high-T_c superconductors, including multigap systems. In addition to phonons other intermediate bosons (IB) mediating the superconducting interaction are considered. Interesting results emerge when the IB energy exceeds the widths of possible narrow peaks in the density of states associated with sub-bands presumably belonging to substructures such as stacked Cu-O planes. Comparison with experiment is made. In particular, data obtained by Warren *et al.* via nuclear-spin relaxation on $Ba_2YCu_3O_{7-\delta}$ can be interpreted within the present framework in terms of a model having an IB of energy ≥ 1 eV, which exceeds the predicted width (≥ 0.3 eV) of a peak in the density of states containing the normal-state Fermi level. This suggests that the IB is not a phonon.

DEPENDENCE OF T_c ON THE NUMBER OF CuO_2 PLANES PER CLUSTER IN INTERPLANER-BOSON-EXCHANGE MODELS OF HIGH-T_c SUPERCONDUCTIVITY, R.C. Casella, Solid State Communications, Vol. 70, No. 1, pp. 75-77 (1989). [IMSE/Reactor Radiation Division]

The critical temperature $T_c(n)$ of systems, such as the $T\ell$ and Bi compounds, containing n CuO_2 planes per cluster is discussed theoretically within semi-phenomenological models which are based upon the exchange of (unspecified) high-energy bosons ($\omega >> \omega_{phonon}$) between different CuO_2 planes. Applied to representative data for the $T\ell_2$ compounds, the models predict rapid saturation of $T_c(n)$ at relatively low n, with the possibility of a diminution beyond n = 3. Within the theoretical framework these qualitative features appear to be quite general for this class of materials.

SOME NORMAL STATE PROPERTIES OF HIGH T_c COMPOUNDS, Mark Rubinstein,* M.Z. Harford,* T.K. Chaki,* L.J. Swartzendruber, and L.H. Bennett, Proceedings of High T_c Superconductors: Magnetic Interactions, Gaithersburg, MD, October 11-13, 1988, Progress in High Temperature Superconductivity, Vol. 17, pp. 292-302 (1989). [IMSE/Metallurgy Division]
*Naval Research Laboratory

The ^{57}Fe Mossbauer Effect of oxygen-depleted YBCO has been investigated. A magnetically split 6-line hyperfine field spectrum was observed, indicating that Fe atoms which substitute for Cu(2) atoms participate in antiferromagnetism of YBCO. In addition, the temperature dependence of the resistivity of BSCCO and YBCO was investigated. Both compounds show a large increase in resistivity as the oxygen content is depleted from the samples with increased temperature.

PROCEEDINGS OF HIGH-T_c SUPERCONDUCTORS: MAGNETIC INTERACTIONS, L.H. Bennett, Y. Flom,* G.C. Vezzoli,† Editors, Gaithersburg, Maryland, October 11-13, 1988, Progress in High Temperature Superconductivity, Vol. 17, 404 p., New Jersey: World Scientific Publishing (1989). [IMSE/Metallurgy Division]
*NASA-Goddard Space Flight Center
†U.S. Army Laboratory Command

This is the collected proceedings of a three-day workshop, sponsored by the National Institute of Standards and Technology and the Goddard Space Flight Center, which was held at NIST in Gaithersburg, Maryland, on October 11-13, 1988. The mission of the conference was to bring together invited experts, which included both world class solid-state theorists and experimentalists, to assess the role of magnetic phenomena as related to the great discovery of superconductivity near and above liquid-nitrogen temperature, as well as to provide a forum for interaction between communities representing fundamental research and application of HTSC materials.

ELECTRON-EXCITED $4d \rightarrow 4f$ Ba RESONANCES IN $YBa_2Cu_3O_{7-\delta}$: SELECTION RULES AT INTERMEDIATE ELECTRON ENERGY, C.W. Clark, J.A.D. Matthew,* M.G. Ramsey,† and F.P. Netzer,† Physical Review A, Vol. 40, No. 9, pp. 4902-4906 (November 1, 1989). [CRR/Radiation Physics Division]
*University of York, England
†Universität Innsbruck, Austria

Observations of electron-excited $4d^{10}4f^0 \rightarrow 4d^9 4f^1$ giant resonances of Ba in the high-T_c superconductor $YBa_2Cu_3O_{7-\delta}$ are compared with previous work on the corresponding La transition in various environments. Breakdown of dipole selection rules is observed even at electron primary energies of a few keV, but for low-angle scattering it is possible to rationalize the results in terms of symmetry-related selection and intensity rules appropriate to intermediate coupling. This interpretation is supported by atomic Hartree-Fock calculations of the final-state multiplets.

7. ENERGY GAP AND TUNNELING SPECTRA

ELECTRON TUNNELING MEASUREMENT OF THE ENERGY GAP IN A La-Sr-Cu-O SUPERCONDUCTOR, J. Moreland, A.F. Clark, H.C. Ku,* and R.N. Shelton,* Cryogenics, Vol. 27, pp. 227-228 (1987). [CEEE/Electromagnetic Technology Division]
*Iowa State University

We have used the break junction technique to determine the energy gap of lanthanum-strontium-copper-oxide, one of the new high critical temperature superconductors. The current-voltage characteristics demonstrated a variety of tunneling behaviors. The best characteristic indicating quasiparticle tunneling between superconducting electrodes implied an energy gap of 7.0 ± 0.1 meV. Derivatives of other characteristics showed weak structure indicating possible energy gaps up to 9 meV.

TUNNELING SPECTROSCOPY OF A La-Sr-Cu-O BREAK JUNCTION: EVIDENCE FOR STRONG-COUPLING SUPERCONDUCTIVITY, John Moreland, A.F. Clark, L.F. Goodrich, H.C. Ku,* and R.N. Shelton,* Physical Review B, Vol. 35, No. 16, pp. 8711-8713 (June 1, 1987). [CEEE/Electromagnetic Technology Division]
*Iowa State University

Detailed structure in the quasiparticle tunneling has been observed in La-Sr-Cu-O superconductive tunneling junctions using the break-junction technique. Variability in the energy gap and associated structure in the current-voltage curves are observed indicating significant inhomogeneity in the superconducting properties. Large energy gaps (7.0 meV) and deep structure in the conductance derivative are evidence for a strong-coupling mechanism.

BREAK-JUNCTION TUNNELING MEASUREMENTS OF THE HIGH-T_c SUPERCONDUCTOR $Y_1Ba_2Cu_3O_{9-\delta}$, J. Moreland, J.W. Ekin, L.F. Goodrich, T.E. Capobianco, A.F. Clark, J. Kwo,* M. Hong,* and S.H. Liou,* Physical Review B, Vol. 35, No. 16, pp. 8856-8857 (June 1, 1987). [CEEE/Electromagnetic Technology Division]
*AT&T Bell Laboratories, Murray Hill

Current-voltage tunneling characteristics in a high-critical-temperature superconducting material containing predominantly $Y_1Ba_2Cu_3O_{9-\delta}$ have been measured using the break-junction technique. Sharp gap structure was observed, with the largest superconductive energy gap measured to be $\Delta = 19.5 \pm 1$ meV, assuming a superconductor-insulator-superconductor junction. This energy gap corresponds to $2\Delta/k_BT_c = 4.8$ at $T = 4$ K, for a critical temperature of 93 K (midpoint of the resistive transition).

ELECTRON TUNNELING MEASUREMENTS IN LaSrCuO AND YBaCuO, John Moreland, J.W. Ekin, L.F. Goodrich, T.E. Capobianco, and A.F. Clark, Materials Research Society, Vol. EA-11, pp. 73-75 (June 1987). [CEEE/Electromagnetic Technology Division]

The break junction technique whereby vacuum tunneling occurs within the fracture of a bulk sample is used to study the LaSrCuO and YBaCuO perovskite superconductors. Structure in the

current versus voltage characteristics is reminiscent of previous quasiparticle curves obtained for BCS superconducting materials. Some curves have anomalous qualities, including large dips in the junction conductance with increasing voltage just above a well defined tunneling gap edge, linearly increasing junction conductance with applied bias, along with features occurring near voltage intervals with the 1, 3, 5 pattern.

ELECTRON TUNNELING MEASUREMENTS OF HIGH T_c COMPOUNDS USING BREAK JUNCTIONS, John Moreland, L.F. Goodrich, J.W. Ekin, T.E. Capobianco, and A.F. Clark, Proceedings of the 18th International Conference on Low Temperature Physics, Kyoto, Japan, August 20-26, 1987, Japanese Journal of Applied Physics, Vol. 26, Supplement No. 26-3, pp. 999-1000 (1987). [CEEE/Electromagnetic Technology Division]

We report on the break junction technique and its application to the high T_c superconductors LaSrCuO and YBaCuO. In this technique, bulk samples are fractured and the freshly fractured surfaces adjusted to form a tunneling junction with vacuum or liquid helium as the insulating barrier. Precise mechanical adjustment permits the study of electron tunneling phenomena between pieces of a bulk superconductor. The current voltage characteristics of these break junctions are variable indicating sample inhomogeneity. However, some junction settings result in the more familiar quasiparticle signatures in the current voltage characteristics. Low leakage junctions indicate the presence of a sharp superconductive energy gap as well as large variations in junction conductance above the gap edge in both materials.

ANOMALOUS BEHAVIOR OF TUNNELING CONTACTS IN SUPERCONDUCTING PEROVSKITE STRUCTURES, J. Moreland, L.F. Goodrich, J.W. Ekin, T.E. Capobianco, and A.F. Clark Advances in Cryogenic Engineering Materials, Vol. 34, pp. 625-632 (1988). [CEEE/Electromagnetic Technology Division]

Our break junction results for electron tunneling spectroscopy of the perovskite superconductors La-Sr-Cu-O and Y-Ba-Cu-O are similar to those obtained using thin film, scanning tunneling microscopy, and point contact methods. Energy gap structures are sometimes observed in the measured current voltage characteristics. More often, however, the characteristics are anomalous when compared to previous tunneling studies of BCS superconductors. The anomalies include linearly increasing conductance with voltage, large deviations in junction conductance above the gap edge, and junction diode action. We discuss some possible explanations for these observations.

RECENT TUNNELING MEASUREMENTS OF 90 K SUPERCONDUCTORS AT NBS, John Moreland, J.A. Beall, R.H. Ono, and A.F. Clark, Materials Research Society, Vol. EA14, pp. 351-353 (December 1987). [CEEE/Electromagnetic Technology Division]

Several tunneling measurements on oxide superconductors have been made at NBS in the last year. These include break junction tunneling measurements of the energy gap, break junction superconducting point contacts, and the operation of a break junction point contact rf SQUID above 77 K. Until recently, these tunneling experiments have been limited to bulk samples cut from sintered pellets and a few small single crystals. We present here further results on thin

films of $YBa_2Cu_3O_x$ (YBCO) using squeezable electron tunneling (SET) junctions. In contrast to the break junction tunneling experiments on bulk samples, where quite often tunneling spectra are without energy gap features, the spectra for thin-film SET junctions are rich with structure.

SINGLE CRYSTAL $HoBa_2Cu_3O_x$ BREAK JUNCTIONS, John Moreland, A.F. Clark, M.A. Damento,* and K.A. Gschneider, Jr.,* Proceedings of the International Conference on High Temperature Superconductors - Materials and Mechanisms of Superconductivity, Interlaken, Switzerland, February 29 - March 4, 1988, pp. 1383-1384 (1988). [CEEE/Electromagnetic Technology Division]
*Ames Laboratory, Iowa State University

Tunneling spectra of $HoBa_2Cu_3O_x$ single crystals using the break junction method show energy gap features. These features are variable from junction to junction possibly due to an anisotropic gap function. The I-V curves show the peculiar square law dependence of the current on voltage seen in many tunneling measurements of polycrystalline samples of 90 K superconductors. This may be an indication of an inherent "granularity" built into the superconducting matrix of a single crystal.

EVIDENCE FOR THE SUPERCONDUCTING PROXIMITY EFFECT IN JUNCTIONS BETWEEN THE SURFACES OF YBa_2Cu_3Ox THIN FILMS, J. Moreland, R.H. Ono, J.A. Beall, M. Madden, and A.J. Nelson,* Applied Physics Letters, Vol. 54, No. 15, pp. 1477-1479 (April 10, 1989). [CEEE/Electromagnetic Technology Division]
*Solar Energy Research Institute

We have used the squeezable electron tunneling (SET) junction technique for testing the electrical properties of the surfaces of $YBa_2Cu_3O_x$ (YBCO) thin-film electrodes. As deposited and annealed, the surfaces of the electrodes were insulating at 4 K. Several methods were used to improve the electrical properties of the electrodes' surfaces including rapid thermal annealing, oxygen sputter etching, and thin Ag coating treatments. The greatest improvement occurred after a deposition of a 5 nm Ag coating and subsequent rapid thermal anneal of one set of YBCO films. Under these conditions it was possible to make a superconducting Josephson point contact between the surfaces of the electrodes. We think that the Ag acts as a normal-metal proximity layer effectively shunting the degraded electrodes' surfaces.

BREAK JUNCTIONS I, John Moreland, L.F. Goodrich, J.W. Ekin, T.E. Capobianco, and A.F. Clark, National Bureau of Standards NBSIR 88-3090, 76 p. (May 1988). [CEEE/Electromagnetic Technology Division]

Measurements of the tunneling current-voltage characteristics of break junctions in conventional superconductors can be used to determine their superconducting energy gap as a function of energy. These results agree with junctions in some exotic superconductors, on the other hand, have anomalous current-voltage characteristics compared to BCS predictions. Energy gaps and the Josephson effect measured for the new high T_c materials YBaCuO (T_c = 93 K) and LaSrCuO (T_c = 36K) indicate that the samples are inhomogeneous with varying gap functions depending on the location of the tunneling contact within the break junction fracture. Break junction data for these materials are within the strong coupling limits of BCS theory.

BREAK JUNCTION MEASUREMENT OF THE TUNNELING GAP OF A THALLIUM-BASED HIGH-TEMPERATURE SUPERCONDUCTOR CRYSTAL, J. Moreland, D.S. Ginley,* E.L. Venturini,* and B. Morosin,* Applied Physics Letters., Vol. 55, No. 14, pp. 1463-1465 (October 2, 1989). [CEEE/Electromagnetic Technology Division]
*Sandia National Laboratories

We have used the break junction method to measure the tunneling gap of a thallium-based high-temperature superconductor crystal in liquid helium at 4 K. The crystal was predominantly $Tl_2CaBa_2Cu_2O_7$ and had a Meissner onset at 105 K. Tunneling data showed a symmetric gap about zero bias between two well defined conductance peaks in the conductance-versus-voltage curve. The gap is consistent with a BCS energy gap (Δ) of 30 meV assuming a superconductor-insulator-superconductor electrode configuration. In addition, a supercurrent could be detected when the break junction was operated in a point contact mode at temperatures as high as 95 K.

BREAK JUNCTION TUNNELING SPECTROSCOPY OF SINGLE-CRYSTAL BISMUTH-BASED HIGH-TEMPERATURE SUPERCONDUCTORS, John Moreland, C.K. Chiang, L.J. Swartzendruber, Advances in Cryogenic Engineering (Materials), Vol. 36, Part A, pp. 619-625 (1990). [CEEE/Electromagnetic Technology Division and IMSE/Ceramics and Metallurgy Divisions]

We have measured the tunneling spectra of some high temperature superconducting crystal break junctions at 4 K. The samples were thin plates of $Bi_2SrCa_2Cu_2O_8$ compound. The tunneling spectra (conductance versus voltage) were not typical of BCS superconductor tunneling electrodes. The spectra of higher-resistance break-junction settings ($R > 1$ MΩ) show a tunneling gap on top of a linearly increasing conductance background signal. "Harmonic" dip features in the spectra of lower resistance break junction settings ($R < 1$ MΩ) indicated tunneling between multiple particles in the vicinity of the primary (highest resistance) contact of the junction. The dips occurred at about the same current but shifted in voltage when the resistance of the break junction was continuously adjusted to new settings.

HIGH CRITICAL TEMPERATURE SUPERCONDUCTOR TUNNELING SPECTROSCOPY USING SQUEEZABLE ELECTRON TUNNELING JUNCTIONS, Thomas Walsh, John Moreland, R.H. Ono, J.A. Beall, M. Cromar, T. Harvey, C. Reintsema,* and T.S. Kalkur,* IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 840-843 (March 1991). [EEEL/Electromagnetic Technology Division]
*University of Colorado

We have performed tunneling spectroscopy measurements on squeezable electron tunneling junctions using Bi-Sr-Ca-Cu-O, Y-Ba-Cu-O, and Nb electrodes in a variety of combinations. A zero-bias conductance peak has been seen repeatedly in the current-voltage [I(V)] and the conductance-voltage [G(V)] characteristics. We present a model to explain this conductance peak in terms of quasi-particle tunneling, phase diffusion and a supercurrent. Two additional structures have been seen repeatedly in I(V) and G(V). One of these structures has the characteristics of an energy gap feature. The other structure, which can mimic the gap feature, is explained in terms of the switching to the voltage state of a grain boundary junction that is in series with the SET junction. The dependence of these features upon temperature and the force applied to the junction is examined.

8. JOSEPHSON EFFECT AND DEVICES

JOSEPHSON EFFECT ABOVE 77 K IN A YBaCuO BREAK JUNCTION, John Moreland, L.F. Goodrich, J.W. Ekin, T.E. Capobianco, A.F. Clark, A.I. Braginski,* and A.J. Panson,* Applied Physics Letters, Vol. 51, No. 7, pp. 540-541 (August 17, 1987). [CEEE/Electromagnetic Technology Division]
*Westinghouse Research & Development Center

We have observed the Josephson effect in a YBaCuO break junction. Critical currents as high as 10 mA were measured at 4 K for break junctions with a point contact within the fracture of a sample. The junction was susceptible to microwave radiation showing Shapiro steps with the ratio of V/f of $2.04 \pm 0.05\ \mu$V/GHz compared to the pair tunneling value of h/2e = 2.068 μV/GHz. These steps were clearly visible in the current-voltage characteristics at temperatures up to 85 ± 5 K.

A JOSEPHSON ARRAY VOLTAGE STANDARD AT 10 V, Frances L. Lloyd, C.A. Hamilton, J.A. Beall, Diane Go, R.H. Ono, and R.E. Harris, IEEE Electron Device Letters, Vol. EDL-8, No. 10, pp. 449-450 (October 1987). [CEEE/Electromagnetic Technology Division]

The technology of Josephson voltage standards has been extended to an array of 14 184 junctions which is capable of generating over 150 000 quantized voltage levels spanning the range from -12 to +12 V. This makes possible the direct calibration of 10-V Zener reference standards without the use of a voltage divider.

OPERATION OF A Y-Ba_2-Cu-O_x RF SQUID AT 81K, J.E. Zimmerman, J.A. Beall, M.W. Cromar, and R.H. Ono, Applied Physics Letters, Vol. 51, No. 8, pp. 617-618 (August 24, 1987). [CEEE/Electromagnetic Technology Division]

An rf superconducting quantum interference device (SQUID) has been made from bulk Y-Ba-Cu-O. The device displays quantum interference effects and operates with useful signal levels up to 81 K. The SQUID is formed from a ring of Y-Ba-Cu-O which is broken in the cryogenic environment and then recontacted. Estimates of the SQUID noise performance are given.

EQUIVALENT FLUX NOISE IN $YBa_2Cu_3O_x$ RF SQUID, J.E. Zimmerman, J.A. Beall, M.W. Cromar, and R.H. Ono, Proceedings of the 18th International Conference on Low Temperature Physics, Kyoto, Japan, August 20-26, 1987, Japanese Journal of Applied Physics, Vol. 26, Supplement 26-3, pp. 2125-2126 (1987). [CEEE/Electromagnetic Technology Division]

We have measured the noise in flux-locked rf SQUIDs made of bulk $YBa_2Cu_3O_x$ both in a He cooled cryostat and in liquid nitrogen (LN_2). Our best results at 75 K show a spectral density of the equivalent flux noise equal to $4.5 \times 10^{-4}\phi_0/\sqrt{Hz}$. There is considerable variation in the performance of SQUIDs made from nominally similar material.

SWITCHING NOISE IN $YBa_2Cu_3O_x$ "MACROBRIDGES", R.H. Ono, J.A. Beall, M.W. Cromar, P.M. Mankiewich,* R.W. Howard,* and W. Skocpol,† Proceedings of the 1988 Applied Superconductivity Conference, San Francisco, CA (August, 1988), IEEE Transactions on Magnetics, Vol. MAG-25, No. 2, pp. 976-979 (1989). [CEEE/Electromagnetic Technology Division]

*AT&T Bell Laboratories, Holmdel, NJ.

†Boston University, Boston, MA.

We have observed intermittent switching in the voltage-current characteristics (VIC) of thin film bridges of $YBa_2Cu_3O_x$. At a fixed bias point there are multiple metastable voltage states with lifetimes which depend on the bias current and applied magnetic field. The microbridges are made of thin (< 500 nm), polycrystalline films of $YBa_2Cu_3O_x$ which are patterned by liftoff into structures with dimensions ranging from less than 1 μm to 100 μm. Details of the fabrication process and the measurements are presented. The results are discussed in the context of fluctuations in the effective resistance of the bridge due to motion of trapped flux.

$YBa_2Cu_3O_{7-\delta}$/INSULATOR MULTI-LAYERS FOR CROSSOVER FABRICATION, J.A. Beall, M.W. Cromar, T.E. Harvey, M.E. Johansson, R.H. Ono, C.D. Reintsema, D.A. Rudman, S.E. Asher,* A.J. Nelson,* and A.B. Swartzlander,* Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 1596-1599 (March 1991). [CEEE/Electromagnetic Technology Division]

*Solar Energy Research Institute

The development of thin-film dielectrics compatible with the epitaxial growth of $YBa_2Cu_3O_{7-\delta}$ (YBCO) is crucial to the fabrication of multilayer device and circuit structures. We have investigated the YBCO/$SrTiO_3$ (STO) system by fabricating YBCO/STO bilayers and simple YBCO/STO/YBCO crossover structures. The thin films were deposited *in situ* by pulsed laser deposition and analyzed using x-ray diffraction and scanning electron microscopy. The film interfaces were characterized by secondary ion mass spectrometry (SIMS) depth profiling. We have developed photolithographic and wet-etching processes for patterning the crossovers which are compatible with these materials. The crossover structures were characterized by resistance and insulator pinhole density as well as the superconducting properties of the patterned top and bottom YBCO electrodes (critical temperature, T_c, and critical current density, J_c). Using $SrTiO_3$ as the insulating layer, we have made crossovers with good isolation between layers (> 100 MΩ) and high J_c even in the top electrode (J_c (76 K) > 10^5 A/cm^2).

JOSEPHSON COUNTING ANALOG-TO-DIGITAL CONVERTER, D.L. Miller,* J.X. Przybysz,* J. Kang,* C.A. Hamilton, D.M. Burnell,† Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 2761-2764 (March 1991). [CEEE/Electromagnetic Technology Division]

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A superconductive analog-to-digital converter which uses a dc SQUID as a quantizer and a flip-flop counter as a digitizer has been designed, fabricated, and tested. The circuit was fabricated using a ten-level niobium process. Tests at 4.2 K demonstrated (1) counting to the full twelve-bit accuracy of the design, (2) monotonic A/D conversion with linearity to less than 1 LSB over the

more than nine-bit range of conversion, (3) read-on-the-fly operation with counter overflow, and (4) counter operation with a gate current bus.

FAR-INFRARED KINETIC-INDUCTANCE DETECTORS, E.N. Grossman, D.G. McDonald, and J.E. Sauvageau, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 2677-2680 (March 1991). [CEEE/Electromagnetic Technology Division]

Extremely sensitive far-infrared detectors suitable for both direct detection and heterodyne applications are possible based upon micrometer-sized thin films with thickness less than a superconducting penetration depth. The penetration depth of such a film, and therefore its inductance, varies with temperature and with quasiparticle population (described by an effective temperature T^*), resulting in both bolometric and non-equilibrium "photoinductive" responses. Incident radiation is coupled into the small-area kinetic inductor by a lithographic antenna, and the resulting inductance changes are amplified and converted to a voltage signal by an integrated microstrip DC SQUID. The device is sensitive because, unlike junction-based devices with large capacitive reactances, the kinetic inductor is naturally well matched to the antenna impedance at the far-IR frequency ($\nu > 2\Delta/h$), and to the pre-amplifier (SQUID) impedance at microwave or video frequencies ($\nu << 2\Delta/h$). The best kinetic inductor materials are those with low electronic mean free path, large penetration depth, and high critical current density. Thus, common magnet alloys such as NbTi are the natural choice for liquid-He temperature operation. A detailed analysis predicts a (phonon-limited) NEP of 4×10^{-17} W$\sqrt{\text{Hz}}$ for a bolometer with an iridium kinetic inductor operated at 0.1 K. A heterodyne noise temperature of 2250 K (single-sideband) at 3 THz, with 200 MHz bandwidth, is predicted for a Nb-Ti mixer operated at 4 K.

SUPERCONDUCTING KINETIC INDUCTANCE RADIOMETER, J.E. Sauvageau, D.G. McDonald, and E.N. Grossman, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 2757-2760 (March 1991). [CEEE/Electromagnetic Technology Division]

We are developing a bolometer based on a differential thermometer that senses temperature changes through changes in the kinetic inductance of a superconducting thin film. The temperature transducer is an inductance bridge patterned as an integrated circuit on a 1 cm^2 Si substrate. Two inductors from opposite arms of the bridge are patterned on a 2 mm^2 thermally isolated Si island which is supported by a 9 μm thick Si:B membrane. The bridge is excited with audio frequency current and the bridge imbalance is detected with a commercial DC SQUID amplifier. The bridge is balanced by applying power to the thermally isolated island. This thermometer is the sensor for a prototype radiometer that will provide an absolute measure of IR power. The radiometer, which is designed for a NEP of about 10^{-11} W$\sqrt{\text{Hz}}$, is intended to measure the spectrally dispersed power of a 300 K black body. This absolute radiometer is being developed for use at the Low Background Infrared (LBIR) facility at NIST, Gaithersburg. The noise floor of the temperature transducer for the radiometer has been measured to be 0.7 pW for a 100 s integration time. This is approximately 150 times lower noise than that of the commercial absolute radiometer currently used at the LBIR facility in Gaithersburg.

A SERIES ARRAY OF DC SQUIDS, Richard P. Welty* and John M. Martinis, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 2924-2926 (March 1991). [CEEE/Electromagnetic Technology Division]
*University of Colorado

We have fabricated a series array of 100 dc SQUIDs using trilayer Nb-AlO_x-Nb junctions. The SQUIDs are modulated with a common flux bias line and produce an output voltage swing of several millivolts across the array. The large output voltage will allow direct connection to room temperature electronics without the transformer coupling and resulting frequency limitations commonly associated with dc SQUID amplifiers. We have measured a bandwidth of dc to at least 175 Mhz for a 100-SQUID array. The series array will be used as the output stage for a multistage integrated SQUID amplifier.

TRAPPED VORTICES IN A SUPERCONDUCTING MICROBRIDGE, George S. Park,* Charles E. Cunningham,* Blas Cabrera,* and Martin E. Huber, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 3021-3024 (March 1991). [CEEE/Electromagnetic Technology Division]
*Stanford University

Laser light pulsed onto a Nb microbridge drives it momentarily normal and changes the quantum flux state of a superconducting inductive loop. The flux state is measured by a SQUID coupled to the loop. With a Nd:YAG laser, vortices are never trapped in the microbridge; with a diode laser, vortices are sometimes trapped. The spatial distribution of the trapped flux was studied. The effect of the optical pulse fall time on the frequency of flux trapping was found to be unimportant from 200 ns to 8 ms. Noise spectrum analysis indicates that the laser diode is 5 to 25 times noisier than the Nd:YAG laser at the characteristic frequency of the loop. This noise is believed to be responsible for flux trapping in the microbridge.

CORRELATION OF FLUX STATES GENERATED BY OPTICAL SWITCHING OF A SUPERCONDUCTING CIRCUIT, Charles E. Cunningham,* George S. Park,* Blas Cabrera,* and Martin E. Huber, Physica B, Vols. 165 and 166, pp. 113-114 (1990). [CEEE/Electromagnetic Technology Division]
*Stanford University

We pulse a superconducting microbridge with light, changing the quantum flux state of a superconducting circuit. Long sequences of pulses are used to measure the degree of correlation between successive flux states. In a series of runs, the pulse length was changed over six decades from 6 ns to 10 ms. The correlations fit a simple Fokker-Planck conditional probability model.

EXCESS LOW-FREQUENCY FLUX NOISE IN dc SQUIDS INCORPORATING Nb/Al OXIDE/Nb JOSEPHSON JUNCTIONS, Martin E. Huber and Michael W. Cromar, Physica B, Vols. 165 and 166, pp. 77-78 (1990). [CEEE/Electromagnetic Technology Division]

We have fabricated thin-film dc SQUIDs (Superconducting QUantum Interference Devices) incorporating Nb/Al-Oxide/Nb tunnel junctions. The spectral density of the voltage noise, S_v, of stripline SQUIDs is characterized between 1 Hz and 2000 Hz. In this frequency range, S_v is proportional to the square of the responsivity ($\partial V/\partial \Phi$) over a significant range of bias conditions with an unusual frequency dependence. In a 7 pH SQUID, the spectral density of the flux noise, S_Φ, at 1 Hz is less than 10^{-11} Φ_0^2/Hz, where $\Phi_0 \equiv h/2e$. The observed noise does not appear to be environmental; also, it is independent of the value of the junction shunt resistance and whether the stripline material is a PbIn alloy or Nb. Subject to the constraint of a constant product of the junction area, critical current density, and SQUID self-inductance in the SQUIDs studied, S_Φ is inversely proportional to the junction area.

OBSERVATION OF QUANTUM SUSCEPTANCE IN SUPERCONDUCTING TUNNEL JUNCTIONS, Qing Hu,* C.A. Mears,* P.L. Richards,* and F.L. Lloyd, Physica B, Vols. 165 and 166, pp. 1587-1588 (1990). [CEEE/Electromagnetic Technology Division]
*Lawrence Berkeley Laboratory

We have made the first direct measurement of the quantum susceptance which arises from the reactive part of quasiparticle tunneling in a superconductor-insulator-superconductor (SIS) junction. The resonant frequency of a resonator which contains an SIS junction depends on the dc bias due to the bias dependence of the quantum susceptance. The observed 19% shift of the resonant frequency, from 73 GHz to 87 GHz, is in excellent agreement with Werthamer-Tucker theory.

OBSERVATION OF NONDISPERSIVE QUASIPARTICLE TUNNEL CURRENTS IN SUPERCONDUCTING TUNNEL JUNCTIONS, Qing Hu,* C.A. Mears,* P.L. Richards,* and F.L. Lloyd, Physical Review Letters, Vol. 64, pp. 2945-2948 (June 11, 1990). [CEEE/Electromagnetic Technology Division]
*Lawrence Berkeley Laboratory

We have made the first direct measurement of the quantum susceptance which arises from the nondisipative part of the quasiparticle tunneling in a superconductor-insulator-superconductor (SIS) junction. The resonant frequency of a resonator which contains an SIS junction depends on the dc bias due to the bias dependence of the quantum susceptance. The observed 19% shift of the resonant frequency, from 73 to 87 GHz, is in excellent agreement with the Werthamer-Tucker theory. This quantum susceptance should exist in other devices such as quantum-well structures.

QUANTUM SUSCEPTANCE AND ITS EFFECTS ON THE HIGH-FREQUENCY RESPONSE OF SUPERCONDUCTING TUNNEL JUNCTIONS, Qing Hu,* C.A. Mears,* P.L. Richards,* and F.L. Lloyd, Physical Review B, Vol. 42, No. 16, pp. 10,250-10,263 (1990). [CEEE/Electromagnetic Technology Division]
*Lawrence Berkeley Laboratory

We have made the first direct measurement of the quantum susceptance that arises from the nondissipative part of quasiparticle tunneling in a superconductor-insulator-superconductor tunnel junction. The junction is coupled to an antenna and a superconducting microstrip stub to form a resonator; the resonant frequency is determined from the response of the junction to broadband radiation from a Fourier-transform spectrometer. A 19% shift of the resonant frequency, from 73 to 87 GHz, is observed, which arises from the change of the quantum susceptance of the junction with dc bias voltage. This shift is in excellent agreement with calculations based on the Werthamer-Tucker theory, which includes the quantum susceptance. We also demonstrate that it is essential to include the quantum susceptance in our theoretical computation to explain the photon-assisted-tunneling steps, which have negative dynamic conductance. Such steps are observed when the junction is pumped at slightly below the resonant frequency of the capacitor and the stub. The quantum susceptance should exist in all tunnel devices whose nonlinear I-V characteristics are due to elastic tunneling.

A PHASE SENSITIVE SIS MIXER TO CIRCUMVENT THE QUANTUM LIMIT, Mark F. Bocko,* Zhao-nan Zhang,* Stephen Martinet,* and Michael W. Cromar, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 3391-3394 (March 1991). [CEEE/Electromagnetic Technology Division]
*University of Rochester, New York

A conventional SIS mixer, which is pumped by a single frequency local oscillator (LO) functions as a phase-insensitive linear amplifier and may achieve a noise level near the "quantum limit" of one-half photon added noise per unit bandwidth. A phase sensitive linear amplifier may have less noise than this "quantum limit." We are attempting to beat the quantum noise limit with an SIS mixer by employing a two-LO technique which makes the mixer's gain dependent upon the phase of the incoming signal. We have experimentally demonstrated the phase sensitive gain of our two-LO mixer; the gain variation from maximum to minimum is more than 20 dB. We also present theoretical predictions of the noise of the two-LO mixer.

9. MAGNETIC MEASUREMENTS

AC SUSCEPTIBILITY MEASUREMENTS NEAR THE CRITICAL TEMPERATURE OF A Y-Ba-Cu-O SUPERCONDUCTOR, R.B. Goldfarb, A.F. Clark, A.J. Panson,* and A.I. Braginski,* Materials Research Society, Vol. EA-11, pp. 261-263 (June 1987). [CEEE/Electromagnetic Technology Division]
*Westinghouse Research & Development Center

The loss component of complex susceptibility of a Y-Ba-Cu-O superconductor near its critical temperature is strongly dependent on ac field amplitude but virtually independent of frequency. This implies that magnetic hysteresis is the major loss mechanism in these materials. The temperature at which the loss first becomes positive upon warming corresponds to an equivalence between the amplitude of the ac field and the lower critical field of the superconductor.

EVIDENCE FOR TWO SUPERCONDUCTING COMPONENTS IN OXYGEN-ANNEALED SINGLE-PHASE Y-Ba-Cu-O, R.B. Goldfarb, A.F. Clark, A.I. Braginski,* and A.J. Panson,* Cryogenics, Vol. 27, p. 475-480 (September 1987). [CEEE/Electromagnetic Technology Division]
*Westinghouse Research & Development Center

The complex susceptibility of a sintered Y-Ba-Cu-O superconductor is strongly dependent on a.c. field amplitude, h. Very small values of h must be used for the real part of susceptibility, χ', to reach a value corresponding to bulk diamagnetism just below the critical temperature, T_c. The imaginary part, χ'', represents hysteresis loss in the sample. Thus, χ'' versus temperature becomes positive when h exceeds the lower critical field, H_{c1}, of the superconductor.

Annealing the material in oxygen gives rise to two distinct components, a relatively high-T_c, high H_{c1} superconductor (denoted as "G" or "good") and a relatively low-T_c, low H_{c1} superconductor (denoted as "B" or "bad"). Curves of susceptibility versus increasing temperature reflect the dual nature of the annealed sample: χ' has an inflection point at T_c of the B component and approaches zero at T_c of the G component, while χ'' has a peak at each T_c. Both critical temperatures decrease linearly with increasing h, though at very different rates. H_{c1} of the G component is considerably greater than H_{c1} of the B component. The lower critical fields are linearly decreasing functions of temperature.

Two models might explain the susceptibility data. In the grain model, the G component consists of superconducting grains and the B component is either intergranular material, unfavourably orientated anisotropic grains, or oxygen-depleted grain boundaries. In the surface model, the G component is in the interior of the sample and the B component is at the sample's surface. This condition could arise if there was oxygen depletion at the surface subsequent to total enrichment during annealing.

MAGNETIC SUSCEPTIBILITY OF SINTERED AND POWDERED Y-Ba-Cu-O, D.-X. Chen,* R.B. Goldfarb, J. Nogues,* and K.V. Rao,* Journal of Applied Physics, Vol. 63, No. 3, pp. 980-983 (February 1, 1988). [CEEE/Electromagnetic Technology Division]
*Royal Institute of Technology, Sweden

The real and imaginary parts of ac susceptibility of a sintered $Y_1Ba_2Cu_3O_{7-\delta}$ superconductor were measured before and after powdering. The temperature-dependent susceptibility may be separated into two contributions, one sensitive and the other relatively insensitive to the magnitude of the measuring field. The former is partially suppressed by coarsely crushing the sample. It is completely suppressed after finely powdering, whereupon the susceptibility curves become insensitive to the magnitude of the measuring field. Several models apparently consistent with the results are discussed.

MAGNETIC EVALUATION OF Cu-Mn MATRIX MATERIAL FOR FINE FILAMENT Nb-Ti SUPERCONDUCTORS, R.B. Goldfarb, D.L. Ried, T.S. Kreilick,* and E. Gregory,* IEEE Transactions on Magnetics, MAG-25, p. 1953 (1989). [CEEE/ Electromagnetic Technology Division and IMSE/Fracture and Deformation Division]
*Supercon, Inc.

Copper-manganese alloys have been proposed as matrix material for the reduction of coupling losses in fine-filament Nb-Ti superconductor wires. Magnetization and susceptibility measurements show that adverse magnetic effects arising from the spin-glass properties of this matrix are minimal for concentrations of Mn up to at least 4 percent.

BEAN MODEL EXTENDED TO MAGNETIZATION JUMPS, R.L. Peterson, Physics Letters A, Vol. 131, No. 2, pp. 131-134 (August 8, 1988). [CEEE/Electromagnetic Technology Division]

I extend the phenomenological Bean model of magnetization in hard superconductors to include the trains of magnetization jumps seen at low temperature in moderate-to-high magnetic fields. As in the original Bean model, no particular mechanisms for flux pinning are invoked. The extended model correctly accounts for the general dependence of the size of the magnetization jumps on sample size and critical current density. The data together with the model show that the shielding fields are approximately equal after each jump.

SUPERCONDUCTING PROPERTIES OF MELT-CAST Bi-Sr-Ca-Cu-O (ABSTRACT), R.B. Goldfarb, T.F. Ciszek,* and C.D. Evans,* Journal of Applied Physics, Vol. 64, No. 10, pp. 5914 (15 November 1988). [CEEE/Electromagnetic Technology Division]
*Solar Energy Research Institute

Sintered solid copper-oxide superconductors generally have an intergranular coupling component that is evident in curves of ac susceptibility versus temperature: a broad peak in the imaginary (loss) part, χ'', and a corresponding inflection in the real part, χ'. Polycrystalline Bi-Sr-Ca-Cu-O samples have been prepared from the melt. They are very different from typical sintered materials. Some have a single superconducting phase, while others have two superconducting phases. Most of the superconducting platelets are partially surrounded by nonsuperconducting material. An unusual feature of the susceptibility curves is that they show almost no coupling losses. This might be explained if the superconducting grains were isolated from each other, as is the case in susceptibility measurements of powdered superconductors. However, the characteristics of the melt-cast samples differ from those of powdered samples in three ways. (1) The resistivity goes to zero at low temperatures, which indicates some connectivity between

crystallites. (2) χ' approaches -1 at low temperature, indicating substantial bulk shielding. (3) χ' decreases in magnitude with increasing field at low temperature, an effect not seen in powdered samples. We therefore believe that there is substantial superconductor grain-to-grain coupling, even though the susceptibility curves show little evidence of the usual intergranular coupling losses. We conclude that the coupling component in these melt-cast samples is virtually lossless.

KIM MODEL FOR MAGNETIZATION OF TYPE-II SUPERCONDUCTORS, D.-X. Chen and R.B. Goldfarb, Journal of Applied Physics, Vol. 66, No. 6, pp. 2489-2500 (September 1989). [CEEE/Electromagnetic Technology Division]

We have calculated the initial magnetization curves and complete hysteresis loops for hard type-II superconductors. The critical-current density, J_c, is assumed to be a function of the internal magnetic field, H_i, according to Kim's model, $J_c(H_i) = k/(H_0 + |H_i|)$, where k and H_0 are constants. Our analytic solution is for an infinite specimen with finite rectangular cross section, $2a \times 2b$ ($a \leq b$), in which a uniform field, H, is applied parallel to the infinite axis. The calculated curves are functions of b/a, a dimensionless parameter $p = (2ka)^{1/2}/H_0$ and the maximum applied field. The full-penetration field is $H_p = H_0[(1+p^2)^{1/2}-1]$. The equations for an infinite cylindrical specimen of radius a are the same as those for a specimen with square cross section, $a = b$. In the limit $p << 1$ and $a = b$, our results reduce to those of the Bean model (J_c independent of H_i) for cylindrical geometry. Similarly, in the limit $p << 1$ and $b \rightarrow \infty$, the results are the same as those for a slab in the Bean model. For $H > 1.5\ H_p$, or $H > 0$ when $p << 1$, the width of the hysteresis loop ΔM may be used to deduce J_c as a function of H: $J_c(H) = \Delta M(H)/[a(1-a/3b)]$.

FLUX CREEP AND ACTIVATION ENERGIES AT THE GRAIN BOUNDARIES OF Y-Ba-Cu-O SUPERCONDUCTORS, M. Nikolo and R.B. Goldfarb, Physical Review B, Vol. 39, p. 6615 (1989). [CEEE/Electromagnetic Technology Division]

We measured the ac susceptibility of sintered $Y_1Ba_2Cu_3O_{7-\delta}$ pellets as a function of temperature and ac magnetic field amplitude and frequency. The imaginary part of the susceptibility, χ'', exhibits two peaks. A narrow peak is located at the critical temperature of the grains. A broad peak at lower temperature is attributed to hysteresis losses at the grain boundaries. There is a small shift in this coupling peak to higher temperature as the frequency increases from 10 to 1000 Hz. We explain the shift in terms of Anderson flux creep on a time scale of milliseconds. The shift depends on the amplitude of the measuring field. The activation energy for flux creep ranges from 11.9 ± 1.0 eV in the zero-field limit [0.8 $A \cdot m^{-1}$ (0.01 Oe)] to 1.2 ± 0.3 eV at 800 $A \cdot m^{-1}$ (10 Oe). We extrapolate our data to find the value for an intergrain decoupling field of 1-2 $kA \cdot m^{-1}$ (13-25 Oe), above which flux creep presumably becomes flux flow at the grain boundaries.

MAGNETIZATION OF IMPERFECT SUPERCONDUCTING GRAINS, R.L. Peterson, Physical Review B, Vol. 40, No. 4 (August 1, 1989). [CEEE/Electromagnetic Technology Division]

A critical-state theory of the magnetization of superconducting grains containing nonsuperconducting regions is presented which shows that the thickness of the sheath of

supercurrents around these regions can be more important than the grain dimension in determining the magnetization. This may explain some apparently conflicting results on the magnetization of high-T_c powders of different sizes.

MAGNETIC FIELD DEPENDENCE OF THE SUPERCONDUCTIVITY IN Bi-Sr-Ca-Cu-O SUPERCONDUCTORS, F.J. Adrian,* J. Bohandy,* B.F. Kim,* K. Moorjani,* J.S. Wallace, R.D. Shull, L.J. Swartzendruber, and L.H. Bennett, Physica C, Vol. 156, pp. 184-188 (1988). [IMSE/Ceramics Division and Metallurgy Division]

*The Johns Hopkins University, Applied Physics Laboratory

The changes in superconducting behavior of Bi-Sr-Ca-Cu-O and Y-Ba-Cu-O high temperature superconductors upon the application of an applied field of 0.5 T are explored, using field-modulated-microwave absorption. Significant differences in the behavior are noted and discussed.

CONSTRICTED DIAMAGNETIC HYSTERESIS LOOPS OBSERVED FOR THE HIGH T_c SUPERCONDUCTORS, U. Atzmony, R.D. Shull, C.K. Chiang, L.J. Swartzendruber, L.H. Bennett, and R.E. Watson,* Journal of Applied Physics, Vol. 63, No. 8, pp. 4179-4181 (April 15, 1988). [IMSE/Ceramics Division and Metallurgy Division]

*Brookhaven National Laboratory

Magnetic hysteresis loops, ac susceptibility, and resistivity measurements have been made on a $Ba_2YCu_3O_{7-x}$-type high-T_c superconductor. The shape of the hysteresis loops well below T_c are reminiscent of constricted hysteresis loops observed in certain ferromagnetic materials which are usually associated with magnetic aftereffects. Similar dynamic effects, with a time constant on the order of 10 s at 40 K, are shown to be present in the superconducting material. This dynamic magnetic viscosity effect is in addition to the flux creep observed for longer time periods.

MAGNETIC PROPERTIES OF A CHEMICALLY SYNTHESIZED Bi(Pb)SrCaCuO SUPERCONDUCTOR, D.R. Lundy, J.J. Ritter, L.J. Swartzendruber, R.D. Shull, and L.H. Bennett, Proceedings of High T_c Superconductors: Magnetic Interactions, Gaithersburg, MD, October 11-13, 1988, Progress in High Temperature Superconductivity, Vol. 17, pp. 263-268 (1989. [IMSE/Ceramics Division and Metallurgy Division]

It has been reported that the presence of lead serves to increase the fraction of high temperature phase in the Bi-Sr-Ca-Cu-O system prepared by a solid state reaction. To test if this is also the case for material prepared by a chemical method, a lead containing bismuth superconductor ($Bi_{1.5}Pb_{0.5}Sr_{1.5}Ca_{1.75}Cu_2O_x$) was chemically synthesized and its magnetic properties measured. The material obtained contained a large fraction ($\simeq 50\%$) of a phase with a superconducting onset temperature near 110 K. Inspection of the AC susceptibility with a small applied transverse magnetic field indicated the presence of additional superconducting phases with lower onset temperatures. Flux depinning was found to occur at relatively low fields.

MAGNETIC LEVITATION BY ATTRACTION IN SOME HIGH T_c SUPERCONDUCTORS, R.D. Shull, L.J. Swartzendruber, C.K. Chiang, M.K. Wu,* P.N. Peters,† and C.Y. Huang,‡ Proceedings of High T_c Superconductors: Magnetic Interactions, Gaithersburg, MD, October 11-13, 1988, Progress in High Temperature Superconductivity, Vol. 17, pp. 328-339 (1989). [IMSE/Ceramics Division and Metallurgy Division]

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Superconducting composites of AgO and $YBa_2Cu_3O_{7-x}$ are found to possess the unusual property at 77 K of being able to levitate by "attraction" (with the composite being suspended in space "below" a permanent magnet). At the same temperature, however, these composites are still able to levitate a magnet above it by means of the better known method of repulsion. Magnetization measurements as a function of field for several composites of AgO and $YBa_2Cu_3O_{7-x}$ are presented to explain the origin and composition dependence of the novel "attractive" force. These data support the previous conclusion that the ability of the material to be suspended below a magnet is dependent upon the degree of magnetic flux trapping in the superconductor and that the ability to obtain a stable equilibrium position depends on the shape of the hysteresis loop.

MEASUREMENT OF MAGNETIC PROPERTIES IN A MELT CAST Bi-Ca-Sr-Cu-O SUPERCONDUCTOR, L.J. Swartzendruber, L.H. Bennett, and C.F. Gallo,* Proceedings of High T_c Superconductors: Magnetic Interactions, Gaithersburg, MD, October 11-13, 1988, Progress in High Temperature Superconductivity, Vol. 17, pp. 303-308 (1989). [IMSE/Ceramics Division and Metallurgy Division]

*Superconix, Inc.

Magnetic measurements were made to examine the superconducting properties of a crystalline chunk, consisting of many small crystals, prepared by casting from the oxide melt with a starting composition of $Bi_3Ca_2Sr_2Cu_3O_x$. AC susceptibility revealed an onset temperature of 82 K and a transition width of $\approx$ 10 K. Hysteresis loops at 70 K showed a very small hysteresis, indicating a low density of effective flux pinning sites at this temperature, similar to the behavior observed for some superconducting samples of the Bi-Ca-Sr-Cu-O system prepared by other techniques. However, the magnetization at 10 K showed no hysteresis for fields greater than $\approx$3.5 kOe, a much smaller value than we have previously observed for other samples of this material regardless of preparation method, or for any other material with a T_c greater than 70 K. This critical field for flux depinning, H_{cp}, varies with temperature as $H_{cp} = 4980(1-t)^{2.5}$, where t is T/T_c.

MAGNETIC MEASUREMENTS ON POLYMER-HIGH T_c SUPERCONDUCTOR COMPOSITES, A.S. De Reggi, C.K. Chiang, L. Swartzendruber, and G.T. Davis, Proceedings of High T_c Superconductors: Magnetic Interactions, Gaithersburg, MD, October 11-13, 1988, Progress in High Temperature Superconductivity, Vol. 17, pp. 263-268 (1989). [IMSE/Metallurgy Division]

Composite materials made by dispersing powdered mixed-oxide superconductors in suitable polymers may become important new materials in superconducting applications where contact from one superconducting grain to another is not essential to the function, such as magnetic

shielding. The principal advantage of the composite over the undiluted superconductor so that it can be processed and fabricated like a polymer using existing polymer processing equipment and methods. Another advantage of the composite is that the polymer, if properly chosen, can protect the surface of the powder grains from exposure to environmental chemicals which might affect the oxides and degrade its superconducting properties.

In this study, composite materials of the 1-2-3 mixed oxide superconductor $Y_1Ba_2Cu_3O_{7-x}$ (BYCO), with $x < 0.1$ and critical temperature $T_c \approx 92$ K, and the vinylidene fluoride polymer, PVDF, with a glass transition temperature ≈ 233 K, were prepared with volume concentrations ranging from 5 to 25% superconductor. Magnetic data were obtained with an ac susceptometer and a vibrating sample magnetometer. Permeability data in the dilute superconductor range and compared to the predictions of a model analogous to the Lorentz model for dielectrics.

MAGNETIZATION OF ANISOTROPIC SUPERCONDUCTING GRAINS, R.L. Peterson, Journal of Applied Physics, Vol. 67, pp. 6930-6933 (1990). [CEEE/Electromagnetic Technology Division]

A critical-state calculation of the magnetization of hard type-II superconducting grains having anisotropic critical current densities is given. The grains are assumed to present rectangular cross sections to an applied magnetic field. The analysis shows how the critical current densities should be inferred from magnetization measurements for various grain dimensions. For grains in the form of platelets, the hysteresis changes with grain size. However for very elongated grains with anisotropic critical currents, such as may be found in the high-temperature superconductors, the magnetic hysteresis is insensitive to the lengths of the grains, and hence to powdering.

AC LOSS MEASUREMENTS OF TWO MULTIFILMENTARY NbTi COMPOSITE STRANDS, E.W. Collings,* K.R. Marken, Jr.,* M.D. Sumption,* Ronald B. Goldfarb, and Robert J. Loughran, Advances in Cryogenic Engineering (Materials), Vol. 36, R.P. Reed and F.R. Fickett, eds., New York: Plenum Press, pp. 169-176 (1990). [CEEE/Electromagnetic Technology Division]
*Battelle Memorial Institute

As part of an interlaboratory comparative testing program conducted in support of the Versailles Agreement on Advanced Materials and Standards (VAMAS), transverse-field DC hysteresis loss measurements were made at liquid-helium temperatures at fields of up to 3 T (30 kG) on two samples of multifilamentary NbTi composite. The strands differed widely in filament number, were comparable in filament diameter, and one of them was provided with a Cu-Ni barrier between the filaments. The results have been analyzed, and magnetically deduced critical current density values obtained (for comparison with directly measured transport data) using various standard techniques. Based on these studies, a figure-of-merit for AC loss is recommended. The Cu-matrix strand, with its interfilamentary spacing of less than 1 μm, exhibited pronounced proximity-effect-induced coupling losses; this was not observed in the mixed-matrix strand which possessed not only a Cu-Ni barrier but also an interfilamentary spacing of typically 4 μm.

MAGNETIC CHARACTERISTICS AND MEASUREMENTS OF FILAMENTARY Nb-Ti WIRE FOR THE SUPERCONDUCTING SUPER COLLIDER, R.B. Goldfarb and R.L. Spomer, International Cryogenic Materials Conference, Los Angeles, California, July 24-28, 1989, Paper FX-02, Advances in Cryogenic Engineering (Materials), Vol. 36, pp. 215-222 (1990). [CEEE/Electromagnetic Technology Division]

In synchrotron accelerator applications, such as the Superconducting Super Collider (SSC), superconducting magnets are cycled in magnetic field. Desirable properties of the magnets include field uniformity, field stability with time, small residual field, and fairly small energy losses upon cycling. This paper discusses potential sources of problems in achieving these goals, describes important magnetic characteristics to be considered, and reviews measurement techniques for magnetic evaluation of candidate SSC wires. Instrumentation that might be practical for use in a wire-fabrication environment is described. We report on magnetic measurements of prototype SSC wires and cables and speculate on causes for instability in multipole fields of dipole magnets constructed with such cables.

MAGNETORESISTANCE OF MULTIFILAMENT Al/Al-ALLOY CONDUCTORS, C.A. Thompson and F.R. Fickett, Advances in Cryogenic Engineering (Materials), Vol. 36, Part A, pp. 663-669 (1990). [CEEE/Electromagnetic Technology Division]

Previously we have shown that composite monofilament conductors consisting of very pure aluminum confined in an Al-Fe-Ce alloy sheath show an anomalously high magnetoresistance compared to pure aluminum. Some monofilament conductors showed values of $\Delta R/R$ in excess of 50 at 4 K in fields of 10 T, whereas pure aluminum values are usually an order of magnitude smaller. Concerns that similar anomalous behavior might occur in multifilament wires of the same materials prompted this study. Multifilamentary conductors with pure aluminum filaments contained in an Al-Fe-Ce matrix have been investigated.

ANOMALOUS MAGNETORESISTANCE IN Al/Al-ALLOY COMPOSITE CONDUCTORS, F.R. Fickett and C.A. Thompson, Advances in Cryogenic Engineering (Materials), Vol. 36, Part A, pp. 671-678 (1990). [CEEE/Electromagnetic Technology Division]

The transverse magnetoresistance of several composite conductors containing a large single filament of pure aluminum in a matrix of Al-Fe-Ce has been measured at 4 K to fields of 10 T. The magnetoresistance ($\Delta R/R$) of the composite is very large, rising to 55 in the "worst" case. Previous measurements on pure polycrystalline aluminum have always shown a rapidly saturating behavior with a very small linear component; $\Delta R/R$ rarely exceeds a value of 5-6. In addition, the magnetoresistance of the composite samples shows a structure as the field is rotated around the current axis.

TUNNELING STABILIZED MAGNETIC FORCE MICROSCOPY: PROSPECTS FOR LOW TEMPERATURE APPLICATIONS TO SUPERCONDUCTORS, John Moreland and Paul Rice, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 1198-1201 (March 1991). [CEEE/Electromagnetic Technology Division]

We have recently demonstrated an imaging technique referred to as tunneling stabilized magnetic force microscopy or TSMFM. TSMFM is performed using a scanning tunneling microscopy (STM) with a flexible, magnetic, tunneling tip in place of the usual rigid tunneling tip. TSMFM images are therefore combinations of topography and the magnetic forces between the tip and the sample. Room temperature TSMFM images of magnetic bit tracks on a hard disk have 100 nm resolution and are comparable to Bitter patterns made using a ferrofluid. We have built a low temperature STM with the hope of getting TSMFM images of the flux lattice in superconductors. Preliminary TSMFM images of a $YBa_2Cu_3O_x$ (YBCO) film (T_c = 88 K) in a 50 mT field show that relatively large magnetic forces are acting on the flexible tip while scanning at 48 K.

DIRECT EVIDENCE FOR AN EFFECT OF TWIN BOUNDARIES ON FLUX PINNING IN SINGLE-CRYSTAL $YBa_2Cu_3O_{6-x}$, L.J. Swartzendruber, A. Roitburd, D.L. Kaiser, F.W. Gayle, and L.H. Bennett, Physical Review Letters, Vol. 64, No. 4, pp. 483-486 (January 22, 1990). [IMSE/Metallurgy Division]

Magnetization measurements are reported on a nearly cubic single crystal of $YBa_2Cu_3O_{6-x}$ which displays predominantly one variant of twin boundary. With this crystal, we were able to compare the effects of different vortex and twin-boundary orientations on magnetic behavior. The results demonstrate that there is a measurable effect due to direct pinning of vortices by twin boundaries for applied fields perpendicular to the *c* axis. We also show that the large anisotropy in flux pinning for applied fields parallel and perpendicular to the *c* axis is not due to pinning by twin boundaries.

TEMPERATURE DEPENDENCE OF VORTEX-TWIN-BOUNDARY INTERACTION IN $YBa_2Cu_3O_{6-x}$, A. Roitburd, L.J. Swartzendruber, D.L. Kaiser, F.W. Gayle, and L.H. Bennett, Physical Review Letters, Vol. 64, No. 24, p. 2962 (June 11, 1990). [IMSE/Metallurgy Division]

Two recent Letters have reported studies of flux-vortex pinning by twin boundaries in single crystals of the high-temperature superconductor $YBa_2Cu_3O_{6-x}$ (with x near 1) containing predominantly one twin-boundary (TB) orientation. Although both investigations report a measurable effect of TB's on flux pinning, they present opposite conclusions concerning the nature of the vortex-TB interactions. One investigation showed by magnetization measurements that flux-vortex pinning at 10 K was greater when the applied magnetic field (H) was perpendicular to the TBs (compared with parallel or at 45° to the TBs). In contrast, the other investigation studied resistivity near the superconducting transition temperature ($\sim$93 K) and demonstrated that pinning was greatest with the field parallel to the twin planes (compared to the full range of H-TB orientations). We present evidence here that both results are correct, with the apparent contradiction arising entirely from the different temperatures employed in the two investigations.

HALL PROBE MAGNETOMETER FOR SSC MAGNET CABLES: EFFECT OF TRANSPORT CURRENT ON MAGNETIZATION AND FLUX CREEP, R.W. Cross and R.B. Goldfarb, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 1796-1798 (March 1991). [CEEE/Electromagnetic Technology Division]

We constructed a Hall probe magnetometer to measure the magnetization hysteresis loops of Superconducting Super Collider magnet cables. The instrument uses two Hall-effect field sensors to measure the applied field H and the magnetic induction B. Magnetization M is calculated from the difference of the two quantities. The Hall probes are centered coaxially in the bore of a superconducting solenoid with the B probe against the sample's broad surface. An alternative probe arrangement, in which M is measured directly, aligns the sample probe parallel to the field. We measured M as a function of H and field cycle rate both with and without a dc transport current. Flux creep as a function of current was measured from the dependence of ac loss on the cycling rate and from the decay of magnetization with time. Transport currents up to 20% of the critical current have minimal effect on magnetization and flux creep.

10. PHASE EQUILIBRIUM

PHASE EQUILIBRIA AND CRYSTAL CHEMISTRY IN THE SYSTEM Ba-Y-Cu-O, R.S. Roth, K.L. Davis, and J.R. Dennis, Advanced Ceramic Materials: Ceramic Superconductors, Vol. 2, No. 3B, pp. 303-312 (July 15, 1987). [IMSE/Ceramics Division]

Preliminary phase equilibria diagrams are constructed for the "binary" systems BaO-½Y_2O_3, BaO-CuO_x, ½Y_2O_3-CuO_x, and the "ternary" system BaO-½Y_2O_3-CuO_x. Some melting data are used to outline an approximate primary field for the phase responsible for high temperature superconductivity in this system, $Ba_2YCu_3O_{7-x}$. A discussion is given of the crystal chemistry of the phase(s) near the composition $Ba_3YCu_2O_x$.

PHASE EQUILIBRIA IN THE SYSTEM Ba-Y-Cu-O-CO_2 IN AIR, R.S. Roth, C.J. Rawn, F. Beech, J.D. Whitler, J.O. Anderson,* Ceramic Superconductors II Research Update, Man F. Yan, Editor, Waterville, Ohio: The American Ceramics Society, Inc., pp. 13-26 (1988). [IMSE/Ceramics Division and Reactor Radiation Division]

*NBS American Ceramics Society Research Association

Phase equilibria studies in the Ba-Y-Cu-O system have continued, to determine the thermodynamic relations and the primary phase field of $Ba_2YCu_3O_{6+x}$. The high Cu-O containing liquid quenches as another phase with a characteristic x-ray diffraction pattern dependent on the temperature of the heat treatment. The compound previously described as 3BaO:1/2Y_2O_3:2CuO which is a tetragonal pervoskite type phase with a = 2a' and c = 2a' is now shown to exist as a solid solution region between, but not including, the 3:1:2, 4:1:2 and 5:1:3 compositions. A series of completely single phase specimens can be prepared at 950°C with the formula $Ba_{3.2}Y_{0.8}Cu_{2-x}O_{6.4-x}$ ($0.1 < x\ 0.4$). This phase does not form at all when prepared with BaO_2 in a pure oxygen atmosphere and it is concluded that the compound is chemically and structurally an oxycarbonate. The superconducting compound $Ba_2YCu_3O_{6+x}$ was found to melt in air through a four phase field with BaY_2CuO_5, $BaCuO_2$ and liquid gradually increasing in amounts from about 975° to the incongruent melting point at 1002° ± 2°C. As four phases cannot coexist in equilibrium, through a temperature range, in the system BaO:Y_2O_3:CuO_x a fourth component, CO_2, is required to explain the experimental melting data.

PHASE EQUILIBRIA IN PORTIONS OF THE SYSTEM SrO-CaO-Bi_2O_3-CuO, PART 1 - THE SYSTEM SrO-CaO-CuO, R.S. Roth, C.J. Rawn, J.J. Ritter, and B. Burton, Journal of the American Ceramic Society, Vol. 72, No. 8, pp. 1545-1549 (1989). [IMSE/Ceramics Division]

A new phase, probably $CaCuO_2$, is stable only below ≃740°C, and it has an orthorhombic average subcell: Cmca, $\underline{a}$ = 10.588(1), $\underline{b}$ = 2.8122(4), $\underline{c}$ = 6.3245(6) x 0.1 nm. Very weak superstructure reflections indicate two distinct supercells: an orthorhombic cell with the $\underline{b}$ axis multiplied by 10, and a monoclinic cell, C2/c or equivalent, with $\underline{a}_{mon}$ = 10.946 ≃ $\underline{a}$, $\underline{b}_{mon}$ = 6.345 ≃ $\underline{c}_{mon}$ = 2.8100 x 12 = 33.72 x 0.1 nm ≃ 12$\underline{c}$, and β = 105.5°. At 950°C the ternary SrO-CaO-CuO system has extensive regions of solid solution: complete solution is observed for the 2:1 series $(Sr,Ca)_2CuO_3$; the 1:1 series $(Sr,Ca)CuO_2$ accepts Ca up to ≃$Sr_{0.25}Ca_{0.75}O_2$; and the 14:24 series "$Sr_{14}Cu_{24}O_{41}$" extends to at least "$Sr_7Ca_7Cu_{24}O_{41}$". A new ternary phase is reported, and it has a very narrow range of homogeneity $(Ca_{1-x}Sr_x)CuO_2$ with

$x \simeq 0.15$. The composition $(Ca_{0.84}Sr_{0.16})CuO_2$ has a primitive tetragonal unit cell with a = 3.867(2) and c = 3.2195(2) x 0.1 nm. Another solid solution series is stable only above $\simeq 1000°C$. It extends from the SrO-CuO binary into the ternary and is probably $(Sr_{1-x}Ca_x)_2CuO_3$. The composition $(Sr_{1.0}Ca_{1.0})CuO_3$ is orthorhombic Immm with a = 3.306, b = 12.319, c = 3.807 x 0.1 nm.

PHASE EQUILIBRIA AND CRYSTAL CHEMISTRY IN THE QUATERNARY SYSTEM Ba-Sr-Y-Cu-O IN AIR, R.S. Roth, C.J. Rawn, J.D. Whitler, C.K. Chiang, and W. Wong-Ng, Journal of the American Ceramic Society, Vol. 72, No. 3, pp. 395-399 (1989). [IMSE/Ceramics Division].

Studies in the Sr-Y-Cu-o and Ba-Sr-Y-Cu-O systems have revealed that Sr will substitute for Ba in $(Ba,Sr)_2YCu_3O_{6+x}$ up to about 60 percent. There are no ternary compounds in the Sr-Y-Cu-O system equivalent to the three ternary phases in the Ba system. A new binary phase, "$Sr_{14}Cu_{24}O_{41}$", CuO $\approx$ 63.158 mole % was found, which forms a solid solution with Y_2O_3 to a Sr:Y ratio of approximately 2:1. This phase can also incorporate considerable amounts of Ba and Ca as well as many other large ions.

CRYSTAL CHEMISTRY AND PHASE EQUILIBRIA STUDIES OF THE BaO-½R_2O_3-CuO SYSTEMS III: X-RAY POWDER CHARACTERIZATION AND DIFFRACTION PATTERNS OF $Ba_3R_3Cu_6O_{14+x}$, R = LANTHANIDES, W. Wong-Ng, C.K. Chiang, B. Paretzkin, and E.R. Fuller, Jr., Powder Diffraction, Vol. 5, No. 1, pp. 26-32 (March 1990). [IMSE/Ceramics Division]

The superconductor related phases $Ba_3R_3Cu_6O_{14+x}$, (or $Ba_{2-z}R_{1+z}Cu_3O_{7+x}$, with z = 0.5), where R = Pr, Nd, Sm, and Eu, have been prepared and characterized by X-ray powder diffraction, ac magnetic susceptibility measurement, resistivity measurement and thermogravimetric analysis (TGA). Attempts to make this compound with R = Gd, Dy, Y, Er, and Lu were not successful. This compound does not appear to form for rare-earth elements, R, with an ionic size smaller than Eu. The oxygen content of these materials was estimated by TGA. The $Ba_3R_3Cu_6O_{14+x}$ compounds which were sintered at 950°C and annealed in oxygen at 550°C were found to be non-superconducting above 10 K. Previously reported results for the R=La compound have indicated that it was superconducting with a transition temperature of 15 K. The oxidation-reduction behavior of these materials is similar to that of the superconductor phases $Ba_2RCu_3O_{6+x}$.

These isostructural compounds are tetragonal with a space group of P4/mmm. The cell parameters range from a = 3.8612(4), c = 11.5624(14) Å, V = 172.38(4) Å^3 in the Eu compound to a = 3.8893(3), c = 11.6370(11) Å, and V = 176.03(3) Å^3 in the Pr compound. The standard diffraction patterns of these materials are given.

CRYSTAL CHEMISTRY AND PHASE EQUILIBRIA OF THE BaO-R_2O_3CuO SYSTEMS, Winnie Wong-Ng, Boris Paretzkin, and Edwin R. Fuller, Jr., Advances in X-Ray Analysis, Vol. 33, pp. 453-465 (1990). [IMSE/Ceramics Division]

Two important factors, the progressively decreasing size of the lanthanides, which is known as the lanthanide contraction, as well as the stability of different oxidation states of these elements influence the prediction of compound formation in the Ba-R-Cu-O systems. A systematic

investigation of these lanthanide systems and comparison with the Y system has revealed a correlation of the effect of the above factors, in particular the size factor, on the trend of phase formation, solid solution formation and phase compatibility diagrams of the Ba-R-Cu-O systems. For example, it has been found that the smaller the size of R^{3+} or the greater the mismatch between Ba^{2+} and R^{3+} in the solid solution formation. This differing extent of solid solution formation influences the ternary phase relationships.

11. PROCESSING—BULK MATERIAL

LOW TEMPERATURE THERMAL PROCESSING OF $Ba_2YCu_3O_{7-x}$ SUPERCONDUCTING CERAMICS, C.K. Chiang, L.P. Cook, S.S. Chang, J.E. Blendell, and R.S. Roth, Advanced Ceramic Materials: Ceramic Superconductors, Vol. 2, No. 3B, pp. 530-538 (July 15, 1987). [IMSE/Ceramics Division and Polymer Division]

Thermal processing of superconducting ceramics $Ba_2YCu_3O_{7-x}$, in the temperature range between 50°C and 750°C was studied. The oxygen content of the ceramic was reversible under thermal treatment. The temperature for optimizing oxygen content was discussed.

THERMAL ANALYSIS OF $Ba_2YCu_3O_{7-x}$ AT 700-1000°C IN AIR, L.P. Cook, C.K. Chiang, W. Wong-Ng, and J. Blendell, Advanced Ceramic Materials: Ceramic Superconductors, Vol. 2, No. 3B, pp. 656-661 (July 15, 1987). [IMSE/Ceramics Division]

Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) of $Ba_2YCu_3O_{7-x}$ using conventional platinum cells yields a series of well-defined reversible thermal events between 850 and 1000°C. At least one of these is interpreted as being due to the products of platinum reaction. However, changes in slope are also observed on TGA curves taken using non-reactive MgO cells. The other thermal events may therefore represent minor phase changes in $Ba_2YCu_3O_{7-x}$ in response to variations in temperature/stoichiometry.

PROCESSING - PROPERTY RELATIONS FOR $Ba_2YCu_3O_{7-x}$ HIGH T_c SUPERCONDUCTORS, J.E. Blendell, C.K. Chiang, D.C. Cranmer, S.W. Freiman, E.R. Fuller, Jr., E.D. Krasicka, Ward L. Johnson, H.M. Ledbetter, L.H. Bennett, L.J. Swartzendruber, R.B. Marinenko, R.L. Myklebust, D.S. Bright, and D.E. Newbury, Advanced Ceramic Materials: Ceramic Superconductors, Vol. 2, No. 3B, pp. 512-529 (1987). [IMSE/Ceramics, Fracture & Deformation, and Metallurgy Divisions, NML/Gas & Articulate Science Division]

Ceramic materials exhibit strong relationships between their physical properties, their ceramic microstructures, and the processing sequence that produced these microstructures and properties. This interrelation between processing, microstructures, and properties is particularly true for the new high T_c superconducting ceramic oxides of the type $Ba_2YCu_3O_{7-x}$. To characterize some of these relations, a number of properties have been measured for these superconducting ceramics, and related to the microstructures and processing sequence. The $Ba_2YCu_3O_{7-x}$ ceramics were prepared by powder processing techniques, followed by dry pressing and sintering. Sintering was conducted in both air and flowing oxygen at various temperatures, followed by annealing at various temperatures and environments. Superconducting properties, such as the transition temperature and the width of the transition, were measured by both electrical conductivity and AC magnetic susceptibility; both showing a strong sensitivity to annealing temperature and annealing atmosphere. The microstructure and density was also strongly dependent on processing conditions. In this regard compositional mapping proved to be an important technique for quantifying the microstructural properties. Structural properties, which will be important for the reliable use of the materials, were characterized through the fracture strength, the fracture toughness, and the elastic moduli.

RELATIONSHIP OF ELECTRICAL, MAGNETIC, AND MECHANICAL PROPERTIES TO PROCESSING IN HIGH T_c SUPERCONDUCTORS, J.E. Blendell, C.K. Chiang, D.C. Cranmer, S.W. Freiman, E.R. Fuller, Jr., E.D. Krasicka, Ward L. Johnson, H.M. Ledbetter, L.H. Bennett, L.J. Swartzendruber, R.B. Marinenko, R.L. Myklebust, D.S. Bright, and D.E. Newbury, American Chemical Society Symposium Series 351, "Chemistry of High Temperature Superconductors," pp. 240-260 (September 1987). [IMSE/Ceramics Division, Fracture and Deformation Division, and Metallurgy Division; and CAC/Gas and Particulate Science Division]

The interrelation between processing, microstructure, and properties is an important factor in understanding the behavior of ceramic materials. This type of understanding will be particularly important in the development of new high T_c superconducting ceramic oxides of the type $Ba_2YCu_3O_{7-x}$. As an initial effort in understanding these relations, a number of properties have been measured for these superconducting ceramics and related to their microstructure and processing sequence. The $Ba_2YCu_3O_{7-x}$ ceramics were prepared by powder processing techniques, followed by dry pressing and sintering in both air and flowing oxygen at various temperatures. The sintered bodies were annealed at various temperatures and environments. Superconducting properties, such as the transition temperature and the width of the transition, were measured by both electrical conductivity and AC magnetic susceptibility; both of these properties show a strong sensitivity to annealing temperature and atmosphere. The microstructure and density were also strongly dependent on processing conditions. In this regard, compositional mapping proved to be an important technique for quantifying microstructural variations. Mechanical properties, such as elastic modulus, hardness, and fracture toughness, which will be important for the reliable use of these materials in large scale structures, were also determined.

DENSIFICATION, SUSCEPTIBILITY AND MICROSTRUCTURE OF $Ba_2YCu_3O_{6+x}$, John E. Blendell and Laura C. Stearns, Annual Meeting of the Basic Science Division of the American Ceramic Society, November 1-4, 1987, Orlando, Florida; Ceramic Transactions, Vol. 1, Part B, Ceramic Powder Science, pp. 1146-1155 (1988). [IMSE/Ceramics Division]

The densification of $Ba_2YCu_3O_{6+x}$ for different sintering conditions has been measured. The effects of compaction pressure, atmosphere and temperature have been investigated. Measurements of the AC magnetic susceptibility have been used to determine the superconducting properties. Dense samples were found to have less superconducting material as compared to porous samples. Compositional mapping of the samples showed variations in composition in the samples, corresponding to Cu-rich regions and Y-poor regions. Sinter-forging was done in order to align the grains.

LOW TEMPERATURE APPROACHES TO SUPERCONDUCTIVE MATERIALS: A CHALLENGE IN CHEMICAL SYNTHESIS, Joseph J. Ritter, Ceramics Powder Science II, American Ceramic Society, Westerville, OH, pp. 79-84 (May 1988). [IMSE/Ceramics Division]

The synthesis of Ba-Y-Cu oxide ceramic powders is an essential factor in the development of superconductive devices. While the major effort in the superconductor research is supported with powders synthesized by conventional high temperature techniques, the problem of generating precursors to these materials by low temperature routes presents a significant challenge. Results from four different chemical approaches ranging from the precipitation of Y-Ba-Cu

hydroxycarbonates to reactions between Y-Ba alkoxides with $Cu(OH)_2$ are given. All of these systems progress through an intermediate triphasic mixture of $BaCO_3$, CuO and Y_2O_3 before being converted to $YBa_2Cu_3O_{6+x}$ between 800 and 950°. Superconductive behavior is measured in each case after annealing the specimens in O_2 at ~600°. The benefits of ideal and practical low temperature processing are discussed.

SYNTHESIS AND MAGNETIC PROPERTIES OF THE Bi-Sr-Ca-Cu OXIDE 80 and 110 K SUPERCONDUCTORS, J.S. Wallace, J.J. Ritter, E.R. Fuller, Jr., L.H. Bennett, R.D. Shull, and L.J. Swartzendruber, Physical Review B, Vol. 39, No. 4, pp. 2333-2335 (February 1, 1989). [IMSE/Ceramics Division and Metallurgy Division]

We report on the observation of both the 80- and 110-K superconducting transitions in a nominally $BiSrCaCu_2O_x$ material produced by a new chemical synthesis procedure as well as by solid state reaction. We also discuss a method for the confirmation of the presence of superconductivity when only a small amount is present.

COMPOSITIONAL CONTROL OF THE MICROSTRUCTURE OF $Ba_2YCu_3O_{6+x}$, John E. Blendell, Carol A. Handwerker, Mark D. Vaudin, and Edwin R. Fuller, Jr., Journal of Crystal Growth, Vol. 89, pp. 93-100 (1988). [IMSE/Ceramics Division]

Although $Ba_2YCu_3O_{6+x}$ exhibits superconductivity at high temperatures, the critical current density (J_c) in bulk polycrystalline materials, prepared by sintering, is several orders of magnitude below useful values. One possible source of low J_c in polycrystalline materials is the presence of resistive grain boundary junctions resulting from second phases or impurity segregation. Chemical composition plays a major role in determining the phases present and hence the grain boundary characteristics. Preliminary composition measurements of $Ba_2YCu_3O_{6+x}$ include the ratio of major elements, impurity concentrations at the trace level, compositional mapping of major elements by electron probe microanalyses, and observations of sintered microstructures. Results on liquid phase formation and carbon contamination introduced during powder processing and from exposure to atmospheric CO_2 indicate that a great degree of compositional control is required for any meaningful characterization of the Ba-Y-Cu-O system.

A STUDY OF THE HYDROXYCARBONATE PRECURSOR ROUTE TO THE $YBa_2Cu_3O_{7-X}$ HIGH T_c SUPERCONDUCTOR, Joseph J. Ritter and James F. Kelly, High Temperature Superconducting Compounds: Processing & Related Properties, S.H. Whang and A. DasGupta, Editors, The Minerals, Metals & Materials Society, pp. 303-310 (1989). [IMSE/Ceramics Division]

The precipitation of Ba, Y and Cu as precursors to the $YBa_2Cu_3O_{7-x}$ high T_c superconductor is an attractive route to this material. However, studies in our laboratory indicate that powder homogeneity is strongly dependent upon ionic concentrations and system pH. Moreover, we have assessed the composition of the precipitated moieties. These data have been incorporated into controlling the precipitation process in order to optimize particle size and powder homogeneity. Further processing of these powders has yielded polycrystalline solids with sharp transitions near 95K and high transport current densities.

CRYSTAL CHEMISTRY AND PHASE EQUILIBRIA STUDIES OF THE BaO-R_2O_3-CuO SYSTEMS. IV. CRYSTAL CHEMISTRY AND SUBSOLIDUS PHASE RELATIONSHIP STUDIES OF THE CuO-RICH REGION OF THE TERNARY DIAGRAMS, R=LANTHANIDES, Winnie Wong-Ng, Boris Paretzkin, and Edwin R. Fuller, Jr., Journal of Solid State Chemistry, Vol. 85, pp. 117-132 (1990). [IMSE/Ceramics Division]

In the BaO-½R_2O3-CuO systems, where R = lanthanides and yttrium, general trends of phase formation, solid solution formation and phase relationships are correlated with the ionic size of R. In air at 950°C the phase relationships in the CuO-rich region of these ternary diagrams progressively change from the La system through the Nd, Sm, Eu, Gd, Y, Ho systems to the Er system. First, the La system has the greatest number of ternary compounds. Second, the superconductor material, $Ba_2RCu_3O_{6+x}$, exhibits a solid solution of $Ba_{2-z}R_{1+z}Cu_3O_{6+x}$ for the first half of the lanthanide elements with a range of formation which varies with the ionic size of R. Third, a trend is observed regarding the tie-line connections between Ba_2RCuO_5, CuO, and the phases $Ba_{2-z}R_{1+z}Cu_3O_{6+x}$, and the binary phase R_2CuO_4, or $R_2Cu_2O_5$. For the first half of the lanthanides, except for La, a compatibility join is found to connect R_2CuO_4 and the tetragonal end member of the $Ba_{2-z}R_{1+z}Cu_3O_{6+x}$ phase. In systems where R has a smaller ionic size, R = Eu and beyond, the tie-line connection switches to join the Ba_2RCuO_5 phase and the CuO phase. For R = Dy and beyond, the binary phase R_2CuO_4 is replaced by the binary phase $R_2Cu_2O_5$.

POSSIBLE "PROXIMITY MATRIX" ROUTE TO HIGH CURRENT CONDUCTORS, John Moreland, Y.K. Li,* J.W. Ekin, and L.F. Goodrich, Advances in Cryogenic Engineering (Materials), Vol. 36, Part A, pp. 413-421 (1990). [CEEE/Electromagnetic Technology Division]
*Chinese Academy of Science, Beijing, China

The conductance of point contacts between the surfaces of superconducting $YBa_2Cu_3O_{7-\delta}$ thin films is very low. This is probably due to a native insulating surface layer. The conductance of these point contacts can be markedly increased by vacuum depositing and subsequently annealing a thin layer of Ag into the surface of the films. We believe that what might be described as a normal-metal superconducting "proximity matrix," is formed at the surface of the Ag coated $YBa_2Cu_3O_{7-\delta}$ films. In this paper, we describe our efforts to adapt this method to $YBa_2Cu_3O_{7-\delta}$ powder. In particular, we have developed a procedure for vacuum deposition of very thin Ag coatings onto the surface of $YBa_2Cu_3O_x$ powder grains. The Ag-treated powder is then pelletized, sintered, annealed, and cut to form small conducting bars for electrical transport testing.

ORIGIN OF GRAIN BOUNDARY WEAK LINKS IN $BaPb_{1-x}Bi_xO_3$ SUPERCONDUCTOR, T. Takagai,*† Y.-M. Chiang,* and A. Roshko, Journal of Applied Physics, Vol. 68, No. 11, pp. 5750-5758 (1990). [CEEE/Electromagnetic Technology Division]
*Massachusetts Institute of Technology
†Permanent address: DENKA, Tokyo Japan

Although $BaPb_{0.75}Bi_{0.25}O_3$ (BPB) has a comparatively large superconducting coherence length of ~7 nm and no reported anisotropy in its superconducting parameters, polycrystalline BPB exhibits the same rapid decrease in transport critical current density (J_{ct}) with low applied field (< ~50 Oe) that is characteristic of grain boundary weak links in cuprate superconductors (e.g., $La_{2-x}Sr_xCuO_4$, $YBa_2Cu_3O_{7-x}$). We have studied the effects of processing thermal history on the formation and morphology of grain boundary phases, and on the composition of BPB boundaries

with and without second phase, in order to understand the origin of these weak links. Scanning transmission electron microscopy and Auger electron spectroscopy results show the presence of a Pb-Bi-Ba-O phase that is wetting and liquid above ~570°C, but which retracts to three-grain junctions upon slow cooling or annealing at lower temperatures. However, weak-link behavior persists in materials with retracted secondary phase, as well as in hot isostatically pressed samples that never exceed the secondary phase melting temperature. It is found that the grain boundaries remain Bi- and Pb-rich even after the retraction of secondary phases; samples that never exceed the melting temperature of the secondary phase show absence of segregation at some but not all grain boundaries. The composition of the grain boundaries as well as J_{ct} vs temperature measurements indicate that the boundaries act as SIS tunnel junctions.

MOLECULAR CHEMISTRY AND THE SYNTHESIS OF PRECURSORS TO ELECTRONIC CERAMIC MATERIALS, J.J. Ritter, J.F. Kelly, D.E. Newbury, and D.B. Minor, NIST Special Publication 804, Chemistry of Electronic Ceramic Materials, Proceedings of the International Conference, August 17-22, 1990, Jackson, Wyoming, pp. 123-136 (January 1991). [MSEL/Ceramics Division and CAM]

The use of molecular chemical reactions to generate precursors to ceramic materials has been the subject of considerable study for more than three decades. This approach, in many cases, offers distinct advantages over conventional high-temperature syntheses particularly in the areas of improved homogeneity, purity, particle size regulation and amenability to intelligent processing control. Generally, within the broad spectrum of chemical schemes available for ceramic precursor synthesis, the molecular processes are poorly understood. Moreover, the reactions are often interpreted either through "chemical intuition" or by analogy to similar chemistries, rather than from measurements on the relevant systems. An additional complicating factor arises from our lack of knowledge about the mechanisms operating during the conversion of precursors to ceramic phases.

This presentation elaborates on several chemical routes which have been used to synthesize ceramic precursors. The discussion includes systems where control over the molecular architecture may lead to ceramic phases at low temperatures. Other systems, which essentially deliver random, intimately mixed assemblies of the elements, are also examined. The various approaches are illustrated with examples from research programs at NIST which have focused on the chemical synthesis of precursors to barium polytitanates, metal carbides and borides, electrically conductive oxides and nitrides, and the YBCO high-temperature superconductor.

THERMOMECHANICAL DETWINNING OF SUPERCONDUCTING $YBa_2Cu_3O_{7-x}$ SINGLE CRYSTALS, Debra L. Kaiser, Frank W. Gayle, Robert S. Roth, and Lydon J. Swartzendruber, Journal of Materials Research, Vol. 4, No. 4, pp. 745-747 (July/August 1989). [IMSE/Metallurgy Division]

A method for the complete removal of twins from single crystals of superconducting $YBa_2Cu_3O_{7-x}$ is described. The process depends on ferroelastic behavior found to exist in the phase, and should be generally applicable to the layered perovskite-type phases containing accommodation twins resulting from a tetragonal-to-orthorhombic transformation on cooling. The twin-free, superconducting single crystals will enable investigation of *a-b* anisotropy of properties as well as crystal structure determination without complication by the presence of microtwins.

12. PROCESSING—THIN FILMS

SUPERCONDUCTIVITY IN BULK AND THIN FILMS OF $La_{1.85}Sr_{0.15}CuO_{4-\delta}$ AND $Ba_2YCu_3O_{7-\delta}$, K. Moorjani,* J. Bohandy,* F.J. Adrian,* B.F. Kim,* R.D. Shull, C.K. Chiang, L.J. Swartzendruber, and L.H. Bennett, Physical Review B, Vol. 36, No. 7, pp. 4036-4038 (September 1, 1987). [IMSE/Metallurgy Division and Ceramics Division]

*Johns Hopkins University

A laser-ablation technique was used to deposit thin films from the bulk oxides $La_{1.85}Sr_{0.15}CuO_{4-\delta}$ and $Ba_2YCu_3O_{7-\delta}$, whose superconductivity properties were investigated by dc resistivity, complex ac susceptibility, and microwave response. The latter technique was employed to establish that the thin films have superconducting regions with properties similar to the bulk materials.

MICRO-RAMAN SPECTROSCOPY OF HIGH-T_c SUPERCONDUCTORS IN THE Y-Ba-Cu-O SYSTEM, E.S. Etz, Winnie Wong-Ng, J.E. Blendell, and C.K. Chiang, Microbeam Analysis—1988, pp. 187-192 (August 1988). [CAC/Gas and Particulate Science Division and IMSE/Ceramics Division]

Preliminary results are reported on the (unpolarized) room-temperature micro-Raman spectra of several types of samples and predominant phases in the Y-Ba-Cu-O ceramic system. These investigations provide microstructural characterization of these materials with a spatial resolution equal to that obtained from electron microprobe microanalysis for elemental composition. Experimental detail is given of the measurement parameters employed in the Raman microprobe study of these materials. The samples examined are polycrystalline ceramic powders, sintered ceramic pellets, and a specimen of single-crystal material. Of interest are the determination of the component phases, mixed stoichiometries, and crystal structures. The micro-Raman spectra are interpreted on the basis of the reported vibrational data (Raman and infrared) for materials in this system of both tetragonal and orthorhombic symmetry. Conclusions are drawn relative to the heterogeneity of these polyphase materials.

PROPERTIES OF $YBa_2Cu_3O_{7-\delta}$ THIN FILMS GROWN ON OFF-AXIS-CUT MgO SUBSTRATES, S.E. Russek, B. Jeanneret, D.A. Rudman, and J.W. Ekin, Proceedings, Applied Superconductivity Conference, September 24-28, 1990, Snowmass, Colorado, in IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 931-934 (March 1989). [CEEE/Electromagnetic Technology Division]

A series of $YBa_2Cu_3O_{7-\delta}$ films has been reactively sputtered on off-axis-cut MgO substrates. All the films were oriented with the *c* axis normal to the substrate regardless of substrate orientation, indicating that growth dynamics is a major factor influencing film orientation on non-lattice-matched substrates. As the substrate orientation is moved off the [100] direction the films showed a decrease in transition temperature and showed properties indicative of an increased density of weak links. The films grown on high-angle substrates showed better properties than the films grown on low-angle substrates. Films grown on (110) MgO were as good as films grown on (100) MgO.

MORPHOLOGY OF SILVER ON $YBa_2cU_3O_{7-\delta}$ THIN FILMS, A. Roshko, R.H. Ono, J.A. Beall, J. Moreland, A.J. Nelson,* and S.E. Asher,* IEEE Transactions on Magnetics, Vol. 27, No. 2, pp. 1616-1618 (March 1991). [EEEL/Electromagnetic Technology Division]

*Solar Energy Research Institute

The morphology of silver layers deposited and annealed on laser ablated $YBa_2Cu_3O_{7-\delta}$ films has been examined. Silver was found to dewet the $YBa_2Cu_3O_{7-\delta}$ (001) surface on annealing in oxygen and nitrogen. This dewetting behavior may be kinetically inhibited by using either thick silver layers or short anneals and low temperatures. Silver layers 70 nm to 2.5 μm thick were studied on films deposited on $SrTiO_3$, $LaAlO_3$, and MgO substrates. Anneal times were varied from 6.5 min to 15 hrs at temperatures from 200 to 700°C.

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* U.S. G.P.O.:1991-311-891:40061

BL-114A
(5-90)

U.S. DEPARTMENT OF COMMERCE
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

BIBLIOGRAPHIC DATA SHEET

1. PUBLICATION OR REPORT NUMBER
NIST/SP-826

2. PERFORMING ORGANIZATION REPORT NUMBER
B91-0289

3. PUBLICATION DATE
November 1991

4. TITLE AND SUBTITLE

High-Temperature Superconductivity: Abstracts of NIST Publications, 1987-1991

5. AUTHOR(S)

Mary E. DeWeese (Editor)

6. PERFORMING ORGANIZATION (IF JOINT OR OTHER THAN NIST, SEE INSTRUCTIONS)

U.S. DEPARTMENT OF COMMERCE
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
BOULDER, COLORADO 80303-3328

7. CONTRACT/GRANT NUMBER

8. TYPE OF REPORT AND PERIOD COVERED
Final

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (STREET, CITY, STATE, ZIP)

Same as item # 6

10. SUPPLEMENTARY NOTES

Supersedes NIST SP 759

11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

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12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

bismuth superconductors; critical current; critical field; critical temperature; high T_c; high-temperature superconductors; low-temperature superconductors; superconducting ceramics; superconductors; thallium superconductors; YBCO; yttrium superconductors

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14. NUMBER OF PRINTED PAGES
90

15. PRICE

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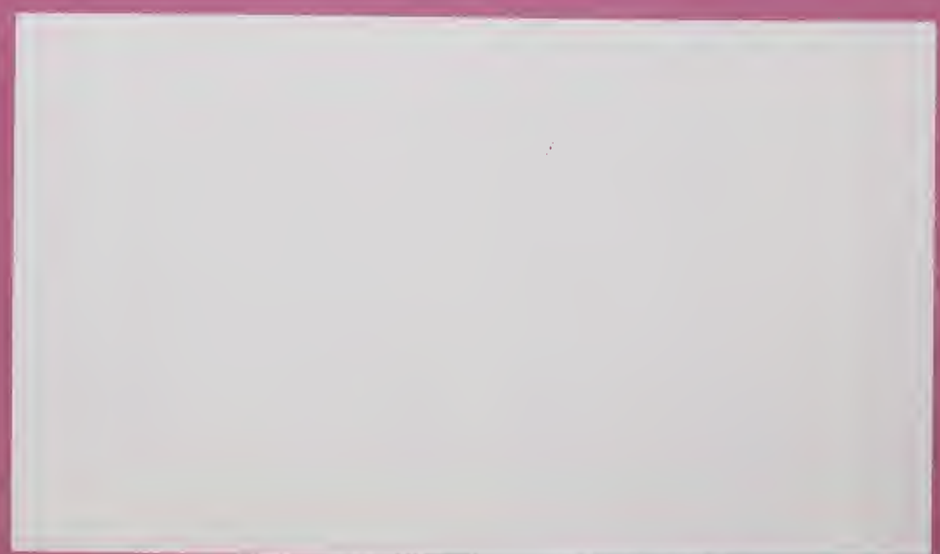

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