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High-Temperature Superconductivity: Abstracts of NIST Publications, 1987–1988

Mary E. DeWeese, Robert A. Kamper, and Ronald M. Powell, Editors

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Edited by
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NOTE: As of 23 August 1988, the National Bureau of Standards (NBS) became the National Institute of Standards and Technology (NIST) when President Reagan signed into law the Omnibus Trade and Competitiveness Act.

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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

IMSE = Institute for Materials Science and Engineering

HIGH-TEMPERATURE SUPERCONDUCTIVITY: ABSTRACTS OF NIST PUBLICATIONS, 1987-1988

Mary E. DeWeese, Robert A. Kamper, and Ronald M. Powell, Editors

We have collected abstracts from 61 papers published between March 1987 and May 1988 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. INTRODUCTION

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.

2. COMPOSITIONAL ANALYSIS

STANDARD X-RAY DIFFRACTION POWDER 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] (14a)
*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] (14b)
*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).

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] (18)

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.

MEASUREMENT OF SUPERCONDUCTOR STOICHIOMETRY BY PROMPT AND DELAYED NEUTRON ACTIVATION, Richard M. Lindstrom, Transactions, American Nuclear Society, Vol. 56, p. 231 (1988). [CAC/Inorganic Analytical Research Division] (49.3)

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.

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] (74.3)

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.

INITIAL STAGES OF DEGRADATION OF SUPERCONDUCTOR SURFACES: O_2, H_2, CO_2, and CO CHEMISORPTION IN $La_{2-x}Sr_xCuO_4$, 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] (76.3)
*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 $\sim 3 \times 10^{-4}$.

3. CRITICAL CURRENT

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] (6.3)

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†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] (9a)

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] (9b)

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] (10.3)

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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 (~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 IN BULK SINTERED $Y_1Ba_2Cu_3O_x$ 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] (12)

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, Vol.37, No. 16, pp. 9848-9851, (June 1, 1988). [CEEE/Electromagnetic Technology Division] (47)

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.

IRREGULARITY IN Nb-Ti FILAMENT AREA AND ELECTRIC FIELD CURRENT CHARACTERISTICS, J. W. Ekin, Cryogenics 1987, Vol. 27, pp. 603-608 (November 1987). [CEEE/Electromagnetic Technology Division] (66.3)

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] (67.3)

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 (U.S.), NBSIR 88-3088, 75 pages (February 1988). [CEEE/Electromagnetic Technology Division] (68.3)

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] (69.3)

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] (70.3)

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.

4. CRYSTAL STRUCTURE

X-RAY STUDIES OF HELIUM-QUENCHED $Ba_2Y_3C_{7-x}$, 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] (15)

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.

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] (17)

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.

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] (22)

*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] (23)

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, 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] (24)

*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-C-67 (1987). [IMSE/Ceramics Division] (34)

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, Vol. 22, pp. 1733-1740 (December 1987). [IMSE/Reactor Radiation Division] (38)

*AT&T Bell Laboratories, Murray Hill

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] (53)

*University of Maryland

†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 $YBa_2Cu_3O_{7-x}$ 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] (73.3)

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] (75.3)

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.

5. 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] (19)

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 ± 17 GPa. Young's modulus was estimated to be 235 ± 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] (25.3)

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] (28.3)

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] (29.3)

*University of South Carolina
†Lawrence Livermore National Laboratory

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.

REENTRANT SOFTENING IN PEROVSKITELIKE SUPERCONDUCTORS, T. Datta,* H. M. Ledbetter, C. E. Violet,† C. Almasan,* and J. Estrada,* Physical Review B, Vol. 37, No. 13, pp. 7502-7505 (May 1, 1988). [IMSE/Fracture and Deformation Division] (39)

*University of South Carolina
†Lawrence Livermore National Laboratory

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.

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] (51.3)

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. [IMSE/Fracture and Deformation Division] (55.3)

*Lawrence Livermore National Laboratory
†Dept. of Physics and Astronomy, 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] (61)

*Materials Science Division, Argonne National Laboratory

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.

6. ELECTRICAL CONTACTS

METHOD FOR MAKING LOW-RESISTIVITY CONTACTS TO HIGH T_c SUPERCONDUCTORS, J. W. Ekin, A. J. Panson,* B. A. Blankenship,* Applied Physics Letters, Vol. 52, No. 4, pp. 331-333 (January 25, 1988). [CEEE/Electromagnetic Technology Division] (44.3)

*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 RANGE, J. W. Ekin, T. M. Larson, N. F. Bergren, A. J. Nelson*, A. B. Swartzlander*, L. L. Kazmerski*, A. Panson†, and B. Blankenship†, Applied Physics Letters, Vol. 52, No. 21, pp. 1819-1821 (May 1988). [CEEE/Electromagnetic Technology Division] (63.1)

*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 in oxygen for 1 h at temperatures above ~500°C and gold/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.

7. 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] (21)
*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.

SYNCHROTRON RADIATION STUDIES OF THE ELECTRONIC STRUCTURES OF HIGH-T_c SUPERCONDUCTORS, Richard L. Kurtz, American Institute of Physics Conference 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., New York, pp. 222-234 (1988). [CBS/Surface Science Division] (43.3)

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.

8. 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 (May 1987). [CEEE/Electromagnetic Technology Division] (1.3)
*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, and 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] (2.3)
*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] (3.3)
*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] (4.3)

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] (8.3)

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, Advanced Cryogenic Engineering, Vol. 34, pp. 625-632 (1988). [CEEE/Electromagnetic Technology Division] (11)

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] (60.3)

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. Gschneidner, Jr., International Conference on High Temperature Superconductors--Materials and Mechanisms of Superconductivity, Interlaken, Switzerland, pp. 1383-1384 (1988). [CEEE/Electromagnetic Technology Division] (64.3)

*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.

9. 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] (7.3)

*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 ± 0.05 μV/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, D. 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] (32.3)

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-Cu-Ox 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] (33.3)

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 A $Y_1Ba_2Cu_3O_x$ 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] (42.3)

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.

10. 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] (5.3)

*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 YBa-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] (30.3)

*Westinghouse

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. Nogués,* 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 (40.3)

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.

11. 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] (13.3)

Preliminary phase equilibria diagrams are constructed for the "binary" systems BaO-$\frac{1}{2}Y_2O_3$, BaO-CuO_x, $\frac{1}{2}Y_2O_3$-CuO_x, and the "ternary" system BaO-$\frac{1}{2}Y_2O_3$-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 $Ba_3YCu_2O_x$.

12. 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] (16)

*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.

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. Drescher-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 Division] (20)

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. Drescher-Krasicka, Ward L. Johnson, H. M. Ledbetter, L. H. Bennett, L. J. Schwartzendruber, 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] (31.3)

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.

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] (48)

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.

13. 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 (27.3)

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.

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