SYMPOSIUM II
Superconducting Materials—Properties, Crystal Chemistry, and Processing
November 28 – December 3, 1999

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superconductors cannot be set up due to mismatch effects between the two LBICO and CCO layers. A strategy is then developed, which allows new superconductors to be realized using a "mixed" structure (LBICO)_m (SCO)_m (CCO)_m (LBICO)_m. Such superconductors show a significant decrease of the resistivity which appears most promising for the appearance of superconductivity.

New superconductors corresponding to the artificial stacking of LaAlO_3 (LAO) and YbMgO_2 layers have also been realized. The (YBCO)_m (LAO)_n superconducting superlattices exhibit Tc up to 85 K. The coupling between the YBCO superconducting layers is a function of Tc but indirectly related to the stacking sequence of the YBCO and LAO layers. HREM observations show an excellent crystallization and a perfect ordering of the layers, with a ruggedness at the interface smaller than 0.2. Structural models, explaining the junction between LAO and YBCO layers are in progress.

9:00 AM #11.2

STRUCTURAL ANOMALIES AT THE Crossover BETWEEN THE UNDER DOPED AND OVER DOPED STATES IN HIGH-Tc
SUPERCONDUCTORS. J.D. Jorgensen, D.G. Hinks, H. Shaked, Materials Science Division and Science and Technology Center for Superconductivity, Argonne National Laboratory, Argonne, IL; O. Chmaissem, B. Dobrovolskii, Dept. of Physics, Northern Illinois Univ., DeKalb, IL; J.L. Wagner, Dept. of Physics, Univ. of North Dakota, Grand Forks, ND, Y. Eckstein, A. Knizhnik, Dept. of Physics and Crown Center for Superconductivity, Technion, Haifa, ISRAEL.

The occurrence of a maximum Tc, separating the under doped and over doped regimes in a unique feature of copper-oxide high-temperature superconductors. Although this behavior is believed to occur for all high-Tc superconductors, there are a limited number of systems in which it can be achieved because of difficulties in the synthesis chemistry. We have studied the crossover from the under doped to the over doped regime in two systems - a copper-substituted 123 compound and High-Tc CuO2 - and have observed structural anomalies at the maximum Tc, in both cases. We conclude that these anomalies are a manifestation of a change in the electronic structure upon passing through the maximum Tc. Changes in the defect chemistry can also be associated with such a change in the electronic structure. Understanding this behavior is important for developing methods for extending the doping ranges of other high-Tc compounds.

This work is supported by the US Dept. of Energy, Basic Energy Sciences - Materials Sciences, contract W-31-109-ENG-38, and the NSF, Office of Science and Technology Centers, grant DMR 91-20000.

9:30 AM #11.3

SUPERCONDUCTIVITY IN CARBIDE HALIDES OF RARE-EARTH METALS. R.W. Hemm, R.K. Kremer, A. Simon, Max-Planck-Institut fuer Festkoerperforschung, Stuttgart, GERMANY.

Superconductors with layered crystal structures have attracted particular interest because of their unusual physical properties. In 1989 discovered superconductivity in carbide halides with metal Nb was found with melting temperatures of 10 K. The NbCl compound was synthesized and then cooled to the superconducting transition temperature. The magnetic properties of this compound were studied using a magnetization apparatus. The magnetic susceptibility was measured using a superconducting quantum interference device (SQUID) magnetometer. The results showed that the compound is a type II superconductor with a critical temperature of 2 K. The magnetic moment of the compound was determined using a SQUID magnetometer.
10:15 AM *III.4
SUPERCONDUCTING HG-BASED MIXED OXIDES AND OXYFLUORIDES. E.V. Antipov, A.M. Akhmedov, M.G. Reznov, S.N. Postnil, K.A. Lezhnev, D.A. Pavlov, V.A. Alyahov, Moscow State University, Department of Chemistry, Moscow, Russia; A.M. Balagurov, D.V. Sheptyakov, Joint Institute for Nuclear Research, Dubna, Russia.
HgBaCu2O6+δ, CuO2+δ, and CuO layers exhibit the highest Tc among known superconductors thus providing a great attention to these compounds. Syntheses under high pressure and under regulated temperature are performed with various pressure, temperatures, and time conditions, and properties of different members of the series will be presented and discussed. There are two parameters influencing Tc, in this family: a width of a perovskite slab (n) and a concentration (δ) of the extra oxygen. The increase of Tc with n, occurs until the third member, while after that it decreases. All members of the series exhibit cupola-shaped dependences of Tc vs. δ. Neutron powder diffraction experiments were carried out for oxygenated and fluorinated Hg1211 samples with different oxygen content and fluorination stages exhibit also the cupola-shaped behavior for Tc vs. δ. NPD showed twice the amount of extra fluorine in comparison with those for the oxygenated Hg1211 phases with close Tc. Fluorination of Hg1223 resulted in a slight increase of Tc in comparison with oxygenated material. Structures of fluorinated and oxygenated Hg1223 were refined by NPD. The influence of pressure on the structure and Tc of Hg1211 strongly depends on the doping level. An increase of the oxygen content on going from underdoped to overdoped states results in the larger compression of the apical Cu-O and Bi-O distances, while the Bi-O dimer is further as a distance between Bi-O and from the (CuO2) layers becomes pressure independent. These results together with the data for fluorinated material allow to determine the structural features responsible for the Tc variation under pressure.

10:45 AM *III.5
EFFECTS OF LEAD SUBSTITUTE ON BISCCO. Mikio Takano, Yasunori Reki, Zenji Hira, Ichiro Chok, ICRI, Kyoto University, Uji, Kyoto, Japan; Yoshihiro Kameko, Tom Takahashi, Faculty of Engineering, Okayama University, Okayama, Japan; Kenzo Ishii, Graduate School of Integrated Science, Yokohama City University, Yokohama, Japan.
Substitution of lead for barium shows several chemical and electrical effects in the BISCO system. The presence of a partially molten phase including lead mediates the formation of the 2223 phase, the modulating superconducting phase is elongated with increasing lead content for all the 2201-2223 phases, the crystal symmetry changes with lead content, resistivity measured normal to the CuO2 planes becomes lower, certain lead-containing fine particles precipitate on the surface of crystals when annealed at low temperatures in oxidizing atmospheres like air but dissolve again into the crystals on annealing at high temperatures in the same atmosphere. Two years ago we showed that the pinning effect is greatly improved in the 2212-lead-substituted 2201 phase. More recent studies of the structure evolution, superconducting properties and transport properties of the effects of lithium on the formation process and structural and electronic properties will be presented.

11:15 AM *III.6
The multi-layered crystal structures of high-Tc superconducting copper oxides are viewed as members of different homologous series. Correlation between spatial inhomogeneity of different dimensions over the crystals and the basic superconducting properties such as Tc, Hir and the peak effect has been investigated. It is shown that the hole distributions and the different content and Tc, Fluorination stages depend on the doping route applied. In general, (i) the more homogeneous the hole distribution along the piling direction of the different layers is, the more improved is the Hir characteristics, while (ii) the broader the distribution of the CuO2-plane stacks, the higher is the Tc. Furthermore, (iii) some hundred angstrom modifications are likely to cause the peak effect.

11:45 AM *III.7
NOVEL SUPERCONDUCTORS SYNTHESIZED BY ELECTROCHEMICAL Zn INTERCALATION IN β-ZNCl AND RELATION TO THE CuO2 FLUID. M.C. Arroyo de Douglass, E. Merino, and M.I. Alario-Franco, Facultad de Ciencias Quimicas, Universidad Complutense, Madrid, Spain; F. Drymais, A.D. Banchs, and Z. Fisk, National High Magnetic Field Laboratory, Florida State University, Tallahassee, Florida.
The structure of β-MNX (M = Zn, Hf, X = Cl, Br) consists of an ordered stacking sequence of hexagonal MXNNMX layers held together by van der Waals interactions, allowing the intercalation of different guests into the host lattice. The intercalated M3NN compounds (M = alkaline metal, cobaltocene) are superconductors, with a critical temperature Tc of the superconducting transition that varies only little with the doping level x and is rather insensitive to the choice of the chemical species A for the doping. To our knowledge, no previous work has been reported on the intercalation of divalent ions into this class of compounds, so we have attempted to introduce Zn into β-ZnCl, β-ZnBr, and β-HCI. The lowing/oxidation of Zn into Zn++ was carried out electrodynamically through the potential /charge discharge of Zn/0.2M Zn[2+][SO42-]/2 + DMso + PC [14] using MNN cells in the voltage window between 1 and 5 V against Zn. Noticeably different results from the previously studied lithium doping are obtained for zinc incorporation. In the case of β-HCl an intercalation reaction takes place, leading to the final compound Zn0.667ZnCl with a Tc of 15 K. For β-MNN an irreversible process is observed yielding the superconducting compound Zn0.667ZnCl with a Tc of 14 K.


SESSION II: CRYSTAL CHEMISTRY AND NEW MATERIALS II
Chair: Ruling L. Meng, Monday Afternoon, November 29, 1999
Room 200 (H)

1:30 PM *III.8
STRUCTURAL PROPERTIES AND STABILITIES OF NOVEL COPPER-RICH OXIDES. Douglas A. Vander Sande, Kenneth R. Poeppelemeier, Sylvia M. Yip, Antoine Masgnen, Northwestern University, Chemistry Dept, Evanston IL; Vojnak Dravil, Northwestern University, Materials Science Dept, Evanston IL.
The chemistry of cuprates pervades the field of perovskite superconductors. Many new solid state phases with [AlO2]x−1, x−2, x−3 stochiometry have been found for which over half of the B-cations are copper, but which do not adopt the perovskite structure at ambient pressure. Archetypal examples LnCu3MoO6 and LnCu4VO6 crystalline in a hexagonal structure at ambient pressure which is a homoeotype of YAlO3. Under 6 GPa of pressure, both convert to a layered perovskite. The cuprate layers of the high pressure molybdenum phase, critical for high temperature superconductivity, disappear upon A-site doping in the oxides mix. The stabilities of the different structures are compared as well as possible decomposition products. Magnetic properties and conductivities for doped and undoped samples reflect the stoichiometry and structural architecture.

2:00 PM *III.9
CREATION OF THE BEST PERFORMANCE SUPERCONDUCTOR BASED ON Cu-1234 (Cu2Ba2Cu3Cu4O8+δ) SYSTEM. Hakan Bengtsson, Per Tegmark, Electrochemical Laboratory, Umea, Sweden, Tsukuba, Japan, and CREST, JST.
The purpose of this paper is to propose how to create the best performance superconductor based on the Cu-1234 system. The Cu2BaCu3Cu4O8+δ system of Cu2Ba2Cu3Cu4O8+δ system of Cu2Ba2Cu3Cu4O8+δ x=3-6 family. The best performance superconductor should have a high critical current density (Jc > 100 A/cm2, 50 A/cm2 at 77 K, 0 T), high critical temperature (Tc > 120 K), high critical current density (Jc > 100 A/cm2, 50 A/cm2 at 77 K, 0 T), high critical temperature (Tc > 120 K), high critical current density (Jc > 100 A/cm2, 50 A/cm2 at 77 K, 0 T), and high critical current density (Jc > 100 A/cm2, 50 A/cm2 at 77 K, 0 T). The best performance superconductor will be realized by the modification of superconducting wave function (MSW) and the development of new preparation techniques of thin film for the Cu-1234 system. The Cu-1234 system means the elongation of Jc, based on the uncertainty principle and the transformation from the Jc to the Jc wave superconductivity. The (Jc-Jc)-wave superconductivity will be realized in the F-superconducting wave function (MSW) and the development of new preparation techniques of thin film for the Cu-1234 system. The Cu-1234 system means the elongation of Jc, based on the uncertainty principle and the transformation from the Jc to the Jc wave superconductivity. The (Jc-Jc)-wave superconductivity will be realized in the F-superconducting wave function (MSW) and the development of new preparation techniques of thin film for the Cu-1234 system.
Molecular and Crystal Engineering of High-Tc Structures and Devices

Over the last decade, the field of high-Tc superconductor research has witnessed major advances in which new oxide formulations have been discovered having a number of interesting properties. In many respects, prototype devices and conductors are now being tested using some of these new oxide compounds. From the prior studies in the area, it is clear that highly ordered structures with near atomic-level control at the interior and interfacial regions are necessary to recognize the full utility of these interesting compounds. Towards this goal, our group has initiated a series of studies designed to explore fundamental issues related to the bulk and surface chemistry of high-Tc compounds. From these studies, both solid-state methods for control of bulk properties as well as advanced methods for control of surface chemistry have been developed. This talk will focus on a discussion of the utility of cation substitution techniques and self-assembled monolayer strategies for the crystal and molecular level control of cuprate superconductor structures.

Abstract Withdrawn.

Effects of Cation Substitutions on Tc in Different HTS Systems

The application of external pressure to cuprate superconductors may induce increases of \( T_c \). In spite of numerous attempts, such effect could not be reproduced by applying chemical pressure by substituting small for the large cations, typically Sr for Ba. The effect of these substitutions depends on the cuprate family, and, with the only exception of the \( \text{La}_2\text{Sr}_2\text{CuO}_4 \) system, it has always resulted in a decrease of \( T_c \). In the \( \text{La}_{2-x}\text{Sr}_x\text{CuO}_4 \) system, \( T_c \) increases either for increasing \( x \) or for decreasing external pressure. In addition, Loquet et al. recently showed that \( T_c \) in this system can be further enhanced by contracting the basal plane (through optimal growth of thin layers on a substrate with a smaller parameter) and expanding the \( c \) parameter. Our studies of the crystal structure and superconducting properties of \( \text{YBa}_2\text{Sr}_x\text{Cu}_2\text{O}_y \) as a function of \( x \) and \( y \) showed that the \( Sr \) substitution in this system reduces the unit cell volume as does external pressure. However, it also reduces the strain of the \( Ba/\text{Sr} \) layers which seems to be needed for optimizing the charge transfer between the chain and the plane \( Cu \) cations. The strain of the \( Ba \) layer seems to scale well with \( T_c \), at least in the \( \text{YBCO} \) system, but the increase in the \( Sr \) substituted \( \text{Hg}(\text{Ba}_{2-x}\text{Sr}_x)\text{Cu}_2\text{O}_{8+4} \) compounds. The \( T_c/\gamma \) coefficient, on the contrary, is positive.
EFFECT OF HOLE FILLING BY Lx AND HOLE DOPING BY Ca ON THE SUPERCONDUCTIVITY OF Nd2Ba2Cu3O8−δ
Amish G. Joshi, M.V. Subbarao, D.G. Kubler and R.G. Kulkarni, Department of Physics, Sastra University, Thanjavur, INDIA.

The structural and superconducting properties of Ln and Ca substituted \( \text{Nd}_2\text{Ba}_2\text{Cu}_3\text{O}_{8−δ} \) samples prepared under identical conditions have been investigated by X-ray diffraction, resistivity, ac susceptibility and oxygen content measurements. Two. The observed lowering of \( T_c \), with increasing \( x \) in \( \text{Nd(Ba,La)}_2\text{Cu}_3\text{O}_{8−δ} \), provides convincing evidence that the filling of holes by \( \text{La}^{3+} \) reduces hole concentration and suppresses superconductivity, varying smoothly through an orthorhombic to tetragonal transition with increasing \( x \). This suppression in \( T_c \) can be compensated by an appropriate hole doping with Ca. The introduction of Ca for Nd in \( \text{Nd} \to \text{La} \) substitution (\( x \leq 0.7 \)) system is investigated by X-ray diffraction and neutron-diffraction, resistivity, a.c. susceptibility and oxygen content measurements. Samples with \( 0.1 < x < 0.7 \) are superconducting and \( T_{c,0} \) is between 27 and 79 K. Rietveld refinement of neutron diffraction data on five samples \( x = 0, 0.2, 0.4, 0.5 \) and 0.7 confirm (i) occurrence of a single phase tetragonal superstructure space group \( \text{P}4/m\text{mm} \), (ii) Ca and Y ions substitute onto the Ln sites with consistent displacement of La onto B sites, and (iii) increasing \( x \) from 0 to 0.5 decreases \( L \) of \( 3 \text{ bond} \) length and increases \( \text{Cu} (2)-O (2) \) bond length with corresponding increase in \( T_c \) from 0 K to 79 K suggesting a correlation between bond lengths and \( T_c \) values.

PROCESSING OF SUPERCONDUCTING COMPOSITES IN THE SYSTEM Bi(Ph)2Sr2Ca2Cu2O8−δ (A:Al, Ga, In)

Phase transformations in the systems \( \text{Bi-Sr-Ca-Cu-O} \) (A:Al, Ga, In) were investigated with the purpose to obtain the two phases superconducting composites. The phase transitions in these systems Bi-2212/A-rich phases were determined above and below the Bi-2212 melting point \( \text{Bi}_{2} \text{Sr}_{2} \text{CaCu}_{2} \text{O}_{8} \), \( \text{Bi}_{2} \text{Sr}_{2} \text{Ca}_{0.8} \text{Cu}_{0.2} \text{O}_{8} \) and \( \text{Bi}_{2} \text{Sr}_{2} \text{Ca}_{0.7} \text{Sr}_{0.3} \text{Cu}_{0.5} \text{O}_{8} \) were determined as chemically compatible with Bi-2212. Changes in the Bi-2212 melting process were observed for \( \text{Bi}_{2} \text{Sr}_{2} \text{Ca}_{1.0} \text{Al}_{0.2} (\text{A} = \text{Al}, \text{Ga}, \text{In}) \) and \( \text{Bi}_{2} \text{Sr}_{2} \text{Ca}_{1.0} \text{Al}_{0.2} \) admixture: \( \text{Bi}_{2} \text{Sr}_{2} \text{Ca}_{1.0} \text{Al}_{0.2} \to \text{Bi}_{2} \text{Sr}_{2} \text{Sr}_{0.5} \text{Ca}_{0.5} \text{Al}_{0.2} \to \text{Bi}_{2} \text{Sr}_{2} \text{Sr}_{0.5} \text{Ca}_{0.5} \text{Al}_{0.2} \). 

A NOVEL 02(1 n)1 HOMOLOGOUS SERIES IN THE Bi2Ca2CuO SYSTEM AND A DERIVATIVE WATER-CONTAINING HOMOLOGOUS SERIES.

A NOVEL SERIES IN THE Bi2Ca2CuO SYSTEM AND A DERIVATIVE WATER-CONTAINING HOMOLOGOUS SERIES.

2NOVEL HOMOLOGOUS SERIES OF MULTILAYERED SUPERCONDUCTIVE COPPER OXIDES WERE SUCCESSFULLY ESTABLISHED. THE 02(1 n)1 PHASES (n = 2 , 4 ) IN THE Bi2Ca2CuO SYSTEM WERE OBTAINED FROM HIGH-PRESSURE SYNTHESIS UNDER OXIDE CONDITIONS. THE DERIVATIVE PHASES FORMED SPONTANEOUSLY FROM THE HIGHLY UNSTABLE AS-SYNTHESIZED 02(1 n)1 PHASES WHEN EXPOSED TO HUMID AIR. FOR EACH PHASE 02(1 n)1 PHASES, THE ORIGINAL UNIT CELL OF BODY-CENTERED SYMMETRY WAS FOUND TO SPIN AROUND THE C-axis BY 5.7° ONO, P. J. J. E. S., 11 (2006) 15, 15811/15, 15931/15.

LATTICE DYNAMICS OF OXYGEN AND CATALYTIC PROPERTIES OF LAYERED LANTHANUM CUPRATES.

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2.4.3 CONTROL OF THE GROWTH MORPHOLOGY AND TRANSPORT PROPERTIES OF NbBCO LARGE GRAINS. Wai Le, M. Hari Bubu, D. A. Cardwell and K. Sahama.

Bulk Nd$_{1.4}$Ba$_{2.6}$Cu$_{2.5}$O$_y$ (NbBCO) large grains fabricated by melt processing have been demonstrated to have higher $T_c$, faster growth rate and superior flux pinning properties than YBCO, and hence have better potential for applications in high current devices. These materials, however, are also known to have complicated processing conditions and difficulties in growth morphology and superconducting properties control. Such difficulties are originated from the sensitive dependency of properties on $x$, as well as the high melting point of NbBCO such that no seed with similar lattice structure and higher melting point could be found. This presentation focuses on the manipulation of growth morphologies of NbBCO large grains by additives and thermal properties of the precursor materials. The evaluation of superconducting properties by both transport and magnetization measurements and the comparison between the results will also be discussed in detail. These superconducting properties will be correlated with the growth morphology of the NbBCO grains.

3.10 THERMODYNAMICS OF OXYGEN INTERCHANGE AND OXGEN- LIMITED NbBCO, Nd$_{1.4}$Ba$_{2.6}$Cu$_{2.5}$O$_y$, SODIUM SOLUTIONS. E.A. Troshenko, High School of Materials Science, N.N. Olevnyk, Yu.D. Trosinskyy, Chemical Department, Moscow State University, Moscow, RUSSIA.

In this work thermodynamic features of Nd$_{1.4}$Ba$_{2.6}$Cu$_{2.5}$O$_y$ were analyzed with respect to oxygen kinetics. Thermodynamical treatment of system Nd$_{1.4}$Ba$_{2.6}$Cu$_{2.5}$O$_y$ was carried out using literature data. Oxygen absorption was considered as two processes: (1) complete reaction of compound with minimum oxygen content and (2) mixing of terminal compounds. Thermodynamics of mixing was calculated in a frame of subregular solution model. It was found that for a subregular solution the oxygen content breaks into two intervals with different oxygen behavior. Changes of these can be explained by the hole transfer processes. For a further kinetic study the thermodynamic diffusion factor $W(T,x,y) = \frac{d\ln A_c}{d\ln C_{O_2}}$, was calculated through a wide range of temperatures. The kinetic study consisted in a PGA investigation of oxygen process of quenched solid solutions using a linear heating regime with rates $2, 5, 10, 20 \text{ K/min}$. In the data were processed according to the Marquardt’s scheme to obtain oxygen content boundaries. Samples of solid solutions with different substitution degrees ($x=0.6, 0.45, 0.6$) were made by a ceramic route using Nd$_2$O$_3$, Ba(NO$_3$)$_2$, CuO. Precursors were annealed at $1000\, ^\circ\text{C}$ in air and quenched into liquid nitrogen as followed by XRD study (guinier-chamber, lattice-parameters calculation), SEM (particle size evaluation), and isoemetic titration. It was found that for a large substitution degree $x$ oxygenation started at a lower temperature. As soon as thermodynamic factor is taken into account, it allows us to extrapolate the thermochemical plot $d\ln A_c/dT$ for Nd$_{1.4}$Ba$_{2.6}$Cu$_{2.5}$O$_y$ and to perform further data processing to quasi-thermochemical conditions. The oxygenation process model $d\ln A_c = B_{\text{O}_2}(x) + E_A H(T)/k_B T + \text{W}(T,x)$ was suggested and its parameters were calculated for the investigated solid solutions.

3.11 PRESSURE STUDIES ON STRONTIUM BISUMATE. Michal Regens, S. Sanfilippo, CRIBIT/CNR, Grenoble, France; T. Buche, Nd$_{1.4}$Ba$_{2.6}$Cu$_{2.5}$O$_y$, CNR, Grenoble, France; S. Kurnakov, J. Pahlin, A. Antipov, Chem. Dept., Moscow State University, RUSSIA; and M. Harland, ESHF, Grenoble, France.

The bisumate superconductors are a widely studied system due to their superconducting properties. Although the apparent disproportionation of Bi atoms in Ba$_2$BiO$_3$ is suggestive of non-stoichiometric superconductivity, studies on Nd$_{1.4}$Ba$_{2.6}$Cu$_{2.5}$O$_y$ have shown conventional electron-phonon superconductivity. However, contrary to cuprates, where the diversity of systems has been capital to their correct understanding, the research on the bisumate materials have been limited to the Pb and K substituted compounds. We have recently succeeded in synthesising a third variety of bisumates based on the new oxide Sr$_2$BiO$_3$. By partial substitution of K for Sr, we found superconductivity with $T_c=12\, \text{K}$ in Sr$_{1-x}$K$_x$BiO$_3$ ($x=0.60$). We report here on the pressure behaviour of both the structure and superconductivity of these new superconductors. We find strong evidence for the passage under pressure from an underdoped to an overdoped material, and as in cuprates superconductors the superconducting transition temperature follows a parabolic dependence with charge carriers density.

3.12 PARTIAL SUBSTITUTION OF COPPER BY TIN IN La$_2$Cu$_{2-x}$O$_4$(S). THE La$_2$Cu$_{2-x}$Sn$_x$(O$_{4+x}$) SOLID SOLUTION. Georges Deign, Glenn Taylor and R. Lockwood, Concordia University, Department of Chemistry and Biochemistry. J. P. Brailovsky, Laboratory of Solid State Chemistry and Massonier Spectroscopy, and Laboratories for Inorganic Materials, Montreal, Quebec, Canada, and K. Gattes, Institute of Physics and Computer Science, Pedagogical University, Cracow, Poland.

One of the criteria for high temperature superconductivity in cuprates is the presence of excess oxygen in their structure. This occurs together with oxidation of some of the copper(II) to the trivalent state. In the present work, we have carried out oxygen enrichment by substituting some of the copper(II) by a higher oxidation state metal, namely tetramin tin. Tin has the convenience of offering a powerful local probe, the tin-119 nuclei, which opens up the possibility of studying specifically the tin site(s) of the massonier spectroscopy. It was found that up to 15% of copper(II) can be substituted by tin to give a disordered La$_2$Cu$_{2-x}$Sn$_x$(O$_{4+x}$) solid solution. Massonier spectroscopy showed that the tin site contains two distinct sites (IV) sites, that are crystallographically very different. One is a moderately distorted [SnO$_6$] octahedron, whereas the other much more distorted. Nearly octahedral coordination is expected for tin in IV in oxides, whereas the square pyramidal and the square planar coordination commonly found for copper[II] are unheard of for tin in IV in oxides.

3.13 CAXIS COPPER DOPATION IN THE YBa$_2$Cu$_{3-x}$O$_y$ SUPERCONDUCTOR BY THE ISOTOPIC DIFFERENCE PAIR DENSITY FUNCTION. Despina Louca, G.H. Kwei, Los Alamos National Laboratory, Los Alamos, NM, B. Dobrozemski, Z. Bukowski, Department of Physics, Northern Illinois University, DeKalb, IL.

The local atomic structure of the YBa$_2$Cu$_{3-x}$O$_y$ (YBCO) superconductor with isotopically pure Cu-63 and Cu-65 was studied. An X-ray diffraction study of the local lattice distortions to the superconducting transition is provided. The analysis was done using the neutron differential pair density function (DPDF) technique. The DPDF provides an atom specific PDF, enabling the distinction between overlapping atom correlations in complicated systems such as the present one. It utilizes the contrast in the scattering amplitude enhanced by the different neutron scattering lengths for Cu-63 and Cu-65 in two isotopically pure samples with identical composition. The data are reduced for the local lattice distortions to the superconducting transition is provided.
Secondary lattice anharmonicities in the chains are also seen, but these arise mostly from defects in the vicinity of oxygen vacancies.

**IB.14**

**CRYSTAL STRUCTURE AND LATTICE DYNAMICS OF Nd$_x$Ba$_{2-x}$Cu$_3$O$_y$ SOLID SOLUTIONS.** Valery Petrykin, Maksim Kakholm, Materials and Structures Laboratory, Institute of Technology, Yokohama, JAPAN; Pedro Berestegui, Inorganic Chemistry Arhenius Laboratory, Stockholm University, Stockholm, SWEDEN; Sten Ericsson, Department of Inorganic Chemistry, University of Göteborg, Sweden, Neutron Research Laboratory, Uppsala University, Sweden.

Structure and lattice dynamics of Nd$_{1-x}$Ba$_x$Cu$_3$O$_y$ solid solutions (x = 0.0-0.9) were investigated using high quality ceramic samples prepared via chemical solution route. Crystal structure of newly reported insulating compound Nd$_2$BaCuO$_4$ was established from powder X-ray and neutron diffraction datasets. It was shown that the superconductivity appears as a result of Nd ordering in Ba sites of neodymium based solid solutions. The phonon assignment for this phase was carried out using results of lattice dynamics calculation based upon shell model for the refined crystal structure and experimental Raman spectra collected in different scattering geometry by micro setup. Structural information and Raman spectroscopy data were used for calculation of free energy of Nd$_{1-x}$Ba$_x$Cu$_3$O$_y$ solid solutions in various geometries of single crystal and room temperature. In addition, structural information, Raman spectroscopic data and chemical analysis was done in the analysis of possible scenario of superconductivity suppression in the neodymium based solid solution associated with the redistribution of holes between different orbitals in conducting planes. The overdoped phase and charge reservoir and conducting planes. This assumption was tested on another type of compound from the Bi-1223 family.

**IB.15**

**HOMOGENEITY AREA OF Bi$_2$Sr$_2$Ca$_{1-x}$La$_x$Cu$_2$O$_{8+y}$** R. V. Kochurov, I. V. Grashev, F. A. Besovtsev, Dept. of Chemistry, Moscow State Univ., Moscow, Russia.

Creation of precipitates in bulk material to act as pinning centers is a perspective way to improve critical current density in high temperature superconductors. It is possible to generate such precipitates via partial decomposition of superstoichiometric solid solution derived from a superconducting phase. To control this process one should go by a knowledge of stability field for these solutions. The objective of present work consists in determination of homogeneity area of Bi$_2$Sr$_2$Ca$_{1-x}$La$_x$Cu$_2$O$_{8+y}$ (R = Nd, La). Semi-quantitative XRD analysis was used to determine phase composition of specimens quenched from various temperatures. Annealing time appropriate to attain equilibrium in powder mixture was evaluated through kinetics measurements. It was found that lower temperature limit of stability field of Bi-2212 based solid solutions in multiphase mixture is 750°C. Monophasic solid solutions exist in relatively narrow temperature interval. This interval is significantly larger in the case of Nd$_{1-x}$La$_x$-2212. It stretches to x = 0.5 while temperature is within 825-850°C. Solubility limit of Nd corresponds to x = 0.75 at 850°C. In the case of Bi$_2$Sr$_2$Ca$_{1-x}$La$_x$Cu$_2$O$_{8+y}$ solid solutions the maximum substitution with La is x = 0.3 and x = 0.5°C. Features of homogeneity fields for both solutions are discussed in terms of ionic radii of the dopants.

**IB.16**

**USING SOFT X-RAY SPECTRA TO DETERMINE VALENCE STATE OF TRANSITION METALS IN THE ELECTRON MICROPROBE.** Karoline Mueller, James K. Meen, Dan Elkon, Univ. of Houston, Dept. of Chemistry and Texas Center for Superconductivity, Houston, TX; Gene Ulmer, Temple Univ., Dept. of Geology, Philadelphia, PA.

The majority of methods for determining valence states of metals are indirect, microscopic, or both. We describe an electron microbeam method of direct determination of the valence state of first-row transition metals. The method uses relative intensities of peaks in the soft X-ray spectra of these elements. The pioneering work of Fischer (1964) showed that the La/La intensity ratio is a function of the valence state for first-row transition elements. More recently, Hofer et al. (1994) applied a similar technique to Fe in which excitation was by electron microbeam and the measured Fe-La intensity ratio was sensitive to the magnetic disorder in the Fe. This technique has the advantage of being applicable to all Fe-bearing oxides. Standard materials with similar chemical compositions and crystal structures to those of the unknowns must be used for calibration. The technique is applicable to all first-row transition metals. Examples of its application to oxides containing Mn or Cu will be emphasized.

**IB.17**

**THERMAL ANALYSIS AND THERMODYNAMICS OF (Cu,Sn)-PLUMBATES.** L. P. Cook, R. Klein and W. Wong-Ng, NIST, Gaithersburg, MD.

(Cu,Sn)-plumbates are thermodynamically important as a buffer of PbO vapor pressures in Pb-BSCCO high Tc phase assemblage. Using a vacuum thermocouple, detailed DTA/TGA studies of (Cu,Sn)-plumbates are being conducted. Simultaneously, solution calorimetric studies are being performed. Data on PbO pressures, heats of solution, and the crystal chemistry and compositions of phases participating in buffering reactions will be presented.

**IB.18**

**PHASE EQUILIBRIA AND CRYSTAL CHEMISTRY OF THE SrO-2O$_2$Cu SYSTEMS.** R. Lanthanides, W. Wang-Ng, J. Dinghong, T. Hougen, Q. Huang, L. P. Cook, H. Brown, NIST, Gaithersburg, MD; X. Chen, Chinese Academy of Science, Beijing, China.

In the past decade, a great deal of phase equilibrium research has been conducted on the Ba$_2$R$_2$O$_5$CuO$_4$ systems (R = lanthanide and Y) due to the presence of high-temperature superconductors such as Ba$_2$Re$_2$O$_5$Cu$_2$O$_7$ and Ba$_2$Re$_2$O$_5$Cu$_2$O$_7$. Substitution of the alkaline-earth element Ba by Sr may provide further insights regarding the crystal chemistry and phase equilibria of the high Tc phases. Although the analogs of the high Tc phases mentioned above were not found in these Sr systems under ambient pressure, the Sr-containing Sr$_4$Cu$_2$O$_4$ phase has been reported to have interesting properties, such as nanomolar microwave and magnetic properties, and Sr$_2$Re$_2$O$_5$Cu$_2$O$_7$ can become superconducting under high oxygen pressures. This paper investigates the trend of phase formation, and the crystal chemistry of various series of compounds, and subclass
phase relationships in the SrO-R$_2$O$_3$-CuO$_y$ systems. A comparison of these phase diagrams with the Ba-analogs will also be made.

**Bi. 20**

BOND-VALENCE ANALYSIS OF YBa$_2$Cu$_3$O$_{6+x}$. Isabella Natale Sorn, INFN and University of Brescia, Dept of Mechanical Engineering, Brescia, ITALY; Anthony Santoro, Qiang Huang, NSRRC, Center for Neutron Research, Gaithersburg, MD.

The molecular and magnetic structures of YBa$_2$Cu$_3$O$_{6+y}$ have been determined from neutron powder diffraction data (Q. Huang et al., Phys. Rev. B 45, 9611 [1992]). The nuclear structure was found to have an atomic configuration very similar to that of YBa$_2$Cu$_3$O$_y$. Bond valences were calculated from the observed bond lengths using the formalism and the bond valence parameters of Brown and Altermatt (Acta Cryst. B 41, 244 [1985]) and O’Keefe and O’Keefe (Acta Cryst. B 47, 192 [1991]). The results of these calculations showed significant changes of the bond valences sums of atomic pairs from the expected values (e.g., V(Ba) = 2.5 v.u. versus 2.0 v.u., and V(Y) = 2.6 v.u. rather than 3.0 v.u.). Since the structure was determined very precisely, with excellent agreement between observed and calculated intensities, these discrepancies cannot be attributed to errors in the experimental results, but rather may be due to structural strains. The bond valence analysis confirms this conclusion. In fact, the bond distances calculated from the theoretical bond valence [I.D. Brown, Zeit. Kristall. 159, 265 (1992); M. O’Keefe, Structure and Bonding 71, 161 (1989)] are incommensurate under the constraints imposed by the site symmetries of the atoms. Under these conditions the presence of structural strains is unavoidable. In the actual structure the two occupancy positions that minimize these strains and resulting in coordination polyhedra of the cations significantly stretched or compressed.

**Bi. 21**

ELECTRICAL TRANSPORT PROPERTIES OF BIPOLARONIC TlO$_x$ UNDER HIGH PRESSURE. Miguel Monteverde, Carlos Achs, Laboratorio de Bajas Temperature, Dept de Física, FCENY, Univ de Buenos Aires, Buenos Aires, ARGENTINA; Miguel Núñez-Requejo, CRIBIT, CNRS, Grenoble, FRANCE; Alisa Kuhn, Facultad de Ciencias Experimentales y Técnicas, Univ San Pablo Bosdalla del Monte, SPAIN; Miguel A. Alario Franco, Facultad de Ciencias Químicas, Univ Complutense, Madrid, SPAIN.

Motivated by the discussion about the bipolaronic nature of high $T_c$ superconductors, we have studied the electrical transport properties of TlO$_x$ (where $x$ varies from 1 to 3) and its phase transition characteristics, were identified as bipolarons. We measured the resistivity of TlO$_x$ twisted single crystal in as a function of temperature (at $T$) for a pressure range from 20 to 50 kbar. We used a conventional DC and a pulsed technique. Depending on the current direction and for P > 20 kbar we observed a semiconducting-like behavior with a $\rho \propto T$ dependence, which we interpret in terms of a metallic-like conduction. A phenomenological model was established to describe our results, that are discussed within the framework of bipolaronic transport of bipolarons scattered either by disorder or by interacting with other carriers.

**Bi. 22**

MAGNETIC CONTRIBUTIONS TO MICROWAVE LOSSES IN CUPRATE SUPERCONDUCTORS. S. Schilling, Z. Zhai, N. Hikim, P.L. Praglowski, C. Kiang, Northeastern University, Department of Physics, Boston, MA.

The origin of the absorption of microwave is of importance from fundamental viewpoints as well as for applications. Despite the very strong evidence suggesting a skew wave parameter from a variety of experiments, the microwave data are not available in their entirety in terms of a pure $\omega$-wave parameter, or even one with mixed $\omega$ dependence. We have carried out extensive measurements on a variety of cuprate (superconducting and non-superconducting), nickelate and manganate oxides. An important theme that emerges from the data is the presence of peaks in the microwave absorption with varying frequency. The presence of similar peaks in superconducting and non-superconducting materials strongly suggests that they are magnetic in origin. This is because the parent compounds for the cuprates and nickelates are antiferromagnetic insulators. Hole doping leads to an incommensurate state with separation into magnetic and charge stripes. The formation of stripes does lead to peaks in the absorption in $La_{2-x}Sr_xNiO_4$. These results therefore suggest that in the superconductors, the superconducting contribution is overwhelmed by a magnetic contribution. Support for these observations from measurements on YBa$_2$Cu$_3$O$_{6-x}$ with varying $O$ concentration and in $H$: $H = 1233$ and $H = 1200$ is discussed. We thank A. Erb (U. of Geneva), A. Revcolevschi (U. Paris-Sud, Orsay), D. Colson (Saclay), A. Maignan (Cen) and S. Cheong for providing samples used in this work. Work supported by NSF-9711100 and AFSOL-9710000349.

**Bi. 23**


Bulk samples of the presumed hybrid ferromagnetic-high T$_c$ superconductor $\text{R}_2\text{Sr}_2\text{Cu}_3\text{O}_8$, with different R's, have been synthesized by solid state chemical reaction. TEM and powder XRD show that the $\text{R}_2\text{Sr}_2\text{Cu}_3\text{O}_8$ compound is the dominant phase. The highest zero-resistance temperature measured via four-probe resistivity is close to 40K. 99Rho Moshaver et al. measurements at 4K reveal a highly anisotropic ferromagnetic peak of around 70T. This result suggests the use of the R$_2$Sr$_2$Cu$_3$O$_8$ compound without any additional impurity to use an internal magnetic field in a high T$_c$ superconductor.

**Bi. 24**

RUTILE REFLECTIONS OF [R bilayer] (R=Ho, Dy, Gd, EU, Sm, Nd). Eric J. Peterson, William L. Hults, Michael Simpson, Stephen R. Follin and Quieti Jia, Los Alamos National Laboratory Superconductivity Technology Center, Los Alamos, NM.

It has recently been shown by magnetic susceptibility measurements that enhanced flux pinning in bulk superconducting YBCO can be achieved by partial substitution of some rare-earth elements for yttrium. Rutile reflections of powder x-ray diffraction data obtained from these compounds suggest that substitution of Eu and YBCO control sample reveal trends in lattice parameters, atomic positions and non-uniform strain as a function of rare earth ionic radius. Non-uniform strain was a minimum in the non-substituted control sample and was found to generally increase with increasing strontium/rare earth ionic size ratio. Maximum strain was found in the samarium-substituted compound, which also exhibited the best flux-pinning behavior. The neodymium-substituted compound showed a dramatic relaxation in this strain, this compound also exhibited the poorest flux-pinning behavior of all of the compounds in the series. These results suggest that inducing strain by substituting large rare-earth cations for some of the relatively smaller yttrium cations may activate a flux pinning mechanism. The relaxation that is observed in the neodymium-substituted sample may be due to yttrium/barium-site occupancy disorder.

**Bi. 25**

STRUCTURAL CHANGES IN YBa$_2$Cu$_3$O$_{6+\delta}$ AND YBa$_2$Cu$_3$O$_{6-\delta}$ DUE TO PHOTODOPING. Lamme Ding, Trevor A. Tyson, New York Institute of Technology, Steven Tidrow, Army Research Laboratory.

YBa$_2$Cu$_3$O$_{6+\delta}$ (YBCO), is known to undergo changes in resistance when exposed to optical excitation. However the origin of the changes in resistance upon optical excitation is not well understood. In our experiments, the distortion of Cu$_x$O$_y$ chains was found to coincide with hole transfer seen in the absorption spectra of the photodoped (x=0.4) system. We have performed a systematic x-ray absorption study of samples in the insulating and superconducting regions. We find significant structural changes in samples from both regions, including the superconducting region. Structural parameters (bond lengths and bond correlations) are extracted to give a picture of the photoduced structural deformations. This research is funded by NSF CAREER grant DMR-9733862.[1] Tyson et al., Physics C 292, 163 [1997].

**Bi. 26**

THE INFLUENCE OF ANNELING ON SUPERCONDUCTING GLASS OF THE Bi$_x$S TERN SYSTEM OBTAINED BY MELTING-QUENCHED METHOD. Céfasio Luiz Carvalho, Keizo Yuki, Victor C. S. Reynoso, João C. S. Moraes, Universidade Estadual Paulista, Dept of Physics and Chemical, Paulista, Brazil; Paulo N. Filho, Group of Superconductivity, Universidade Federal de São Carlos, Dept of Physics, São Carlos, SP, BRAZIL.

Bi$_x$S ternary system has been studied intensively because a presents crystallographic phases with different transition temperatures: 2201, 2212 and 2223 are the principal phases with critical temperatures around 30K, 85K and 110K, respectively. The most important between them is 2223, but this phase requires long heat treatments, it is unstable and to solve this problem it is necessary to add Pb in the composition. Then, the ideal composition is known as BPSCCO (1:6:0.4:2:2:3). Different methods have been used to obtain this material like conventional, precipitation, evaporation method, etc, but all of them spend too much time mixing oxides, exist
prolong problems with the stoichiometry mainly with Pb because it is a volatile element. In this work, we used glass route and controlled atmosphere to obtain the Pb. After obtaining the material and submitted it at different thermal treatments, X-ray diffraction, energy dispersive X-ray, electric and magnetic measures were used to characterize it and we observed the presence of 2123 and 2223 phases for short and long time annealing, respectively.

**IE.27**

**LOW SUPERCONDUCTING ANISOTROPY AND HIGH DIMENSIONALITY OF (Cu–X)12n+1**. Yasumasa Tanaka, Kosuke Tanaka, Akira Iyo, Masaaki Tomono, Hideo Enna, Electrochemical Laboratory, Tsukuba, JAPAN and CREST of JST, Tokyo

The anisotropy and the dimensionality of the copper based superconducting cuprates (Cu–X)12n+1 (n = 1) was estimated by the normal state conductivity, temperature dependence of Hc2, torque measurement and flux-flow resistivity where X is Ti or C or vacancies or cation. The latter three among the four techniques had given the superconducting anisotropy. The estimated anisotropy was slightly varied between ~1.6 in the case of applying the second technique to Cu–1224 and ~12 in the case of applying the third technique to Cu–1223 depending on X and the techniques. The origin of the discrepancy and the reliability of the values is discussed because there are some conditions like dimensionality or anisotropy of X and Y and of the field given to the reliable value. We pay attention to the relationship between low anisotropy and high dimensionality and the properties of the charge reservoir layer. We discuss if the superconducting pair potential works between the holes resident at different CuO2 sheets or different superconducting blocks.

**IE.28**

**CHARACTERIZATION OF SUPERCONDUCTING MATERIALS AND THEIR CHEMICAL AND PHYSICAL FEATURES USING STANDARD REFERENCE MATERIALS**. Boris N. Kodess, V.K. Ouchterlony, A. Bateman, I.L. Kameel, VNIIGiS, Moscow, Russia; ICASA, Department of Chemistry, University of Pretoria, F.M. Reyonswalt, Division of Electron Materials, Donetsk, UKRAINE

There is a strong relation between physical properties and composition within the homogeneity region for both conventional (for example A15 and C15 compounds) and new high-Tc superconductors. Samples of the same composition of HTSC materials were measured dozens times. But the results were usually different even within one institution for micro and macro ranges of the length scale as well as for measurements of atom structure and electron density distribution. To minimize typical errors an interlaboratory experiment have been conducted on measurement of chemical composition, interplane distances, lattice parameters and impurities for a set of pure elements and compounds including superconducting silicide vanadium and yttrium cuprates. The X-ray data has been collected from a number of single crystal set-ups (12-16 laboratories) using single crystal and powder X-ray experiment was collected also for powder samples of Si, V and Y-Ba-Cu-O systems to determine their composition (mass ratios of major components and impurities) using various physical and chemical methods (more 30 methods of analysis). Such Standard Reference Materials and associated reference X-ray patterns provide high accuracy and establish basis for reproduction of experimental results. For example, details of kinetics and phase evolution of melt-textured samples were investigated for YBCO with high Tc and Tc. Precision experiment data yields fine details of distribution and/or arrangement of atoms of components of these materials, including sublattice of vacancies. As a result of series experiments we established chemical tendencies of change in properties if changes in composition or special treatment or external influence are applied. Also electron densities, charge transfer and atomic thermal vibration for each atom for all crystallographic units of stoichiometric and non-stoichiometric V3Si and YIn4Cu8O16 phases have been determined from diffraction data with a higher level of precision.

**IE.29**

**PHASE TRANSFORMATION AND DISTRIBUTION IN MECHANICALLY DEFORMED BSCCO SUPERCONDUCTOR TAPES.** Shen Li, Nanyang Technological Univ., Dept. of Materials Engineering; Hong Li, Hangzhou Normal Univ., Dept. of Chemical and Materials Engineering, NEW ZEALAND

Mechanical deformation has widely been used to produce microstructure to achieve high critical current density in Bi-2223 superconducting tapes. But the effect of mechanical deformation on the phase transformation in the BSCCO system has not been well understood. In the present research, the effects of mechanical deformation on the kinetics of Bi-2212 to Bi-2223 phase transformation has been investigated in detail. The results showed that the Bi-2223 phase abundance decreased with increasing mechanical deformation (thickness reduction) from 20-60%, and then increased with further increasing deformation ratios above 65%. The 60% deformation produced the lowest abundance of Bi-2223 phase. The phenomena can be explained with the different deformation energies that are dominant in the different deformation process. In the relatively low mechanical deformation regime (up to 60%), an increase in the deformation energy is required against the nucleation of the Bi-2223 phase. In the relatively high mechanical deformation regime (above 60%), an increase in deformation provides higher strain energy which results in the nucleation and phase formation of the Bi-2223 phase. The effects of mechanical deformation on the distribution of Bi-2223 phase abundance across the thickness of the tapes were also studied. The microstructure profiles were different for the tapes with different deformation ratios. For the tapes with high deformation ratios, the Bi-2223 phase abundance is maintained at a high level from the surface to a certain depth; while the concentration of the Bi-2223 phase dropped sharply to a low level underneath the surface in the tapes with a low mechanical deformation. It is suggested that the Bi-2223 phase profile is related to the grain alignment distribution and the density of the materials in the tape.

**IE.30**

**MAGNETIC ARRANGEMENT AND REVERSIBLE MAGNETISATION IN PLANE MAGNETIC FIELD OF Bi2Sr2CaCu2O8+δ, CuO, Cr2O3, CuO2, O2-NON-FERROMAGNETIC MATERIALS.** T. Tomita, J. Kibenge, S. Shindo, Honda Electric, Japan; Advance Electronic Materials Group, School of Physics, University of South Wales, Sydney, AUSTRALIA; R. Puzijn, Institute of Physics, Polish Academy of Sciences, Warsaw, POLAND; N. Koshihara, Superconductivity Research Institute, National Superconductivity Research Center, Tokyo, JAPAN

One of the most important intrinsic properties of high Tc superconducting materials is its extremely high magnetic fields. The magnetic properties of the plane of the crystals have not been known for lack of the large size single crystals. The large, high quality single crystals of Bi-2212 phase superconductors have been grown by floating zone method. The Bi-2212 single crystal of 5.3 x 0.4 x 3 mm3 were used in magnetic field of 0-2 tesla as the Bi-2212 phase was maintained to the high field of 1 tesla. The results show that the high field susceptibility of Bi-2212 superconducting transition temperature Tc increased with the increasing field. The high field magnetic measurements were used in the study of the crystal properties. The phenomena of the superconducting transition temperature in the layered superconductor were affected by a peculiar interplay between doping and pair breaking scattering.

**IE.31**

**RADIAL CHANGE IN THE APPROACH TO SEMICONDUCTOR-METAL SUPERCONDUCTOR TRANSITION IN SOLIDS.** Valery P. Kiselev, Institute of Solid State Physics, Chernogolovka, RUSSIA

Changed ion conductivity, electrostatic and electrical breakdown properties effects on the crucial role of deformation mechanisms in the electric properties of crystalline (CS [1]) and organic (OS [2]) solids. The approach allows one to describe the superconductor transition (MST) through the increase of crystal work-hardening (WH) which decreases the plastic deformation losses for conventional and ion damaged solids. This lowered by the field of vitrification of CS, OS [1]. The works [3,5] directly evidence the strict correlation between the mechanical (WH, structural instabilities) and electrical properties of solids. The same features of the motion of deformation units in CS [9] and OS [7] confirm the universality of WH-mechanisms in CS and OS. The change of resistance in strained (due to hydrostatisca pressures, inhomogeneous phases, compacted powder) samples closely varies with the increase of softening of soft crystal and ion damaged solids [4,8]. The sharp increase of WH at low temperatures seems the sudden decrease in electrical resistance (the MST), the higher WH of crystal prompts the higher transition temperatures of low-temperature WH abundance and of the ion damaged solids and vice versa. The remarkable finding of this work is the scaling of mechanical [3,10] and electrical parameters of superconductors in
different scales of observation, which confirms the new approach to IMST.


13.23
Abstract Withdrawn.

13.24
Abstract Withdrawn.

13.25
THE NONCONTACT METHOD OF CRITICAL CURRENT DENSITY MEASUREMENT IN HTSC SINGLE CRYSTALS.
Kh. R. Rastam, Institute of Radio Engineering & Electronics, Moscow, RUSSIA.

One of the central tasks at synthesis and further applications of HTSC single crystals in the applied purposes is the maximum precise and quick measurements of their critical currents. We design a simple method for measurement of the critical current density in HTSC single crystals of the arbitrary form, according to which the Jc value is determined on measurements of axial allocation of an entrapped magnetic field from a sample. According to our research which have been carried out on YBa2Cu3O7-δ and Bi2Sr2CaCu2O8+δ single crystal samples of the various forms and sizes, density of a critical current in them can be exactly defined by the expression: 

\[ J_c = \frac{\text{constant}}{B_{Bc2}B_{Bc1}} \] 

where \( B_{Bc2} \) is the value of an external magnetic field B, at which \( B_{Bc1} \) reaches saturation, \( B_{Bc2} \) is the first critical magnetic field of a sample, h - the thickness of a sample, R - reduced radius of a sample c - the speed of light. The value \( B_{Bc2} \) was determined from the dependence \( B_{Bc2} \) on B for the mode ZFC and \( B_{Bc2} \) was determined at the moment of detection of a non-zero B0 signal by the sensor located near to a central part of the sample surface. In the measurements we utilized the Hall sensor with a size of the working area 0.15 on 0.45 mm with sensitivity 10 mA/V. The compensating circuit allowed us to register fields with 10⁻⁶ Gs accuracy. Usage of more responsive sensor of a smaller size will allow to raise accuracy of definition of the value Jc.

13.34
Abstract Withdrawn.

13.35
MODEL FOR SUPERCONDUCTOR STRIP SURFACE-BARRIER AT A LOW FIELD. D. Agassi and R.J. Cullen, Naval Surface Warfare Center, Carderock Division, Bethesda, MD.

Flux pinning and entry/exit to/from a thin film in the presence of a weak magnetic field is controlled by the film's surface barrier. This barrier depends on the film's intrinsic physical parameters and, more importantly, on its geometrical dimensions. For the one-dimensional configuration of a field parallel to a semi-infinite superconductor planar surface, this barrier has been discussed a while ago. On the other hand, for a thin superconductor film, where the external-field lines wrapped around the film imply a two-dimensional geometry, the analysis is more involved. By adopting a new approach, a divergence-free expression is obtained for the underlying screening current. The results are compared to alternative approaches which entail a divergence at the film's edges. With regard to the screening current distribution at the film's surface, comparison with the 'canonical result' shows that the new approach compares well with the latter provided the evaluation point is not too close to the film's edges. The associated surface barrier, however, entails a new qualitative feature which will be presented and discussed. A corollary is an expression for the film's vertex penetration-field and the total screening current carried by half of the film.


13.36
INDUCED SUPERCONDUCTIVITY AT LONG DISTANCE AND PROXIMITY EFFECT IN SN’’ STRUCTURES.

A. Giana Giosche, M.J. Holcomb and W.A. Little, Stanford University, Stanford, CA.

We consider a SN’’ structure where, under certain circumstances, the Andreev resonances between electrons and holes at the S/N interface can be manipulated to yield a resonance in the superconducting pair amplitude in N’’ (thereby becoming S’’). This phenomenon leads to interference effects in the current-voltage characteristics, similar to multiple beam interference in optics. Between the Andreev reflected particles at S’/N’ and at the N’/S’ interfaces. We show that the relevant characteristic length is not the superconducting coherence length, as in the conventional proximity effect, but the much larger inelastic mean free path. The theory we have developed shows agreement with experimental results, and provides a more general framework for the study of the conventional, and long range proximity effects, as well as transport in SNS structures.

13.37
EFFECT OF SINTERING ATMOSPHERE ON THE WEAK LINK BEHAVIOUR OF YBCO SUPERCONDUCTORS.
Lokeshe Chandrapatik, S.K. Das, S.K. Mishra, National Metallurgical Laboratory, Jamshedpur, INDIA.

The transport properties of the superconducting materials are affected by the weak-link characteristics of the grainboundaries. The weak-link behaviour has been investigated extensively during last one decade. However, several aspects of the weak link formation is not been studied. In this paper the role of sintering atmosphere on the weak-link characteristics of bulk YBCO superconductors is discussed. The pellets were sintered at 960°C for 1h in argon, air and oxygen atmosphere and subsequently oxygenated at 500°C for 2h and cooled down to room temperature at the rate of 1°C/min. The weak-link behavior has been studied analyzing the critical current densities near the temperature. A detailed investigation of the chemical composition across the grain boundaries was carried out to understand the weak-link behavior. The variation of critical current densities near the sintering transition temperature indicated that the weak-link characteristics were changed from S-I-S to S-S-N-S and S-N-S at the sintering atmosphere varied from argon to air and oxygen respectively. As discussed earlier, SEM/EDS studies were carried out in the samples to understand the weak link behavior which showed a continuous variation of composition across the grainboundaries when the samples were sintered either in argon or air. Whereas, no variation of chemical composition was observed in the samples sintered in oxygen atmosphere. The widths of the grainboundaries were also found to increase with the decrease of pO2 in the sintering atmosphere. The EDS analyses indicated the presence of excess Ba and Cu in the grainboundaries of the samples sintered in air and argon atmosphere. From our earlier studies it was known that the sintering is enhanced with the decrease of pO2. The possibility of the formation of Ba2CuOx rich liquid phase at the grainboundaries of the samples is possible for the case enhanced for the sintering parameters of the samples and weak link characteristics of the samples.

13.38
SINTERING CHARACTERISTICS OF DOCTOR BLADE CAST YBCO-Ag TAPES. Lokeshe Chandrapatik, S.K. Mishra, National Metallurgical Laboratory, Jamshedpur, INDIA, Thin Film Lab, Dept. of Physics, Indian Institute of Technology, Delhi, INDIA.

The fabrication and subsequent processing of high temperature superconducting powders is an integral part for its possible application in industries. Amongst the several techniques, tape casting of ceramic powders by doctor blade is important and has been investigated extensively during last one decade particularly the Y-Ba2Cu3O7-δ (YBCO) superconductors. However, little effort has been made to study the sintering characteristics of these doctor blade cast tapes. In this paper, the binder removal, densification and grain-growth characteristics of YBCO-Ag (10 mol%) have been studied. Submicron-sized sintered YBCO-Ag (10 mol%) powder was prepared by a solution combustion technique using a chemical pyrophoric reaction. The powder was calcined and slurry was prepared by adding binder, plasticiser and deflocculating agent. The slurry was dried and subsequently tape cast by a doctor blade technique. The decomposition characteristics and the dimensional changes were investigated using thermal analyser and thermomechanical analyser (TMA). The tapes were sintered for 1h at temperatures ranging from 860°C to 940°C. The weight loss and properties of the sintered tapes were also measured. The superconducting properties of the sintered tapes were measured after oxygenation at 500°C for 5h. Thermal analyses and TMA studies indicate that the binder removal from the tapes leads to shrinkage of the tapes. The generation of volatile gases during binder removal and the subsequent flow of these gases through capillaries has lead to shrinkage of the tapes. Anisotropic shrinkages
of the tapes during binder removal is also observed. The sintering studies of the YBCO-Ag tapes showed two stages of sintering and grain growth. The apparent activation energy of sintering has been estimated to be 57.2 ± 1 kJ/mol below 800°C and 327 ± 6 kJ/mol above 800°C. Whereas, the apparent activation energy for grain growth is estimated to be 252 ± 1 kJ/mol below 800°C and 183 ± 2 kJ/mol above 800°C. This has been observed to enhance after attaining 80% of the theoretical density.

IE.30 COMPARISON OF THE MICROSTRUCTURE TO THE CRITICAL CURRENT DISTRIBUTION UNIFORMITY ACROSS JOSEPHSON JUNCTIONS. Michael W. Cremo, Argonne National Laboratory and Northwestern University; K.L. Meekle, Argonne National Laboratory; L.D. Marks, Northwestern University.

Critical current vs. applied magnetic field measurements were measured across 104 degree, 45 degree and interface engineered ramp-edge YBCO Josephson junctions. The critical current distributions along the length of the boundary were calculated using a phase retrieval algorithm. The uniformity of the various current distributions are discussed in terms of microstructure features along the boundary analyzed by high resolution electron microscopy.

IE.40 MEASUREMENT AND ANALYSIS OF HARMONIC POWER FROM YBaCu3O7−δ MICROWAVE RESONATORS. N using the fact that the critical voltage characteristics and the resistive transitions follow the KT prediction for both the YBCO/STO and the YBCO/buffer. The comparison between the theory and the experiment allowed us to calculate the superconducting carrier density, the vortex dielectric constant, and the effective penetration depth. The KT parameters were found to be $T_{KT}=34.8$ K and $T_{KT}=55$ K for the YBCO/STO and $T_{KT}=55$ K for the YBCO/buffer. A transition width gives information about the changes of vortex-antivortex interaction with pair scale. Significant difference in $T_{KT}$ values for the YBCO/STO and the YBCO/buffer we believe to indicate the absence of the vortex pairs with a larger separation distance in the YBCO/buffer; so pair breaking starts at higher temperature than for the YBCO/STO. Indeed, an ultrathin YBCO deposited on YBaCu3O6 buffer we found experimentally to have a lot of voids with a typical distance among them of about 100 nm. This appears to prevent the binding of the vortex-antivortex pairs with a separation larger than 100 nm. [1] G. Grekov, et al. Physica C, 286, 18-24 (1997).


Irradiation with high energy heavy ions is a powerful and well known tool to study the mixed state, mainly phenomena related with pinning. The effect of irradiation by lower energy and lighter ions produces a very different scenario. In this case, the main effect could be due to Oxygen disorder, mainly in the chains. The YBaCu3Oy films were obtained by dc magnetron sputtering in pure Oxygen pressure of 30 mTorr and followed isothermally for 1 hour at 500°C. The results of the measurements with an applied field of 50 mTorr. The films were irradiated with He ions, using a microelectronic ionizer, at a dose of 4 $10^{13}$ cm$^{-2}$ and 80 keV, at 7° to avoid channeling. Due to the irradiation the $T_{c}$ is decreased from 90 K up to 62 K [1]. Lebedev, K. Verbit, G. Van Tendeloo, EMAT, University of Antwerp (RUCA), Antwerp, BELGIUM.

Segregation and competition of Y-based impurities in Y-rich biaxial YBCO samples is investigated. The analysis of distribution and on the structure of the precipitates is performed by high resolution Transmission Electron Microscopy. Y2O3 precipitates are found in YBCO films grown on [110] SrTiO3 substrates. On the other side, Y2BaCu3O6 precipitates are found in YBCO films deposited on an intermediate [110] MoO3 seed layer. Agglomeration of Y2BaCu3O6 precipitates is observed near the edge in the MoO3 seed layer. The experimental data is discussed in context of phase competition, nucleation, epitaxy and surface migration in the YBCO sintering process. It is deduced that the charge of superconducting and surface energy, also governed by the favorable epitaxy of the secondary phase with the substrate or the YBCO matrix, plays a major role in determining the segregation of either Y2BaCu3O6 or Y2O3 precipitates.

IE.45 SYNTHEMIC VARIATIONS OF ELECTRONIC STRUCTURE IN Bi2212 SINGLE CRYSTALS WITH Pb- AND CARRIER DOPING LEVELS. J. Shimizu, T. Shimoyama, Y. Morii, M. Yoneda, K. Otsuchi, K. Kitazawa and K. Kishio, Department of Superconductivity, University of Tokyo, Tokyo, JAPAN.

We have reported that the flux pinning properties of Bi2212 single crystals were systematically improved by increasing Pb-doping level up to its solubility limit, ~25% of Bi site [1, 2]. The introduced inhomogeneity corresponding to the distribution of Pb atoms is believed to contribute to the enhancement of pinning force in the present structure. Hughes Network Systems, Germantown, MD; present address: Philips Research Laboratories, Eindhoven, THE NETHERLANDS.

The effect of silver doping on the power handling capacity of YBa2Cu3O7−δ thin films is investigated by both microscopic (imaging) and macroscopic measurements (device characterization) techniques. C nanoparticle films [5.5 GHz] are fabricated using PVD-grown YBa2Cu3O7−δ targets with different doping levels of silver. Nonlinearities are characterized through intermodulation distortion (IIM) of two nearby pure tones. Spatially resolved IIM measurements are performed using a cryogenic ac millimeter wave microscope, and the results are compared to the macroscopic (device level) measurements. The influence of the silver doping is studied for both types of IIM observations.
SESSION II/4.L2/02: JOINT SESSION: BIAxially TEXTURED SUBSTANCES FOR HIGH-TEMPERATURE COATED CONDUCTORS

Chairs: Ron Feenstra and James M. Harper
Tuesday Morning, November 30, 1999
Room 200 [H]


Progress made in the fabrication of Rolling assisted biaxially textured substrates (RABiTS) and epitaxial deposition of HTS on such substrates is reported. Significant progress has been made in the fabrication of non-magnetic, strengthened, biaxially textured metal templates, deposition of oxide and other buffer layers and in the fabrication of long length substrates and superconductors. Ni-Ge alloy substrates fabricated using thermomechanical processing show a single orientation cube texture (~100%) with sharp in-plane and out-of-plane textures, essentially identical to that obtained for pure Ni. High Jc NiGe (~1 MA/cm²) substrates have been demonstrated on epitaxially grown YBCO films on RABiTS using Ni-Ge as the starting template. Tensile tests and magnetic hysteresis and susceptibility measurements show that the substrates have greatly reduced magnetic properties compared to that of pure Ni, and are significantly stronger. In this area of buffer layer development, significant progress has been made in the formation of single orientation oxide buffer layers on Ni using sol-gel processes. A variety of ReO₂ type materials have been fabricated in this manner. The buffer layers are dense and crack-free and electron backscattered diffraction patterns show that the films have a high crystalline quality. High Jc's exceeding 1 MA/cm² have been demonstrated on such substrates using intermediate vapor deposited buffer layers. Progress made in the area of long length deposition using both vapor deposition and sol-gel will be reported. Efforts are underway to fabricate longer length superconductor samples exceeding 1mm and results obtained will also be summarized.


9:00 AM  *II/4.L2/02.2  INCLINED SUBSTRATE DEPOSITION BY EVAPORATION OF MAGNESIUM OXIDE FOR COATED CONDUCTORS; Markus Bauer, Ralf Meister, Robert Semenzau, Paul Berberich, Helmut Kienle, Technische Universität München, Physik Department, Garching, GERMANY.

Thin films of YBa₂Cu₃O₇ (YBCO) must be highly textured in order to have good superconducting properties. One way to achieve this is to deposit textured buffer layers on arbitrary polycrystalline substrates by inclined substrate deposition (ISD). This was first proposed by Hasegawa et al using pulsed laser ablation. We use evaporation techniques to make the ISD process scalable to large areas and high production rates. Buffer layers of MgO are deposited at very high rate either by e-beam or simply by thermal evaporation onto substrates of Hastelloy or stainless steel inclined by typically 40°. This leads to columnar growth with biaxial texture, improving with thickness up to 2 μm. YBCO films grown on these buffers are highly textured with (103) around 8°. The Cu₂O planes of the YBCO are typically tilted with respect to the normal direction of vapor incidence. Therefore the critical current density in (100) is anisotropic, with lower j|| along the vapor direction and twice as much across. This direction must be chosen along the tape for coating. The highest j|| are obtained for YBCO films of 2 μm², resistively measured. To understand the texturing mechanism we have carried out Monte Carlo simulations. These confirm the columnar growth modelled. We find that this mechanism is likely to yield order of magnitude improved texture compared to the effects, namely bimodal hopping of the particles due to their initial momentum, and mutual shadowing of the columns selecting the fastest growing orientation. Issues of magnetic anisotropy, production rate, upsizing of tape length, and cost will be addressed.

9:30 AM  *II/4.L2/02.3  ION BEAM-INDUCED GROWTH STRUCTURE OF FLUORITE-TYPE OXIDE FILMS FOR BIAxially TEXTURED HTS COATED CONDUCTORS; Yasuhiro Hijima, Masako Kikuma, Takashi Satoh, Fujikura Ltd., Material Technology Lab., Tokyo, JAPAN.

The achievement of sharp biaxial alignment of Yttrium Stabilized Zirconia (YSZ) films by off-normal ion-beam-assisted deposition (IBAD) produced a hopeful application as flexible HTS coated conductors using metallic substrates. Quite high-Jc values were successfully achieved by removal of intergranular weak links in Y-123 films on the YSZ templates. Till now 2.3cm length Y-123 tapes were fabricated using random polycrystalline Ni-based alloy tapes coated with textured YSZ layers. Ar⁺ ion bombardment had significant effects on the crystalline structure of the YSZ films; to align n<100> axis with the substrate normal, and m<111> axis with the bombarding beam axis. These two effects were induced simultaneously at room temperature and resulted in peculiar biaxially textured structure without epitaxial relationship to substrates. This paper discussed the alignment mechanism with the structural properties of several fluorite or related type oxide films including YSZ, CeO₂, Y₂O₃, etc., based on several proposed models. Films were formed on polycrystalline Ni-based alloy by dual ion beam sputtering method. Textured structures were characterized with X-ray diffraction (XRD), transmission electron microscopy (TEM), atomic force microscopy (AFM) etc. Peculiar structural evolution of the crystalline orientation was observed and its development was well described by an exponential equation which agreed with Bedworth's selective growth model. It could be explained as a collaboration among in-plane and out-of-plane anisotropic growth of surface crystallites, and also homoeptaxial growth onto a crystalline surface beneath, both induced by Ar⁺ ion bombardment. Very smooth surfaces were observed by AFM imaging with a roughness of 23 nm and a peculiar ripple structure. The surface topographic structure was discussed by relating to Rosensweig's surface binding energy model without using ion channelling. Because the energy of the bombarding ionic species is as high as 1000-3000 eV, the origin of unusual aligning effect is still under controversy.

10:30 AM  *II/4.L2/4/02.4  BIAxially TEXTURED BUFFER LAYERS ON LARGE-AREA POLYCRYSTALLINE SUBSTANCES; H.C. Freyhard 1,2,3, J. Droz 1,3, S. Sievers 1,2, J. Hoffmann 1, K. Thiele 1, F. Garcia-Moreno 1,2, A. Usoskin 1 and Ch. Joas 1, Institut fuer Materialphysik, Universitaet Goettingen, Goettingen, GERMANY; 2 Zenit Zeiss Xerion GmbH, Zentrums fuer Funktionenwerkstoffe GmbH, Goettingen, GERMANY; 3 Kabelnetz Elektro GmbH, Hannover, GERMANY.

Biaxially textured buffer layers on polycrystalline metallic or ceramic substrates are required as templates for high-current-carrying HTS films, particularly coated YBCO films. In this contribution we report on our present understanding of the mechanisms governing the ion-beam-assisted deposition (IBAD) process employed for the preparation of textured YSZ as well as CeO₂ and Ga-doped CeO₂. On Ni, Hastelloy, SS as well as on ceramic tapes IBAD buffers could be produced with high-quality in-plane textures characterized by a FWHM of considerably smaller than 20°. Two systems were used, one with two cm spacer sources and a 21 cm Kaufman ion source for the spacing beam and a second one with 11 cm sources for spattering and the assisting beam. Furthermore, the IBAD process is now developed to an extend to allow for continuous tapes (up to 20 cm x 50 cm) with well textured buffer films. Maximum current densities of PLD-YBCO layers on IBAD-buffered substrates reach values up to 2 MA/cm².

In parts supported by the German BMFT, Kabelnetz Elektro GmbH and Siemens AG under grants 13 N 694/10 and 13 N 689/2 respectively.
11:00 AM *II.4.5/II.2.5/02.5

Ion-beam deposited metal was used to fabricate biaxially aligned cubic zinc oxide films on metal substrates. These films are used as templates for heteroepitaxially deposited YBCO films. The quality of the crystalline texture of the template films has a direct influence on the superconducting properties of the final YBCO film. We describe our efforts to fabricate high-quality templates on small-area substrates processed in sputtering mode and metal-oxide substrates processed in continuous mode. Cubic zinc oxide templates were deposited on the metal-oxide substrates and magnetic templates were deposited on the small-area substrates. Our best films, with FWHM values for the films on small-area substrates of 5.6 degrees for the template and 3.6 degrees for the overcoated YBCO. We will also describe x-ray scattering measurements of the topmost layers of the template films as well as TEM measurements of the film microstructure.

11:30 AM II.4.6/II.2.6/02.6
LEVEL SET SIMULATION OF ION BEAM ORIENTED MgO GROWTH. T. Balakrishnan, Dept. of Physics, University of Michigan, Ann Arbor, MI; Peter S. Smirnov, Dept. of Mathematics, University of Michigan, Ann Arbor, MI; David J. Socolov, Princeton Materials Institute, Princeton University, Princeton, NJ; Giovanni Russo, Dept. of Mathematics, University of L'Aquila, L'Aquila, Italy.

We have developed a general-purpose algorithm for the growth of faceted thin films from the vapor based upon the level set method. In the present simulation, we focus on the growth of polycrystalline MgO from the vapor in the presence of a low energy ion beam, which is used to establish in-plane texture. While out-of-plane texture [100] forms naturally, the ion beam selects grains which are oriented in a channeling direction with respect to the oblique beam. Growth rates of individual grains vary with grain orientation. We determine the polycrystalline microstructure, grain size and the width of the orientation distribution as a function of ion beam properties.

11:45 AM II.4.7/L2.7/02.7
QUANTITATIVE RHEE ANALYSIS OF BIAXILY-TEXTURED POLYCRYSTALLINE MgO FILMS ON AMORPHOUS SUBSTRATES GROWN BY ION BEAM-ASSISTED DEPOSITION. R.T. Brewer, J.W. Hartman and Harry A. Atwater, California Institute of Technology, Dept. of Applied Physics, Pasadena, CA.

We have developed a computer simulation based on analytic calculation of RHEED patterns in the kinematic approximation for mosaic polycrystalline films for given values of electron beam incidence angle, polycrystalline texture, in-plane orientation distribution, and grain size. Although RHEED is most accurately modeled using a dynamical scattering model, the computational efficiency of kinematical scattering lends itself to development of a model suitable for real-time control of biaxially-textured film growth by ion beam-assisted deposition (IBAD). Using the simulation results, we can quantitatively determine how RHEED spot shapes and relative intensities depend on the mosaic film characteristics. RHEED patterns taken at 15 keV with incidence angle in the range 1.5 degrees from 10 mm thick nominally [100] textured MgO films grown on amorphous Si substrates by IBAD were analyzed by comparing experimental RHEED spot shapes and relative intensities with those predicted by the simulation results. For some films, an additional 200 nm thermally grown MgO homoeoplastic layer was grown on top of the IBAD MgO layer. Results are also compared to X-ray rocking curve film analysis, and the quantitative correlation between biaxial texture and model-based RHEED analysis was discussed.

SESSION III: PHASE EQUILIBRIA, THERMODYNAMICS AND KINETICS
Chair: Winnie Wong-Ng
Tuesday, November 30, 1999
Room 200 (H)

1300 PM *III.1
SUBSTITUTION FOR Ba By Pr, La, AND Eu IN Equi[Re1-xjcujuO4], SOLID SOLUTION. R.W. McCullagh, K.W. Dennis, A. O'Connor and M.J. Kramer. Ames Laboratory, Iowa State University, Ames, IA; Yousen Xu, University of Minnesota, Minneapolis, MN; S.K. Malik, Tata Institute of Fundamental Research, Bombey, INDIA; W.B. Velez, University of Missouri, Columbia, MO.

Except for the Pr1-xBaxCu2Oy (Pr123)40, all other light RE1-yBaxCu2Oy (RE123)40 exhibit superconductivity with varying degrees of Ba substitution on the Ba site. Previous study of Nd123a system has shown that Pr is a well-behaved intercalated ion when nominally substituted for Ba. Neutron diffraction results demonstrated that Pr substitution in Nd123a was random on the Nd site, partially on the Nd site and partially onto the Ba site. To try to differentiate the effect of charge from structural distortions arising from Ba substitutions on the Ba site, we chose a solid solution based on Equi[Re1-xjcujuO4], RE = Eu, La, or Pr because Eu has an effective size (0.95 A) considerably smaller than that of trivalent Pr [1.01 A] or La [1.06 A] unlike Nd whose ionic radii [0.85 A] is more similar to Pr, resulting in a tendency for Re to exchange. If ionic radii is the controlling factor for site preference, Pr and La should have a greater tendency to substitute for Ba rather than Eu. Neutron diffraction results show that the larger RE remains on the Ba site while Eu remains on the RE site. The contraction of the lattice with increasing c is systematic with ionic radii, the La showing the least sensitive and the Eu most sensitive. However, DC magnetization shows that the depression of Tc with c is less pronounced with La and more pronounced with Pr as compared to Eu. If the valence state of the RE on the Ba site alone is the sole factor in hole localization, there should be no effect of varying RE on Ba site in suppressing Tc. However, the site of the RE could come into play either due to non-random substitution or varying charge localization due to differences in RE-O bond lengths. Both of these hypotheses will be discussed in light of crystal chemistry and magnetic behavior.

2:00 PM *III.2
PHASE EQUILIBRIA AND MELT PROCESSING OF Re-Ba-Cu-O CERAMICS. Gernot Knölker, Wolfgang Biegler, Peter Schützle, Gunter Fuchs, TFW-Institute of Solid State and Materials Research Dresden, Dresden, Dresden, GERMANY.

The paper will present the thermodynamic approach to modifications of the melt processing for process large single grain RE-Ba-Cu-O bulk materials up to 2 inch diameter. The size of field larger than 1 T [at 77 K] and 9 T [at 44 K] on the top face of a YBaCuO cylinder, levitation force of 100 N at 77 K [20 N/cm2 related to the top face of the 25 mm SmCuO permanent magnet] and the critical current densities at 77 K of 45kA/cm2 [YBaCuO] or 70kA/cm2 [NdBaCuO] in absence of an applied field and the appearing 2 T applied field. Univarint equilibration define the framework for the process model which takes into account the growth of 123 phases by primary crystallization whereas 211 or 422 for Nd is dissolvated in a certain distance from the growing front. The entrainment of remaining 211 particles into the growing crystal can be considered by defining an effective distribution coefficient of 211 between the melt suspension and the solidified bulk.

Considerable alterations of the process route can be realized by choosing appropriate values for the thermodynamic properties, concentration activities, concentration independent. A solid solution RE1-yBaxCu2Oy is typical for RE = Eu, Nd, Sm due to partial substitution of Ba by RE ions. The superconducting properties [Tc(Tc)] deteriorate with increasing y. The expression of the stability field was found to be applicable to the experimental condition, optimal conditions for melt processing of these materials have been achieved either by controlling the composition by monocristal phase equilibrium [realized by admixing appropriate secondary phases] or by controlling P(O2).

It will be shown that phase stabilities and properties can also be influenced by annealing after growth as well as by chemical doping.

2:30 PM *III.3
PHASE FORMATION, THERMAL EQUILIBRIUM AND TEXTURING IN Ag/Bi(2223) AND Ag/Bi(123) TAPES. René Flikkger, Dept. Phys. Cond. Matter, Univ. of Geneva, Geneva, SWITZERLAND.

In Ag/Bi(2223) tapes, the phase relationships are strongly influenced by the heating ramp, the reaction history and the cooling conditions. A recent high temperature diffusion study at the high flux neutron reactor in Grenoble confirmed the formation of the Bi(Pb)2223 phase by nucleation and growth. During the cooling process, the phase Bi(2223) was found to remain stable, i.e. the phase transitions are almost temperature independent. At the same time, however, a newly formed, non aligned Bi(2223) phase was observed, as a result of the decomposition of the Bi(2201) and 14/19 phases. The consequences for the Jc values are discussed. We have recently found that the Ag content inside Bi(2223) filaments shows a continuous decrease with reaction time, correlated to an enhancement of Tc from 107.8 to 109.2 K after 200h in air at 838°C. The question is discussed.

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whether the decrease of Pb content could explain the reasons about
the various unsuccessful attempts to recover the Bi-Pb 2223 phase
after treating above the decomposition temperature: the solution of
this problem, involving a better knowledge about the formation
conditions, may lead to an improvement of jc. A study of texturing by
ODF and EBSD showed no correlation between the orientations of the
Ag sheets and of the Bi-Pb 2223 in the Ag sheath. In Ag-Re 123
systems, however, there is commensurability between the Ag surface
and the superconducting cell. A single orientation was found in
R.E. (123) layers, sputtered on both Ag single crystal and textured
Ag ribbons, but only for the [110] orientation. The best results were
obtained for Nd-123, where sharp lines with FWHM values of 3°
were observed. The specific problems to be solved on the way towards
higher Jc R.E. (123) layers on Ag ribbons are discussed with regard to the
thermal mismatch of the composite.

3:30 PM #15.7
SOLIDIFICATION PROCESSING OF RE-SYSTEM HIGH-Tc
SUPERCONDUCTING MATERIALS. Tetsuo Izumi, Yudhi
Nakamura, Takatoshi Umeda and Yuh Shishime, Superconductivity
Research Laboratory, ISTEC, Tokyo, JAPAN. #Department of
Materials Science and Metallurgy, Tokyo, JAPAN.

Solidification processing and crystal growth on HTSC are reviewed.
Continuous progress has been achieved and some of them have
recently led to the applications. In this paper, the progress of the
solidification processing, understandings of crystal growth mechanisms
and developments to applications are discussed. The solidification
processes of HTSC oxides are classified into two groups, which are the
growth from single crystallites and that from solutions. The peritectic
reaction with dissolution through the liquid for fabricating RE-123 crystals
was discovered in the series of research of unidirectional solidification
processes. This idea has been widely utilized for understandings of the
RE-123 growth mechanism. The pulling phenomena of RE121 particles at the crystal growth interface have been
observed firstly in the bulk growth, which have resulted in
changes for the non-steady state growth. The semicrystalline processes
are suitable for not only investigating the mechanism but also obtaining
higher Jc. Actually, the high Jc have been reported in the samples
grown by several different semicrystalline processes. On the other hand,
solution growth has been recognized to be much suitable for realizing
high quality grain boundaries where single crystals with higher crystallinity
were realized by optimizing the growth conditions in the crystal pulling.
The single crystal wafer is expected as the substrate for the electronic
device applications. LPE method is another process to obtain high
crystallinity. Recently, the self-refinement in the crystallinity during
the growth was observed. The preferential dissolution/growth of the grains
in the initial stage was responsible to the refinement. The LPE process
is also expected to obtain higher Jc values, which can be
realized by introducing micro-defects as a flux pinning center at substrate/film interface. This idea has been started to apply for the developments of the coated tape conductors. This work was supported by New Energy and Industrial Technology Development Organization.

4:00 PM #15.5
DIFFUSION OF Cu IN Ag SHEATH MATERIAL OF SSCCO
TAPES. Peter J. Majernik, Andre Aubele, Fritz Ackinger
Max-Planck-Institut fuer Metallforschung, Stuttgart, GERMANY.

The Ag sheath material of the tapes has been found to diffuse up to
0.3% Cu. Experiments with Ag/(Bi,Pb)2223 diffusion couples show that
the electromigration of Cu is high in Ag sheath. When Ag is alloyed with Mg the diffusion coefficient of Cu appears to be higher. As (Bi,Pb)2223 couples consist of about 80% of Ag this aspect is of great importance for the stoichiometry of the ceramic material, because during processing the Cu content of the ceramic decreases due to diffusion of Cu into the Ag sheath material. Experiments with Ag-B2212 diffusion couples show comparable results.

4:15 PM #15.6
THE EFFECT OF UNDERCOOLING ON THE GROWTH RATE OF NbTiCO GRAINS IN REDUCED O2 PARTIAL PRESSURE.
N. Hari Babu, HRC in Superconductivity, University of Cambridge,
Madingley Road, Cambridge, UNITED KINGDOM, W. Lo, Dept of
Mechanical Engineering and Texas Center for Superconductivity,
University of Houston, Houston, TX, D.A. Cardwell, Y. Shi, HRC in
Superconductivity, University of Cambridge, Cambridge, UNITED
KINGDOM

The growth rate of Nb2SnCu2O7 - 0.15SnNb2SnCu2O7 composite in a
1 % O2 in N2 atmosphere as a function of undercooling temperature has
been investigated in detail using isothermal top seeded melt growth.
The grain growth rate parallel to the (100) [001] planes, Rg, and Rh,
have been observed to increase with increasing undercooling. In addition, Rh
has been found to exceed Rg at all undercooling temperatures. Single, large grain NbTiCo superconductors have been
fabricated on the basis of this and complementary studies performed at fixed undercooling temperature from which the homogenous
nucleation of satellite grains has been eliminated.

4:30 PM #15.7
PHASE EQUILIBRIA OF Ag WITH Pb-BSCCO 2223 UNDER
VARIOUS OXYGEN Pressures. L.P. Cook and W. Weng-Ng,
Ceramics Division, NIST, Gaithersburg, MD.

The melting reactions of the Pb-BSCCO 2223 phase in the presence of
Ag are very complex, with several multiple reactions occurring over a
short temperature interval. These reactions are being studied
beginning with the initial appearance of liquid to the disappearance of
Pb-2223. Data on reactions and phase chemistries will be presented at
various oxygen pressures.

4:45 PM #15.8
PHASE RELATIONS IN THE BiO1_xSrO_xCaCuO_y SYSTEM.
Vance J. Styve, Marie-Laure Carvalho, Joey Gney, James K. Meen,
Don Elickson, University of Houston, Department of Chemistry and
Texas Center for Superconductivity, Houston, TX.

A systematic study of phase equilibria in the quaternary BiO1_xSrO_x
CaCuO_y is being conducted to determine chemographic relationships
between various superconducting and non-superconducting phases.
Previous research on superconductors within the system and its
subsystems have concentrated on maximizing modal content while
minimizing amounts of impurity phases. Complete knowledge of the
phase equilibria within key regions can play an important role in
determining the optimal conditions for synthesizing superconducting
materials. In order to fully understand the phase relations of the
quaternary, knowledge of bounding binary and ternary systems is
vital. Examining the subsystems shows that most of the phase volume is
occupied by primary phase volumes of alkaline earth oxides and
alkaline earth cuprates, leaving very little phase volume of
bismuth-bearing phases. This agrees with our previous study which
showed CaO (with minor amounts of disordered SrO) to be the liquidus
phase over the entire range of f(O2) for compositions of the
homologous series Bi12Sr23CaO33-y (y=0-3.33) includes three
known superconducting phases). Only for small m and high oxygen
fugacities are compositions in the primary phase volume of a
bismuth-bearing phase. Research is being conducted in ternary
and quaternary subsystems into the quaternary system to show that the
primary volumes of bismuth bearing phases are confined to a limited region
from BiO1_x to ~80% [Ca, Sr]O and ~20% CuO. We will discuss the
projection of the SrCaCuO-CaO and BiCaCuO ternaries into the
quaternary and examine supersolidus phase relations, modal
proportions of liquid and crystalline phases, and reaction relationships
of liquids and solids at various compositions, oxygen fugacities and
temperatures. Some recorded examples of melting experiments in a
heating stage will be demonstrated and relevant chemographic relationships will be discussed.

SESSION I16: CRITICAL CURRENTS
Chair: Stephen J. Pennycook
Wednesday Morning, December 1, 1999
Room 200 (I)

8:30 AM #15.1
CRITICAL CURRENTS IN EPITAXIAL YBCO FILMS: ISSUES
RELATED TO COATED CONDUCTORS. D.K. Christen, Oak Ridge
National Laboratory, Oak Ridge, TN.

Approaches to the fabrication of second-generation, coated conductor HTS wire involve the epitaxial deposition of YBCO films. These goals are similar to those of previous efforts to develop high-quality thin films for electronic devices, although the physical characteristics and properties required of coated conductors are somewhat different, mostly due to the need to economically achieve large overall current densities in the presence of substantial magnetic fields. Systematic studies of epitaxial films, deposited under a variety of controlled conditions that simulate the several less-than-ideal constraints posed by tape substrates and solderable deposition processes, can provide useful insights for understanding and optimizing the properties of coated conductors. Here we describe how individual isolated effects of low-angle grain boundaries, vicinal substrate surfaces, misoriented strains, and film growth rates, thickness, and processing approaches can affect the critical current densities. In some cases the resulting defect structures are beneficial, providing flux pinning sites that enhance the high-field transport properties. Results will be discussed in relation to their consistency with the observed properties of short-length coated conductors. Research sponsored by the USDOE under Contract No. DE-AC05-96OR22164 with Lockheed Martin Energy Research Corp.
TRANSPORT PROPERTIES OF BICRYSTALS AND GRANULAR COATED CONDUCTORS
Jan E. Evets, Dept of Materials Science and IRR in Superconductivity, University of Cambridge, UNITED KINGDOM

Grain boundaries in oxide superconductors are of central importance both for engineering applications and for issues in fundamental science. Although closely studied for more than a decade there is still no means to assess understanding the properties of individual boundaries or their collective behaviour in a granular material. The situation is made complex by the multiplicity of grain boundary types, the variation of grain boundary angle and facetted sub-structure, as well as the uncertainty effect of doping and impurities on the local electronic band structure. Detailed measurements of transport properties for individual grain boundaries are essential to elucidate common trends and characteristics that override this complexity. The range of available critical current and flux flow behaviour as a function of angle will be briefly surveyed and possible explanations for the scatter in results will be assessed. Measurements will be presented of critical current and flux flow characteristics for low angle bicrystal boundaries in the range of temperature, field and angle of the applied field. The results will be interpreted in terms of collective vortex channelling effects at the grain boundary. Finally the problem of current percolation and the voltage-current transition in a granular coated conductor will be addressed.

THE ORIGIN OF HIGH CRITICAL CURRENTS IN YB2BaxCu3O7-δ THIN FILMS
B. Dam, J. M. Huijbregts, F. C. Klinrnan, R.C.F. van der Geest, G. Doornbos, J.C. H. Rector and R. Griessen, Faculty of Sciences, Division of Physics and Astronomy, Vrije Universiteit, Amsterdam, NETHERLANDS.

The high critical current densities in YB2BaxCu3O7-δ thin films, point to the importance of strong pinning along extended defects. So far, however, it was not at all clear which type of defect is operative. Recently we found a clear correlation between the number of line defects as revealed by weak etching (not only the screw dislocations) and the characteristic magnetic field Bc1. The all films sofar can be characterized by a certain characteristic field Bc1 up to which junctions remain constant. While Bc1 is proportional to the linear defect density nld, we find no correlation with Jc. We can reproducibly change nld by changing the PLD growth conditions. It appears that in PLD-grown films there is a correlation between the island size and the defect density. The growth process also enhances a self-organised short-range order of the linear defects. The radial defect distribution function approaches zero at small distances which enhances the efficiency of these pinning sites. By repeated etching and Atomic Force Microscopy (using markers to identify a specific film area) we are also able to obtain a depth profile of the linear defect density. We find that in 15nm films more than 70% of the defects is formed during growth. It appears that the distribution length and the spatial distribution of the defects is similar in all films. By annealing the films after deposition we find that some linear defects are annihilated. As a result the both the density and the distribution of linear defects decreases with increasing substrate temperature. YB2BaxCu3O7-δ films thus offer therefore an attractive possibility to investigate vertex matter in a superconductor with tailored disorder. For technological applications the challenge is to find ways to increase the flux density, preferably keeping the short-range defects.

ADJUSTING GRAIN BOUNDARY TRANSPORT PROPERTIES FOR APPLICATIONS OF HIGH-Tc SUPERCONDUCTORS
H. Hilgenkamp, R.R. Schulz, C.W. Schneider, B. Goetz, A. Schmelz, H. Bielefeld, J. Männhart, Exp. Phys. VI, Electronic Correlations and Magnetism, University of Augsburg, Augsburg, GERMANY.

The electronic properties of grain boundaries play a central role in many applications of high-Tc superconductors. Over the years, basic electronic properties of the grain boundaries have been studied in great detail by numerous groups, but a comprehensive understanding of the mechanisms controlling their properties is still in development. Here, we point out the important influences of the dzy2 symmetry component of the order parameter and of space charge layers associated with it and bending at the interface [1]. These space charge layers can also alter the properties of grain boundaries for a given application, e.g. in large current carrying conductors or in electronics. The influence of the critical current densities and reducing the normal state resistivities of grain boundaries to unprecedented values, by appropriately doping the superconductor [2]. [1] H. Hilgenkamp and J. Männhart, Appl. Phys. Lett. 72, 1096 (1998); A. Schmelz, H. Bielefeld, H. Hilgenkamp and J. Männhart, to be publ. in Eur. Phys. Lett. (1999).

10:45 AM *IE.5
CRITICAL CURRENT AND LOW ANGLE GRAIN BOUNDARIES IN YBCO COATED CONDUCTORS
D.T. Verebelyi, R. Feenstra, A. Goyal and D.K. Christen, Oak Ridge National Laboratory, Solid State Division, Oak Ridge, TN; C. Provencio, School of Metallurgy & Materials, University of Birmingham, Edgworth, UNITED KINGDOM, P.N. Arendt, Los Alamos National Laboratory, Superconductivity Technology Center, Los Alamos, NM.

Biaxial texturing gives coated conductors the advantage of having primarily low angle (θ<10°) grain boundaries (GB). There is a consensus that high-angle, YBCO [001] tilt boundaries reduce Jc, exponentially with increasing misorientation angle. We find at 77 K, Jc(0), θ(0)~exp(-θ/θ0) for both critical GBs and grainy GBs. This is expected because the GB is disordered and therefore weakly-linked. Alternatively, low angle GBs have periodic dislocations separated by strongly linked material. This low angle regime would therefore be expected to have a weaker than exponential dependence on θ. We pursue this low angle region by analyzing the best YBCO films on NbN and RABITS, with Jc>1.5 MA/cm2. Electron backscattering images allow mapping of the GB microstructure sizes and a “threshold” value of θ is assigned where perveance current transport occurs along the simple. We find the Jc values for the coated conductors appear to also closely follow the exponential dependence on θ found for high angle GBs. Research co-sponsored by the DOE Division of Materials Sciences, the DOE Office of Power Technologies, under contract DE-A05-96OR22464 with Lockheed Martin Energy Research Corp.

11:00 AM *IE.6
THE FORMATION OF YBCO [001] TILT GRAIN BOUNDARY STRUCTURES AND THEIR EFFECT ON THE TRANSPORT PROPERTIES
J.P. Bulan, Y. Pan, N.D. Browning, University of Illinois at Chicago, Dept of Physics, Chicago, IL.

Atomic resolution scanning transmission electron microscopy (STEM) observations have shown that [001] tilt grain boundaries in perovskite structured systems show similar atomic arrangements. In view of the similarity between the bulk structures, i.e. they are not necessities, this may not be surprising, but it does suggest that the boundary structures may be controlled by the same parameters that control the formation of the bulk crystal. For perovskites, the bulk crystal structure can be formed from consideration of the close packing of the [111] planes. In the case of YBCO, the hexagonal arrangement of the [111] BxO and Y-O planes form packing layers that alternate with [111] O planes. By consideration of how these planes interact at an [001] tilt boundary and making allowances for strain and stoichiometry, the experimentally observed structures can be reproduced. The interaction of the close packed planes at the [001] tilt grain boundaries leads naturally to the formation of 2D reconstructions in the boundary core on either the Y-O or Ca-O columns in the [001] projection, and the under coordination of copper to oxygen. Whether this under coordination is interpreted as causing the formation of localized donor states or to a decrease in the hybridization between cobalt and oxygen, the net effect is a reduction in the number of charge carriers at the boundary. As the number of these reconstructed sites increases linearly with misorientation angle, the growth of this carrier depletion will also increase linearly with misorientation angle and creates a tunnel barrier for high-angle grain boundaries. Calculating the tunneling of the current across such a barrier using Ginzburg-Landau theory gives a good match with various experimental measurements of the criteria.

11:15 AM *IE.7
MICROSTRUCTURE IN YBCO COATED CONDUCTORS

A key to both understanding and optimizing YBCO coated conductors lies in understanding the microstructural detail of the oxide layers. The interrelationships among the choices of buffer layers, deposition method, physical properties and crystallinity are complex issues. This talk will summarize and compare the microstructural features we have observed in a variety of coated conductor samples with a view toward better understanding the critical current density and temperature dependences of the superconductor. The samples studied include YBCO deposited by both the barium fluoride precursor method and pulsed laser deposition on both IPAD and RABITS substrates and YBCO deposited on YSZ, CeO2 and Yb2O3 coated buffer layers. This work is supported by the University Partnership Initiative through Oak Ridge National Laboratory and the NSF MRSEC at the University of Wisconsin-Madison.

11:45 AM *IE.8
TRANSMISSION ELECTRON MICROSCOPY CHARACTER...

The benefit of in-plane grain alignments has been well documented in YBCO and other high Tc superconductor (HTS) bicrystals, in which high critical current density (Jc) has been observed across small angle grain boundaries. In-plane alignment can be obtained by providing a textured substrate, such as a buffer or substrates, ion beam assisted deposition (IBAD) technique. Current critical densities exceeding 1 MA/cm2 at 77K, zero field, have been reported by the Los Alamos team in these textured YBCO films. These findings illustrate the potential in controlling in-plane grain alignment to approach large-scale HTS applications. In this study, we have investigated the microstructure, composition, and grain boundary structure in YBCO coated conductors by transmission electron microscopy. Specifically, the local chemical, habit planes, misorientations, and dislocation structures of grain boundaries have been characterized. The effect of substrate roughness, choice of buffer layer, and processing parameters in affecting the grain size and grain boundary characteristics will be examined. Dislocations affecting the critical current transport will be discussed.

SESSION I7: COATED CONDUCTORS
Chair: Paul N. Arensd
Wednesday Afternoon, December 1, 1999
Room 200 (H)

130 P.M.  **I7.1**


For the past several years we have been engaged in the development of superconducting tapes for electric power applications such as magnets, motors, transformers and transmission lines. Among the variety of ways for constructing such coated conductors, the method of choice at Los Alamos is the YSZ deposited YBCO (YSZ-YBCO) on a flexible metal substrate with an intermediate textured layer of yttria-stabilized zirconia (YSZ) produced by ion beam assisted deposition (IBAD). In our efforts to improve tape performance, we have found that the achievable critical current (Ic) of active grain to reach a maximum value of about 200 A/cm of conductor width at a coating thickness of 1-2 microns. Additional YBCO beyond this thickness does not improve and can actually reduce Ic. To investigate, critical current density (Jc) was measured for samples with YBCO thickness values ranging from 0.39 to 6.3 microns, in this range, Jc decreased from over 2 MA/cm2 to about 0.25 MA/cm2. Several films were then thinned by ion-milling and remeasured with two significant results: 1) no supercurrent is carried at thickness levels greater than 2 microns; and for films thicker than 3 microns, Ic is drastically reduced near the substrate as well.

260 P.M.  **I7.2**


One approach to fabricating high-current superconducting wires involves the epitaxial growth of oxide buffer layers (YSZ/CoO) and YBa2Cu3O7 films on rolling-assisted biaxially textured Ni substrates (RA/TS). We have investigated the microstructure of these heteroepitaxial multilayers using synchrotron x-ray microbeam diffraction at the Advanced Photon Source. White and monochromatic x-ray beams were focused to sub-micron size, enabling high-resolution, grain-by-grain diffraction studies of the orientation and strain in each epitaxial layer. The use of x-ray microbeams provides measurements from a large number of different vicinal grains grown under identical conditions on a single sample; i.e. it enables a combined multiplicity of different growth temperatures (600°-800°C), we find that successive layers are not strictly epitaxial, rather, each heteroepitaxial layer exhibits a crystallographic tilt with respect to the underlying vicinal layer. The tilt angle typically depends linearly on the local miscut angle with a ratio that is consistent with a model based on elastic deformation at step edges. In contrast, growth at low temperatures (450°C), yields approximately aligned layers, suggesting that reduced kinetics suppress the influence of the step edges.

Martin Energy Research Corp. for the U.S. Department of Energy under contract DE-AC05-96OR22964. Supported by the Advanced Photon Source operated by Argonne National Laboratory.

2:15 PM  **I7.3**

PULSED LASER DEPOSITION OF YBCO THICK FILMS FOR COATED CONDUCTOR APPLICATIONS. Gregory Kozlowski, Rand Biggers, Iman Munsterte, Timothy Peterson, David Dempsey, John Jones, John Busbee, Richard Kleimait, Materials and Manufacturing Directorate, AFRL; Rahn Naink-Ranjan, John McDaniell, Paul Barnes, Charles Oberly, Propulsion Directorate, AFRL, OH; Mike Tsenic, Marvin White, and Aso Sarkar, Planstronic, Inc., Tipp City, OH.

Coated-conductor technology is being developed world-wide as a potential technique to process YBCO conductors for high current applications in high-magnetic field electric power devices at liquid nitrogen temperatures. Our approach to this task is to deposit biaxially textured YBCO onto buffered metallic substrates (Ni or Ni alloy) which have different degrees of in-plane and out-of-plane alignments by using pulsed laser ablation. The main goal of our effort is to maximize the critical current density of the coated conductors. Our best result so far is 2 x 10^6 A/cm^2 (equivalent to a current critical value of 55A at 77K) through a 0.3-μm thick YBCO film with a CeO/YSZ/CoO arrangement of buffer layers on a Ni substrate. CeO2 and YSZ have been deposited by using laser ablation with emphasis on optimal processing in terms of deposition rate and thickness of these buffers. Many issues are addressed to produce this important result. Among them, buffer-layer arrangement and characterization process in YBCO films are discussed. In addition, chemical and structural analyses (SEM, EDAX, X-rays, electron backscatter Kikuchi patterns) are provided to obtain a correlation between the physical and chemical properties of YBCO films.

2:30 PM  **I7.4**

CONVERSION KINETICS OF OXYFLUORIDE-DERIVED YBCO FILMS. Michael J. Cimaglio, Igor Selenev, and Merv Gopal, Massachusetts Institute of Technology, Dept. of Materials Science and Engineering, Cambridge, MA.

Epitaxial thin films of YBa2Cu3O7-δ [YBCO] high temperature superconductors have been prepared by metal organic deposition (MOD). Single crystal lanthanum aluminate and buffered single crystal [YBCO] were used as substrates for single MOD coatings. Kinetics of fluoride removal from oxyfluoride films during high temperature [T>700°C] heat treatment was studied at different P(O2)2, temperature, and moisture levels of the furnace atmosphere. Influence of buffer layer on fluoride removal rate during high temperature heat treatment was also investigated. Thin film YBCO coatings were derived from a trifluoroacetate precursor solution. Buffers for single crystal nickel samples had two-layer architecture and were made by the e-beam deposition. The precursor films were converted into glassy solid, heating to temperature 840°C under controlled conditions. Conversion to YBCO films is carried out at temperatures greater than 700°C while exposed to water vapor. These films were quenched after different times in varying amounts of water vapor. Quenched samples were then observed in a non-contacting acidiic liquid acid solution and the concentration of residual fluoride in these samples was determined by a fluoride selective electrode. It was shown that different levels of moisture strongly influence kinetics of conversion. Our observations also indicate that buffer layer had mostly no influence on rate of fluoride removal, during conversion into crystalline YBCO.

3:30 PM  **I7.5**


Bulk solution techniques have emerged as viable, potentially low-cost non-vacuum methods to produce long length conductors. In this technique, typically the precursorsolution is either spin-coated or dip-coated onto the substrate and heat-treated in a furnace to obtain dense crystalline film. By using the solgel alkoxide route, we have recently shown that buffer layers such as gadolinium oxide and ytterbium oxide can be deposited. The grown epitaxially on [001] YSZ textured-Ni substrates. To demonstrate the feasibility of producing meter lengths of the solgel buffered-Ni substrates, we have constructed a reel-to-reel dip-coating unit. This unit is capable of producing buffered substrates up to 10 meters per hour. We will present our results related to the scale-up of the solgel process. We will also discuss in detail about the choice of our buffer layers and their microstructures. Attempts will be made to grow YBCO films on these dip-coated buffers.
3:45 PM I17.6

Solution deposition of oriented coated conductors on RABIT or IBAD substrates is a potential method for rapid, low cost production of superconducting tapes. Deposition of YBCO(001)/SrTiO3(100)/YBCO structures has been accomplished on substrates including LaAlO3(100), RABIT Ni(200), and CeO2(200), with Jc [80K] 1 MAm^-2, and YBCO thicknesses up to 1.8 um by solution deposition. Strategies for effective integration of buffer layers and thick YBCO films will be presented, including understanding of intermediate phase formation and multi-layer processing. Structure property relationships, including buffer layer epitaxial quality effects on superconductor Jc and XRD properties will also be discussed. Sandia is a multiprogram laboratory operated by Sandia Corp., a Lockheed Martin Company, for the US Dept. of Energy under contract DE-AC04-94AL85000.

4:00 PM I17.7

We present an experimental study of growth mechanism and nucleation of superconducting properties of 1.5 um thick YBCO layers on flexible metallic substrates for coated conductor applications. We analyze factors influencing the growth kinetics and nucleation of thick, post-annealed films on CeO2 buffers. Nucleation of non-epitaxial YBCO grains and strain relaxation in the YBCO and CeO2 buffer is considered as a major limitation on Jc of these films. Two processes are assumed to be of importance for stable growth of c-axis oriented thick films: diffusion of H2O vapor down to the growth front and diffusion of HF to the film surface. A simple model of the film growth is proposed, which suggests possible ways of improving thick films critical current. Using a modified growth technique we obtained good properties for 1 and 3 um thick YBCO layers on RABITS tapes, Tc = 94 K, Jc (77K, 1T HIC) = 1.1 x 10^6 A/cm^2 for a 3 um layer on RABITS tape and 0.8 x 10^6 A/cm^2 for a 5 pm deposit. The transport properties of thick film samples are analyzed along with TEM and XRD data to establish a correlation between Jc and the structural features of the YBCO layer and the substrate-YBCO interface.

4:15 PM #17.8
LARGE AREA YBCO FILMS FOR RESISTIVE FAULT CURRENT LIMITERS. B. Ung, R. Nies, W. Schmidt, B. Seebach, H.W. Neumuller, Siemens Corporate Technology, Erlangen and Munich, GERMANY.

A very promising application of HTS is the resistive type fault current limiter (FCL) designed to enhance power quality in power distribution networks. Based on the superconducting transition of YBCO thin films, this device is a challenge especially to material science and engineering of oxide superconductors. While high critical current density of large area YBCO films are required for power levels above 1 MVA, the homogeneity of both Jc and thickness proves to be crucial as well. Poly crystalline YSZ substrates are coated at room temperature with a YSZ buffer bi-textured by ion beam assisted deposition (IBAD) to avoid using ion beam sputtering. The subsequent YBCO layer is grown at 650°C by thermal evaporation (TCE) of the metal constituents Y, Ba and Cu while periodically oxidizing the film in a high oxygen partial pressure pocket. Substrates up to 200x200 mm^2 in size can be coated simultaneously at YBCO growth rates of 1.05 Mm/min. The films are characterized in terms of crystalinity and morphology by XRD and SEM. The superconducting properties are mapped using a scanning Induction measurement setup. Key figures on HTS quality and FCL performance are presented.

4:45 PM I17.9

Electrodeposition offers a promising and economical approach for fabricating high-temperature superconducting (HTS) wire or tape for the Tl-oxide superconductors. The TlOxides, moreover, offer the potential for operation at 77K in practical magnetic fields of 3-5 T, which is supported by measurements on the irreversibility behavior of the Tl-1223 wire layer compound Pb, Bi, and Sr substitution. Along with Tl-1223, we are also investigating chromium doped TlOxide [1212] superconductors. Recent advances in the development of high-quality textured electrodeposited films on Ag coated single crystal substrates will be discussed. We are initially studying the processing conditions and proper buffer architecture using pulse laser deposition (PLD) technique to achieve high-quality textured films with high transport properties which will then be applied to the low cost laser deposition process. As an example, we are making two layers of electrodeposited films to increase the thickness and also the uniformity of the films. The transport measurement of the electrodeposited Th3Bi2Sr2Ca4Cu4O10 (1223) and Tl2CaSr2Cu4O8 (1213) films showed critical current density of about 10^5 A/cm^2 at 7 T in zero field.

SESSION 18: HTS FILM GROWTH
Chair: Ron Feenstra
Thursday Morning, December 2, 1999
Room 200 (H)

8:30 AM #18.1
GROWTH MECHANISM OF HG-HTS FILMS IN CATION-EXCHANGE PROCESSES. J.Z. Wu, Y.Y. Xie, T. Ayug, A. Gaspai, R. Aga, L.Y. Yan and L. Feng, Department of Physics & Astronomy, Univ. of Kansas, Lawrence, KS; D.K. Christen and J.R. Thompson, Oak Ridge National Lab; Oak Ridge, TN; M. Siegal, Sandia National Lab, Albuquerque, NM.

Epitaxial Hg-HTS thin films can be formed from Tl-HTS precursor films via Th-Hg exchange in a newly developed cation-exchange process. In contrast to the conventional thermal reaction process that necessitates the stringent requirement of thermal physical equilibrium, the cation exchange has been found to be a simple kinetic process that can be carried out in a large processing window, yielding significantly improved sample quality and reproducibility. The optimum processing temperature for the cation-exchange process is determined by the activation energy of the Th-cations, while the maximum, by melting temperature of the Th-HTS. Hg cations diffuse into the Th-HTS matrix, as suggested by surface microstructure and depth profile studies, through anion exchange, and then into Hg-HTS grains at atomic scales. While the diffusion rates for the two steps differ, the overall rate depends on the partial pressure of the Hg vapor applied during the cation-exchange process. As the cation-exchange process provides a simple and robust way of doping Hg-HTS grains, growth of Hg-HTS thin films and thick films on oxides and metal substrates has been studied and promising results have been obtained. Meanwhile, the cation-exchange process has been applied to fabricate Hg-HTS devices directly from Tl-HTS devices. This facilitates development of Hg-HTS devices by taking advantages of the matured Tl-HTS device technology.

9:00 AM #18.2
INTERVAL-PULSED LASER DEPOSITION OF REBaCuO. Dave H.A. Blank, Guss J.H.M. Rijnders, Horst Rogalla, University of Twente, Dept of Applied Physics, Low Temperature Division, THE NETHERLANDS.

With the introduction of the possibility to use Reflective High Energy Electron Diffraction (RHEED) at standard Pulsed Laser Deposition (PLD) conditions, it became possible to grow high quality films of materials under different oxygen and temperature conditions. In this contribution we applied this technique to study the growth of the high temperature superconductor, using modified etch treated single terminated Sc-TeO3 single crystal substrates. In addition to the RHEED oscillations another phenomenon is observed, typical for PLE. The pulsed way of deposition leads to discontinuities in the intensity of the diffracted pattern. This is caused by the mobility of the deposited material from the disordered distribution till an ordered one and leads to a characteristic exponential shape with characteristic relaxation time constants. These time constants give extra information about relaxation, crystallization, and nucleation of the deposited material during growth. From these results (intensity oscillations as well as relaxations), a new approach to deposit these complex oxide materials will be introduced. The basic idea of this so-called interval deposition is to deposit an equivalent of one unit cell of the material in such a short time that no relaxation in larger islands can occur, followed by a relaxation time before the next unit cell layer is deposited. This interval deposition leads to an imposed layer by layer growth. The latest results on the infinite layer structure as well as R_EbA_CuO superconductors will be presented.

9:30 AM #18.3

The program at Stanford University to develop the processes and the
material science of the high rate electron beam deposition of copper coated conductor consists of two parts. The development of new technologies to control the three metal fluxes in the presence of oxygen flux, and the material science of the high growth rate of a complex oxide like YBCO. The technology of flux control is Atomic Absorption using Tunable Diode Lasers. Atomic Absorption is also used to monitor the atomic oxygen flux, using a laser source. At the present time the effect is directed to the in situ process in which the three metal fluxes are co-evaporated along with oxygen, either molecular or atomic. The technology of the AA monitoring of the X and Y fluxes have been demonstrated — the Cu is under development. If necessary, the velocity as well as the number density are measured and combined to give the flux normal to the substrate. The material science issue concerns the requirement for both kinetic and thermodynamic stability. At rates above 10Å/s the oxygen needed for chemical composition exceeds the stability requirement. It is generally expected that oxygen fluxes in excess of this might restrict the mobility of the deposited material and prevent the growth of the desired c-axis grains with suitable dimensions and coupling for good superconducting current carrying. The experimental program is exploring this issue. Supported by DOE through ORNL-lookhead Martin, and by the 3M Corp.


In thin YBCO films pinning results from strong extended defects that form during the growth process. In the pulsed-laser deposition technique the pulse repetition rate used during film growth is reported to influence film morphology and crystallinity. We observed a significant difference in pinning properties of YBCO films grown by PLD using different deposition rates. YBCO films of the same thickness were grown under the same deposition conditions except for the pulse repetition characteristics. Pulse rates were varied between 0.1 and 100 Hz, and modulated in bursts separated by a time delay. All deposited films have high Tc, and a Jc [H = 0 T, Tc = 77 K] > 3 MA/cm². The data show a systematic suppression of Jc, in large applied fields for films grown at low pulse rates compared to films grown using repetition rates ≥ 2 Hz. The irreversibility field is also suppressed by more than 2 T for the films grown at lower rates. These data suggest that the time constant for some atomic scale agglomeration, disconnection, and diffusion processes occurring during film growth is the order of seconds, and that films grown at lower rates develop with a lower density of flux-pinning defects. We will present systematics of the current critical, pinning energy, and irreversibility line for films grown in different deposition regimes.

Research cosponsored by the DOE Division of Materials Sciences, the DOE Office of Power Technologies, under contract DE-AC05- 96OR22404 with Lockheed Martin Energy Research Corp.

10:45 AM *IBS.5 GROWTH OF SUPERCONDUCTOR-RELATED LAYERED RUTHENATES AND LAYERED TITANATES. D.G. Schlenk, J.H. Hafner, M.A. Subramanian, A.L. Ruoff, R. Jin, A.H. Maitra, Materials Science and Engineering, Penn State University, University Park, PA; W. Tian and X.Q. Pan, Dept of Minter Science and Engineering, Univ of Michigan, Ann Arbor; M. L.-N. Zou and Y. Liu, Dept of Physics, Penn State University, University Park, PA; G.W. Brown and M.F. Hawley, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, N.M. We have grown epitaxial films of layered ruthenates [Sr2RuO4, Sr2RuO4, Sr2RuO4, and layered titanates [SrTiO3, Sr2TiO3, Sr2TiO3, and SrTiO3] and studied their structural and transport properties. These layered perovskite phases are all in the Aurivillius family. The growth of Sr2RuO4 single crystals and doped Sr2TiO3 are well-known superconductors, but epitaxial films of Sr2RuO4 as well as the effect of reducing the dimensionality of Sr2TiO3 from 3D to 2D (i.e., Sr2TiO3) have never been reported.

The film synthesis conditions will be discussed in the context of the relevant phase diagrams and thermodynamic stability. The use of in situ growth monitors, specifically atomic absorption spectroscopy (AAS) and RHEED oscillations, to achieve these phases and Sr2RuO4, which have never been prepared as epitaxial films, will be described. By monitoring changes in the RHEED intensity oscillations as the strontium and titanium are sequentially deposited, the Sr:Ti ratio can be controlled within 1% of stoichiometry. Furthermore, the presence of a hint frequency in the intensity oscillation envelope allows the adjustment of the strontium and titanium fluxes so that a full monolayer of coverage is obtained with each shuttered dose of strontium or titanium. Crystals of Sr2RuO4 which are only 100nm thick are only 100nm thick.


Spin Ladder compounds are grown by molecular beam epitaxy under atomic oxygen pressure, using real time control by the RHEED intensity. The growth of these MCo3O4+Ax films is controlled on MgO and SrTiO3 substrates for M = pure Co, pure Sr or Sr/Cu, Sr/Ba. The detailed structure is studied by X-ray and HEED techniques. The structure of SrCo3O4+Ax is extremely close to the structure of the SrCu2O4 bulk compounds grown under high pressure by other teams. In both films and bulk compounds the ladder planes exhibit the same structure, while the stacking is slightly different. To our knowledge there is no other report for the synthesis of CaCu2O4+Ax spin ladders which seems to be difficult to stabilize as a bulk compound. In all these cases the films contain pure phases of two-leg ladders. The copper valence and the charge transfer were measured from X-ray Fluorescence near the L3 copper edge for these films and for Sr1+2CaCu2O4 and Sr1+2Ca2Cu2O4 compounds by purpose of comparison. According to the preparation process, the charge transfer of the films vary in the range 0.1 to 0.25, which are of the same order of magnitude as the values measured respectively for Sr1+2CaCu2O4 and for Sr1+2Ca2Cu2O4. The transport properties were of the SrCo3O4+Ax compounds show localization in agreement with previous value hopping, while the SrCu2O4 Spin Ladder is metallic for T>150 K and show localization below 150 K. The possibility will be discussed that the spin transfer is determined by the 3D structure and the disorder level.

11:30 AM IB.7 STRUCTURAL PROPERTIES OF BaCu2O2CuO2 INFINITELY-LAYERED SUPERLATTICES GROWN BY PLD. L. DeCaro, C. Giannini, M. Nascetti, L. Taglieri, Pavia CNRSM, Brindisi, ITALY, G. Balestrino, P. G. Meda, G. Petrocelli, INFN, Universita di Roma Tor Vergata, Roma, ITALY.

The stoichiometry and structural properties of BaCu2O2CuO2 and CuCu2O2 thin crystalline films and of (BaCu2O2CuO2) superconducting superlattices (SL), grown by the pulsed laser deposition technique on (100) STO substrates, are investigated by X-ray diffractometry and electron microprobe (EPMA). Simulations of the XRD reflexion (XRD) and X-ray specular reflectivity (XSR) data, considering the microstructural data, lead to an accurate structure refinement of the investigated films. By varying the structure factor and, consequently, the chemical composition of the hypothesized structure a precise and quantitative description of the structural configuration of the investigated SL was obtained. We found that the BaCu2O2CuO2 compound can be deposited in the infinite layer phase, even if the real structure can present many percents of copper vacancies in the CuO2 planes. In the BaCu2O2CuO2 layers of the investigated superconducting Sls the Cu:Ba ratio is less than one. Here, however, the copper vacancies are localized in the CuO2 planes, while in the BaCu2O2CuO2 they are present not in other CuO2 planes. Non-stoichiometric compounds with an excess of oxygen are compatible with the experimental data, indicating the BaCu2O2CuO2 layers as the reservoir charge block in the superconducting Sls.

11:45 AM IB.8 HOW MUCH CAN T, BE INCREASED IN TETRAXIAL Lu2−xSrxCuO4 THIN FILMS? J.W. Searle, J.P. Jerabek, L. Perre, J. Pompeynek, F. Seigwarr, P. Menzel, J.-P. Lecours, 1Université de Neuchâtel, Institut de Physique, Neuchâtel, SWITZERLAND, 2IBM Zurich Research Laboratory, Rüschlikon, SWITZERLAND.

The critical temperature Tc of a superconductor can be increased under hydrostatic pressure. This pressurized state is unstable and Tc returns to its ambient value upon unloading. In our current strain, the pressure induced in a film can be stabilized. A recent study showed that by optimizing the strain, Tc, of Lu2−xSrxCuO4 (214) thin films could be doubled to 49 K, compared to the bulk material. This increase of Tc, however, required the amount of strain [0.5%, i.e. compressive strain, whereas tensile strain decreases Tc. However, what are the limits of this Tc, change? The actual amount of strain is determined by the microstructure, e.g. structural morphology, defects not necessarily the applied strain and what limits the elastic deformation process, the influence of each component must be studied. We systematically investigated the structural and physical properties of 214 thin films grown on different substrates. In particular, we focus on the effect of the substrate material & its surface morphology and correlate the film...
microstructure with the changes in resistivity and T. Preliminary results suggest that about twice as much compressive strain to above could be induced locally in 214 films.

SESSION II: BSCCO PROCESSING AND PROPERTIES
Chair: Peter J. Majewski
Thursday Afternoon, December 2, 1999
Room 210 (H)

1:30 P.M. #IE.1
CURRENT ROADBLOCKS FACING B-2223 WIRE DEVELOPMENT
G. N. Riley, Jr., American Superconductor Corporation, Westborough, MA.

Conductors based on silver-sheathed B-2223 are enabling commercialization of High Temperature Superconducting wires. Since a breakthrough in their fabrication methodology in 1990, work has focused on improving the supercurrent carrying ability of B-2223 wires to levels required for practical application. Using a research approach based on understanding the relationships between processing, structure, and properties, and a process development approach based on statistical process control, superconducting performance levels sufficient for practical application have recently been achieved via a reliable manufacturing process. However, further performance improvements are required to enable broad scale commercialization of HTS wires. To further drive performance improvements, new research paradigms, mechanistic insights, and process solutions are required. In this presentation, the history of B-2223 wire development will be reviewed from the perspective of framing current superconducting performance “roadblocks” and identifying potential approaches for their elimination.

2:00 P.M. #IE.2
ON TEXTURE DEVELOPMENT IN HIGH-Τc SUPERCONDUCTORS
E. Cocchi, P.J. Ferreira and J.B. Vander Sande, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA.

Textured BSCCO High-Τc superconductors are studied and the various mechanisms for alignment of BSCCO grains addressed. So far, surface energy effects leading to texture development of BSCCO superconductors are considered. In this context with respect to the free surface of melt-processed Bi-2212 thick films. However, these previous considerations have not included the surface interactions between BSCCO crystals and other solid surfaces present in the BSCCO system. The present work attempts to analyze systematically the implications of these mechanisms through a simple formulism based on surface thermodynamic properties. A careful examination of the different stages involved during the partial melt process gives an explanation for the various observed phenomena. During the early stage of solidification, when the peritectic liquid is abundant, BSCCO crystals are rather mobile, facilitating their contact and interaction. As a result, if a crystal has its wide planar surface in contact with a foreign surface, the surface phase, free surface or another BSCCO crystal, it can minimize its surface energy and likely adhere to that surface. This mechanism, applied to a system with planar contacts like a 2212/Ag film, will result in a textured sample, depending on the thickness of the shearing superconductor layer. In a bulk sample however, BSCCO crystals may only minimize their surface energy by adhering to other BSCCO crystals, which consequently will form clusters of locally aligned crystals, with no long-range texture. In this fashion, we can also address the role of silver in promoting texture development in BSCCO superconductors.

2:15 P.M. #IE.3
THE PHASE FORMATION OF THE HIGH-Τc SUPERCONDUCTING Bi(Pb)-2223 COMPOUND STUDIED BY ELECTRON MICROSCOPY TECHNIQUES
Olivier Ehab, Department of Applied Physics, University of Tübingen, Tübingen, GERMANY.

The (Bi,Pb)2201-2212 phase formation has been studied in Ag-doped multilayered tapes. The samples prepared for the series were pre-characterized by the critical current density, ac-susceptibility and x-ray diffraction. The formation of the Bi(Pb)-2223 phase occurs at initial stages of the heat treatment, in which (Bi,Pb)12SnS2Ca2O8, alkaline earth cuprates and Pb compounds act as the precursor phases. These initial stages of the heat treatment were studied intensively by EDX in the SEM and TEM, electron spectroscopic imaging (ESI) and high-resolution TEM. The obtained data show that the growth of the Bi-Pb-2223 compound occurs in a two-dimensional way by transforming one or several atomic layers of composition Bi(Pb)-2212 into the composition Bi(Pb)-2223. Ca and Cu correlation diagrams obtained by quantitative EDX microanalysis are particularly useful for understanding the intergrowth phenomena, which is the typical microstructures in intermediate stages of the heat treatment, however, which is mostly ignored in x-ray diffraction studies. We determine the volume fractions of the secondary phases by EDX elemental mapping and subsequent quantitative multi-phase analysis by the concentration histogram (CH) method. Finally, these data are compared to the chemical composition of the Bi(Pb)-2212/2223 crystallites. In the microstructure of the totally processed tapes the Bi(Pb)-2223 crystallites contain 5-10% intergrowth lamellae of Bi(Pb)-2212 and the volume fraction of all secondary phases is about 4%, the intergrowth detectable fraction of secondary phases by the applied method is much smaller, around 0.1 vol%. With a combination of these methods a quantitative phase formation of the Bi(Pb)-2223 phase is achieved. A detailed model for the Bi(Pb)-2223 phase formation is presented which is based on the layer-by-layer growth and summarizes the microstructural findings. The current critical density of the tapes closely correlates with the Cu content fraction and the intergrowth fraction of the various mol fractions of the Bi(Pb)-2223 crystallites.

2:30 P.M. #IE.4
BSCCO LATTICES CONTAIN Cu(I). PHASE RELATIONS AND CONSEQUENCES FOR GROWING PHASE-PURE BSCCO SUPERCONDUCTORS
James K. Meen, Karoline Mueller, Vance J. Styve, Univ of Houston, Dept of Chemistry and Texas Center for Superconductivity, Houston, TX.

Most Cu-bearing crystalline materials that coexist with liquids in the Bi2O3-Sr-Ca-Cu-O system under oxygen contain Cu with a valence of at least two. Cu in a stable crystalline phase is at 1117°C in oxygen. Nevertheless, BSCCO lattices (and liquids in cuprate subsystems) contain Cu(I) as well as Cu(II) at temperatures well below 1117°C. Evidence for the presence of Cu(I) includes direct analysis of quenched glasses for Cu(I) and Cu(II) by isotope dilution and microbeam techniques, indirect determination of the Cu valence state by analysis for cations and oxygen, and chromatographic analysis of the CuO liquidus surface. The liquids for CuO when projected from a multicomponent system to pure CuO will, in this case, reproduce the melting temperature of CuO. The presence of Cu(I) requires that phase relations be considered in the quinary Bi2O3-Sr-Ca-Cu-O system. Liquids that have a composition of Bi2Sr4Ca2Cu6O14⁺ have a peritectic of CuO at 1117°C. Variation in p(O2) causes a liquid that has a Bi2Sr4Ca2Cu6O14⁺ composition to move along a line denoting variation in CuO. CuO If the composition line cuts the primary phase volume of Bi-2212 at some value of p(O2), the liquid will crystallize entirely to Bi-2212 at that value of p(O2) and a unique temperature. If, on the other hand, the line does not intersect the primary phase volume of Bi-2212 at an appropriate oxygen pressure, the liquid cannot crystallize entirely to Bi-2212. Similar considerations hold for Bi-2223 and other cuprates.

2:45 P.M. #IE.5
PRESSURE DEPENDENCE OF IRREVERSIBILITY LINE IN BSCCO
Mark E. Reeves, Department of Physics, The George Washington University, Washington, DC; Marc Raphael, Department of Physics, Catholic University of America, Washington, DC; Earl Shekon, Chris Kondzioni, Naval Research Laboratory, Washington, DC.

One of the important problems of high-temperature superconductivity is to understand and utilize the irreversibility in flux motion. We present the results of a new technique for measuring the pressure dependence of the transition to superconductivity in a diamond anvil cell. By measuring the third harmonic of the ac susceptibility, we determine the onset of irreversible flux motion. This enables us to study the effects of pressure on flux motion. The application of pressure changes interplanar spacing, and hence the interplanar coupling, without significantly disturbing the intraplanar superconductivity. Thus we are able to separate the effects of coupling from other properties that might affect the flux motion. Our results directly show the relationship between lattice spacing, effective-mass anisotropy, and the irreversibility line in Bi2Sr2Ca2Cu2O8⁺. Our results also demonstrate that an application of 5.5 GPa causes a four-fold decrease in the effective-mass anisotropy.

3:30 P.M. #IE.6
SPATIAL VARIATIONS IN COMPOSITION AND STRUCTURE IN B2223 MULTILAYERED TAPES
Terry G. Holsinger, John F. Binger, Los Alamos National Laboratory, Los Alamos, NM; Qi Li, Ronald D. Parrella, Martin W. Rugpich and Gilbert N. Riley, Jr., American Superconductor Corp., Westborough, MA.

Structural and compositional defects that may act as potential current limiting mechanisms (CLMs) were examined in Bi-2223 tapes from American Superconductor Corporation and in TEM (transmission electron microscopy) (TEI). Of particular interest in this work are the length scales at which the CLM’s would operate, correlations with transport.
properties, and the time scale for the development of CLM's during processing. Potential CLM's were divided into three categories based on whether they existed within a colony structure (mesoscale), or between different regions of the filamentary structure (macroscale). Particular attention was placed on two areas where complimentary work with low dose imaging and high-energy imaging [9] was done. Crystalline structures had shown relationships to the transport properties. Compositional studies in the TEM show heterogeneity in composition throughout the tape and in particular, lead depletion in Bi2223 near the leads. Electrolytic treatment of the tapes was used to identify the process steps during which CLM's developed within the tapes. The formation of crack structures and their partial healing near the final silver treatments were examined. Secondary phases present in the tapes during intermediate deformation are prime sources for formation of large cracks. Structures, localized crack structures were also found which results from the mechanical deformation.

6.00 PM 1B.7
OBSERVATION OF LOCAL PHASE CHANGES AT GRAIN BOUNDARIES IN Aг-SHEATHED BSCCO TAPEs.
Ryoji Takei, Nigel D. Browning, Dept. of Physics, Univ. of Illinois at Chicago, Chicago, IL

Grain boundaries contained within the superconducting filaments of highly textured Bi-Pb, Sr, Ca, CuO$_2$ Ag (Bi-2223)/Ag composite tapes are known to strongly influence the overall transport of large currents. Although several models have been proposed to describe the macroscopic current pathways in these wires, the underlying mechanism controlling the properties of individual grain boundaries has yet to be identified. In order to elucidate the exact role of grain boundaries in these tapes, systematic studies of the atomic structure and chemistry changes that occur at the boundaries are required. For this purpose, the microstructure of Bi-2223/Ag multifilament tapes have been investigated by scanning transmission electron microscopy (STEM). In particular, use of the high-angle annular dark field or Z-contrast technique has allowed direct atomic resolution images of grain boundaries structures to be obtained.

The majority of the grain boundaries observed in these tapes are (001) pure twist boundaries. These boundaries are seen to be located in the middle of the double BiO layers and are atomically flat. However, it is important to note that although some amorphous layers have been observed at these boundaries, local phase variations near the grain boundaries are often observed, i.e. 1/2 - 2 unit cells of Bi-2212 or Bi2224 phase are formed on either side of the boundaries. This trend of forming the boundary plane on the Bi-O double layers, accompanied by periodical phase variations is also observed at low-angle symmetric c-axis tilt boundaries, where one side of the boundary is parallel to the (001) plane. Such local phase variations may affect the transport of the Bi-O layers as these boundaries may have a significant impact on the local transport properties. Electron energy loss spectroscopy (EELS) analysis of these boundaries will also be discussed in relation to the effect of the observed structure variations on the local carrier concentration.

4:15 PM 1B.8
LOCAL VARIABILITY OF Jc IN HIGH-Jc MULTIFIILAMENTARY Ag/Bi2223 TAPEs.
X.M. Cai, J. Jiang, A. Polyanskii, Y.H. Wu, S.E. Baldo and D.C. Larbalestier, Applied Superconductivity Center, University of Wisconsin, Madison WI.

We have extracted filaments from multifilamentary Ag/Bi2223 tapes with very high Jc at 77K (0.75 km) values of 50 - 60 km.A. Using a laser cutter to dissect the filament bundle, we could establish the position of filaments within the cross section of the tape and, where useful, isolate particular filaments and sections of filaments for local measurements of Jc(H). Defined by Jc/A where A is the cross sectional area measured with an error < 5% Local Jc at 77K (0.75 km) values are very repeatable, values ranging from 20 - 100 km.A, different by a factor of 5. Both self-field and in-field Jc(H) show an inverse linear dependence on the thickness of filament section, suggesting that thin layers near the interface of Ag/Bi2223 are still the major conductive regions even though the filaments are already <0.1 micrometer thick.

1:40 PM 1B.9
DIRECT OBSERVATION OF THE GROWTH OF RIBBON-LIKE THIN FILMS OF Bi2212. Shimami Ariyama, 1, 2, Hung Tran, 2, Akira Inishi, 1, Yoshibiko Takeo, 1, Yoshimasa Satoh, 1, Hikoki Fujii, 1, 2, T. Hatano, 1, Kumman Kogano, 1, Nat. Res. Inst. for Metals, Tsukuba, JAPAN, 4, CREST, Japan Science and Tech. Corp., Tokyo, JAPAN, 5, Univ. of Tsukuba, JAPAN.

We have demonstrated that ribbon-like single crystaline Bi2212 films can be grown on Ag substrates by a very simple process [1]. In this paper, we report on the in-situ observation of the growth of the ribbon-like films by high temperature optical microscopy and the improvement of the substrates. Small pellets of the Bi2212 were put on silver substrates. In a high temperature microscope, the samples were heated up to 900 C and kept for 10 minutes. They were then gradually cooled down to 850-880 C and kept for several hours followed by furnace cooling. The heat treatment was carried out under 1 atm of oxygen. Very thin ribbon-like films of Bi2212 crystals grew on the Ag substrates by this simple method. The ribbon-like films showed the superconducting transition at the onset temperatures of 70K. By the in-situ observation of the growth of the ribbon crystals, it was found that most of the ribbons grew within some 10 minutes when the starting materials were melted. During the slow cooling, a little retreat of the ribbons was seen and this retreat was strongly dependent on the heat treatment schedule. Further, the improvement of the substrate will be shown. In our previous work, the surfaces of the heat-treated substrate were very rough by the recrystallization of the silver. We have succeeded in reducing the roughness by adding Pb in the Ag substrate. [1] Avanaw et al., Physica C 314 (1998) 155

11:00 PM 1B.10
EFFECT OF COOLING RATE ON THE MICROSTRUCTURE AND SUPERCONDUCTING PROPERTIES OF Bi-Pb-Sr-Ca-Cu-O Ag COMPOSITE TAPEs.
H. Fujii, H. Inaguchi, H. Kurosawa, K. Takano, National Research Institute for Metals, Tsukuba, JAPAN; Y. Hishinuma, Advanced Materials Research and Development Center, Meiji University, Hino, JAPAN.

It is known that Ca$_2$PbO$_4$ is one of the main secondary phases acting as an obstacle for supercurrent path in Bi$_x$Pb$_{1-x}$Sr$_2$CaCu$_2$O$_y$ (Bi-2212)/Ag tapes. We initially investigated the stability region of Ca$_2$PbO$_4$ in oxygen partial pressure (PO$_2$, temperature (T)) diagram using bulk (Bi-2212)2 samples. With increasing temperature and decreasing PO$_2$, Ca$_2$PbO$_4$ becomes less stable and Pb in Ca$_2$PbO$_4$ dissolves into the PbO2 phase. Above 1100K, Ca$_2$PbO$_4$ was not observed under PO$_2=0.21$ atm for x=0.2. These results indicate that rapid cooling from high temperature is required to avoid the segregation of Ca$_2$PbO$_4$. Taking these results into account, we investigated the effect of cooling rate on the microstructure and superconducting properties of (Bi-Pb)-2212/Ag tapes of x=0.2 heat treated under PO$_2=0.21$ atm. For the tapes obtained by rapid cooling from 1100K where Ca$_2$PbO$_4$ is not present, the amplitude dependence of the real part of susceptibility was smaller and the Jc values were higher than those of the sample obtained by furnace-cooling. These results suggest that rapid cooling is effective to reduce the segregation of Ca$_2$PbO$_4$ at the grain boundaries and to improve the grain connectivity.

SESSION IIII: POSTER SESSION:
FILM GROWTH AND COATED CONDUCTORS
Chair: David K. Christen
Thursday, December 2, 1999
8:00 P.M.
Exhibition Hall D (H)

III.0.1
THE INFLUENCE OF AN ELEVATED MAGNETIC FIELD ON THE TEXTURE FORMATION OF MULTIPROCESSED Bi2212. E. Cicioletti, P.J. Ferrand and J.B. Vudrane, University of Technology, Department of Materials Science and Engineering, Cambridge, MA.

Mult-processing of BSCCO high-Tc superconductors under an elevated magnetic fields is an effective technique for producing superconductors with enhanced critical current. This is a consequence of the high degree of crystallographic texture achieved in the polycrystalline superconductor high magnetic field. Possible mechanisms for the orientation of Bi-2212 plates-like crystals under the influence of a magnetic field have been analyzed, in particular, the orientation of superconductor or grains during nucleation, crystal growth and grain growth. In order to understand the relevance of each of these mechanisms, we have studied the effect of an applied magnetic field during the different stages of the partial-melt process. Experimental results confirm that most of the alignment is achieved in the early stages of crystal growth. This result may have important consequences for the large-scale implementation of this process.

III.0.2
DIFFUSION AND CRITICAL CURRENT DENSITY OF
Ag-Bi2223 TAPES. H. Wu, P. Skov-Hansen, W. G. Wang, Z. Han and P. Vase, Nordic Superconductor Technologies, DENMARK.

Understanding the mechanical deformation process is very important for achieving high engineering critical current density in long length multilayer Ag-Bi2223 tapes. In this paper, we will investigate the effects of the rolling process on the microstructure and critical current density. The rolling process before the heat treatment will be discussed with an emphasis on the powder deformation behaviour. The effects of intermediate rolling process on the deformation microstructure and the microstructure evolution in the heat treatment will also be discussed.

I10.3 EVOLUTION OF CURRENT-CONTROLLING FACTORS IN Ag-Bi2223 TAPES THROUGHOUT THE HOT PROCESS. V. Beling, A. Goldgrish, E. Yashchuk, M. Roth, I. Felsher, The Hebrew University of Jerusalem, Jerusalem, ISRAEL; A. Polyanskii, D. Larbalestier, University of Wisconsin, Applied Superconductivity Center, Madison WI.

Critical current density, \( J_c \), in self-and external magnetic fields, magnetostriction loops, magnetic susceptibility as well as core phase composition were studied at various stages of tape processing. Intermediate deformation has been shown to facilitate 2212 to 2223 phase transformation as well as growth of core connectivity and its pinning ability. \( J_c \) measurements as well as bending tests and magneto-optical studies reveal very fast healing of deformation-induced damage in the first hour of the subsequent sintering step. A-bis-link area oriented along the dominating deformation-induced stresses are developed during the sintering operation. These areas behave as channels for preferential magnetic field penetration into the sintered tape core. The data on the field penetration into an as-deformed tape give insight on the real picture of stress distribution in the core under intermediate deformation.

I10.4 CORRELATION BETWEEN SIZE/STRAIN PARAMETERS AND CRITICAL CURRENT DENSITIES IN BSCCO/Ag TAPES. Fernando Ribeiraz, Joao Marinkovic, Alexander Polshke, Pontificia Universidade Catolica do Rio de Janeiro, Departmento de Cinco dos Materiais e Metalurgia, BARR, Rio de Janeiro, BARRAZ; Na Smo, Mauricio Liashon, Eduardo Serra, Centro de Pesquisas de Energia Electrica, Electrobras, Rio de Janeiro, BARRAZ.

Size/strain evolution and critical current densities, were studied in BSCCO/Ag tapes filled with two different precursor powders. The precursor powders were obtained by conventional so-called `one-powder’ technique and also, by `two-powder’ processing route. The phase content of precursor powders is already well-established. The size/strain investigations were performed after each step of thermomechanical treatment, using X-ray diffraction line-broadening approach. Convolutions-fitting method was used for obtaining (intrinsic) line-broadening parameters. Size/strain parameters were analyzed by `double-Voigt’ method. Existence of low-angle boundaries inside grains were considered as a potential flux pinning sites. Potential presence of chemical inhomogeneity in 2214/2223 BSCCO and 2223/BSCCO were monitored by x-ray behavior over column length orthogonal to investigated drawing planes.

I10.5 SYNTHESIS AND CHARACTERIZATION OF THALLIUM-BASED 1212 FILM WITH HIGH TRANSPORT CRITICAL CURRENT DENSITY. Jingyu Lao, J.H. Wang, SUNY at Buffalo, Department of Chemistry, Buffalo, NY.; D. Z. Wang, S.X. Yang, Y. Tu, H.L. Wu, Z.F. Ren, Boston College, Department of Physics, Chestnut Hill, MA.; T. Veredelby, M. Parulantrim, T. Aytu, D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.; R.N. Bhattacharya, John H. Blaugher, National Energy Renewable Laboratory, Golden, CO.

A new type of Cr doped Ti-1212 film has been successfully synthesized on LaAlO3 substrate. The Ti containing precursor film was deposited by PLD method, and annealed in static air with Ti containing pellets to become superconductive. The TiO of the film is in the range of 9% - 10%, and the transport \( J_c \) of the film reaches 1.5 x 10^8 A/cm^2 at 77 K and self field. According to our knowledge, this is the highest transport \( J_c \) at 77 K for Ti-1212 film up to now. XRD & 29scan, TI scan and 8 scan shows the dominant, epitaxial growth of 1212 film on the substrate. TEM analysis verifies the high quality of the film.

I10.6 SURPRISING THERMAL STABILITY OF Hg-1212 FILMS. M.P. Siegel, D.L. Oevermyer, E.L. Venturini Sandia National Labs, Albuquerque, NM.; J.J. Wu and Y.Y. Xie, Dept. of Physics & Astronomy, Univ. of KS., Lawrence, KS.

We studied the high-temperature thermal stability of high-quality Hg-1212 thin films by incrementally annealing samples in a pure oxygen ambient from 200 to 800°C. The films for this study were grown via the citron-exchange process where a high-quality Ti-1212 or Ti-2212 film acts as a precursor. Typical as-grown Hg-1212 films have \( T_c > 120 K \) and \( J_c = 1 MA/cm^2 \) at 100 K. By monitoring surface morphology, x-ray diffraction microstructure, and Je (SK) from magnetization measurements, we find that Hg-1212 films are stable to temperatures > 750°C, and in the absence of the necessary Hg-oxide overpressure for their growth! This is even more surprising given the severe degradation of the Ti-superconductor precursor films at 700°C in the pure oxygen ambient to TiO2 oxide. The onset of this degradation in Ti-superconducting films generates defects associated with significant enhancements in flux pinning. Comparisons will be made with Hg-1212 films annealed to similar conditions. 8 Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. DOE under Contract DE-AC04-94AL85000.

I10.7 REVERSAL OF UNDER-DOPED TO OVER-DOPED TTBi22CaO6 THIN FILMS. D.Z. Wang, S.X. Yang, Y. Tu, Z.F. Ren, J.I. Oh, Y. Sun, H. Ha and M.J. Vanghiiet, Boston College, Dept of Physics, Chestnut Hill, MA.; Jingyu Lao, J.H. Wang, SUNY at Buffalo, Dept of Chemistry, Buffalo, NY.

Epitaxial superconducting thin films Ti2Bi22CaO6(1220) with different doping levels have been synthesized. The films were made by rf magnetron sputtering and post-deposition annealing. The doping levels were controlled by varying the oxygen content. At first, the as-grown Ti-2201 thin films were annealed gradually from the initially overdoped state, through the optimally doped state, to the under-doped state in flowing argon or vacuum. Then, the same samples were brought back through the optimally doped state, to the overdoped state in air oxygen. Upon additional depletion of oxygen there was no structural change measured by XRD. Transmission electron microscopy bright-field images and selected area diffraction patterns showed that the tetragonal structure didn’t change either.

I10.8 FABRICATION OF YBa2Cu3O6 THICK FILMS ON NON-TEXTURED METAL SUBSTRATES BY ELECTROPHORETIC DEPOSITION. R.L. Meng, J. Hildebrand, J. Cminaldak, D. Pham and C.W. Chu, Dept of Physics and Texas Center for Superconductivity, Univ of Houston, Houston, TX.

A practical electrophoretic process has been applied to deposit YBa2Cu3O6 thick films on a non-textured Ag alloy substrate. X-ray diffraction patterns show a high degree of \( \alpha \)-axis texturing perpendicular to the surface. There is no observable reaction between the substrate and the YBCO thick films. A sharp transition with an onset temperature of 90 K and a zero-resistance temperature of 87 K indicates the high quality of the thick films. A transport \( J_c \) of up to 7 x 10^5 A/cm^2 has been achieved repeatedly at 77 K and zero field. The microstructure and physical properties of the YBCO thick films strongly depend on the process conditions, which will be discussed.

I10.9 MICROSTRUCTURE DEVELOPMENT AND INTERFACE STUDY IN PERITECTICALLY GROWN YBa2Cu3O6 TAPES ON SILVER ALLOY SUBSTRATE. Donghao Shi, X. Wen and D. Qiu, Department of Materials Science and Engineering, University of Cincinnati, Cincinnati, OH.

A series of cutting experiments have been carried out to study the initial nucleation and growth of YBCO on silver alloy substrate. Although the substrate has a random orientation, the YBCO grains have been found to be highly in-plane textured. The work is motivated to study the nucleation mechanism of YBCO from the liquid state during the peritectic solidification. Our results show that during the peritectic reaction, YBCO grains precipitate along the surface of the silver alloy substrate which acts as the nucleation site. As the liquid wets the substrate, the precipitation of YBCO cannot assume a uniform growth normal to the substrate surface due to liquid surface tension. As a consequence, they epitaxially grow at a high rate upon quenching along the substrate surface, which minimizes the system energy. Our x-ray data show a consistent evolution of YBCO precipitation as the quench temperature is lowered from slightly above peritectic point to 99°C. Corresponding microstructure study results will also be presented.

I10.10 MICROWAVE PROPERTIES OF A NOVEL SUBSTRATELESS RESONATOR USING SINGLE DOMAIN YBa2Cu3O6. Donghu Shi and D. Qiu, Department of Materials Science and Engineering, Allan Ferencii, Dept. of Electrical and Computer Engineering, David Mint, Dept. of Physics, University of Cincinnati, Cincinnati, OH.
Large single domain YBa$_2$Cu$_3$O$_7$ materials have been successfully fabricated with superb RF properties by employing the seeded-melt growth (SMG) method. The SMG YBCO samples have been treated with flowing oxygen in a wide range of temperatures up to 700°C. After oxygenation, the surface resistance has been measured at 12 GHz in a magnetic field parallel to the c-axis. The oxygenation treatment results in sharp superconducting transitions and magnetic field dependence below T_c. Based on this new design, a cryocooler has been constructed using the single domain YBa$_2$Cu$_3$O$_7$ processed by SMG. The cryocooler can be used as a low-temperature cooler in a bulk superconducting plate, which operates in the TM$_{010}$ mode. All cryocooler parts are made of single-domain YBa$_2$Cu$_3$O$_7$ without any dielectric materials. The measured Q$_0$ has reached a high value of 10,000 at 1.84 GHz. Experimental data show great promise in the development of RF components using single domain high temperature superconductors.

**II.1.11 MICROWAVE PROPERTIES OF SCREEN-PRINTED Bi2223 THICK FILMS ON DIELECTRIC CERAMIC Ba$_2$Sr$_2$CaCu$_2$O$_y$ A. Oota and T. Takahara, Tokyo University of Technology, Tottori-cho, Toyohashi, JAPAN, N. Matsui, H. Tezuka and Y. Ishikawa, Maruzen MFG Co., Ltd., Nagakute, Tokyo, JAPAN.**

Screen-printed Bi2223 thick films are prepared directly on both sides of dielectric Ba$_2$Sr$_2$CaCu$_2$O$_y$ (abbreviated as BSMT) with relative dielectric constant ε~24. Microscopic properties of the films such as unloaded quality factor Q$_0$ and surface resistance R$_s$ are measured as a function of temperature in the range between 20 and 130 K on a TM$_{010}$ mode, while checking the incident-power dependence. In an attempt to suppress chemical reactions, the nominal composition of superconducting paste is changed and the influence on microscopic properties (in particular on the incident-power dependence) is discussed through multiphase studies from a microstructual point of view. The present best data for the Q$_0$ value at 1.0 K is 30,000 at 70 K and 90,000 at 20 K, which correspond to the R$_s$ value of 0.8 and 0.3 M, respectively. Debye absorption operating at 1.8 GHz on a TM$_{010}$ mode is under construction using 25 mm-cubes of BSMT with Bi2223 thick films as electrodes and the result will be presented.

**II.1.12 MICROWAVE SURFACE RESISTANCE OF SCREEN-PRINTED Bi2212 THICK FILMS A. Oota, T. Takahara, Tokyo University of Technology, Tottori-cho, Toyohashi, JAPAN.**

Microscopic surface resistance (R$_s$) of screen-printed Bi$_2$Sr$_2$CaCu$_2$O$_y$ (Bi2212) thick films on Ag substrate subjected to the partial-melt solidification process was investigated using the dielectric resonance method at 10.7 GHz in the TM$_{010}$ mode. Film thickness and heat treatment conditions on the partial-melt solidification process influence the R$_s$ value of thick films through changes in microstructure formation. The value of C$_e$ (inferred from microstructure formation) was obtained by heating the 27 mm-thick film up to the maximum temperature at 880°C, subsequent slow cooling at 4 ºC/h from 880 to 860°C, isothermal annealing for 10 hours at 860°C and final cooling to room temperature. To our knowledge, this R$_s$ value is the lowest recorded for Bi2212 thick films subjected to the partial-melt solidification process. From a microstructural point of view, the dominating factor for the R$_s$ value of Bi2212 thick films was investigated. The film with low R$_s$ value shows the large grain size, small amount of impurity phases and also a high degree in grain alignment.

**II.1.13 THE EFFECT OF NANO-PARTICLES ADDITION ON FLUX PINNING IN HIGH TEMPERATURE OXIDE SUPERCONDUCTORS. Gui-Wen Qiao, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, CHINA.**

To increase the magnetic flux pinning ability of high Tc oxide superconductors, several kinds of nano-particles were added into the oxide superconductors and the Tc and Je were measured. The experiments showed that the addition of proper amounts of nano-Ca, nano-MgO, nano-SiC and nano-ZrO$_2$ ultrafine powders can improve the critical current density Je of the HTSCs. SEM and HRTEM observations showed that the nano-MgO and nano-SiC particles were solved into the HTSC matrix and caused local segregation of chemical elements and lattice distortions which may play a role in flux pinning centers and contribute to increasing of Je. But nano-Al$_2$O$_3$ particles can not be solved into the matrix and keep their original sizes and shapes which may not contribute to the flux pinning.

**II.1.14 CONTROL OF STACKING FAULT CONFIGURATIONS IN MULTITEXTURED YBCO - A PATH TOWARDS HIGH CRITICAL CURRENTS. J. Pliii, Instituto de Ciencia de Materiales de Barcelona (CSIC), Barcelona, SPAIN, and Universite de Poitiers, Lab de Metallurgie Physique, Poitiers, FRANCE, J. Sanchenzo, T. Puig, Instituto de Ciencia de Materiales de Barcelona (CSIC), Barcelona, SPAIN, J. Rubier, Universite de Poitiers, Lab de Metallurgie Physique, Poitiers, FRANCE, X. Obradors, Instituto de Ciencia de Materiales de Barcelona (CSIC), Barcelona, SPAIN.**

YBCO (1:2:3) is metastable under a wide area of P(Oxygen)-T space covering typical processing conditions. Extrusive CuO stacking faults (SFs) are nucleated due to a local phase transformation to the 1:2:3 phase. Motivated by the interest in potential of SF-assisted flux pinning centers, we explore different routes to develop SF configurations exhibiting a high perimeter to surface ratio in order to maximize the length of partial dislocation. Two forces are required for the nucleation and growth of such SFs: 1) a thermal driving force F1, which measures the distance from the stability line of 1:2:3 at which the sample is processed, and; 2) a mechanical one, F2, necessary to move the bounding partials on (001). In the present experiments results from an isochoric pressure due to the anisotropy of the composite. Under 200 MPa in Ar at 300°C it is found that F2 is dominant, the total SF area remains almost constant, but owing to the different SF energies on both twin domains, a selective re-organization of pre-existing SFs to a low energy configuration in one twin domain occurs resulting in an increase of the total partial dislocation length. A 100% increase of the critical current is achieved by this procedure. On the other hand, at 400°C and 100 bar oxygen F1 dominate over F2. Accordingly the SF area is increased through the nucleation of new SFs at 2:1:1 interfaces, which owing to the strong non-equilibrium conditions, expand across twin walls and display irregular dendritic-like shapes. In this case critical currents are augmented by 150%. Pinning mechanisms are discussed based on the basis of transmission electron microscopy observations and inductive magnetization measurements with the magnetic field applied parallel to the c-axis.

**II.1.15 NOISE CHARACTERISTICS OF ASYMMETRIC MULTI-JUNCTION HTS RF SQUID MAGNETOMETER AND GRADUOMETER ON BICRYSTAL SUBSTRATES. M. Fardmanesh, K. Bartels, J. Schubert, Institut fuer Schicht- und Ionentechnik [ISI], Forschungszentrum Juelich, Juelich, GERMANY.**

Asymmetric multi-junction YBCO rf SQUID magnetometer and gradiometer have been made of 200 nm thick patterned (standard photolithography) PLD YBCO films on the symmetric bi-crystal SrTiO$_3$ substrates. Low 1/f noise performance has been obtained for the rfSQUIDs (magnetometers and gradiometers) on the bi-crystal substrates by implementing a design principle avoiding large area films on the bicrystal grain boundary of the substrates. Washer rfSQUID magnetometers are made with a design principle same as for a do SQUID on bi-crystal substrates using different silk structure and asymmetric junctions with width ratios of 1/2, 1/3, and 1/4. The 1/f noise behavior of the above devices while is lower compared to that of the conventional designs, show a dependence on the width of the asymmetric junctions, increasing with the increase of the junction width.

A new multi-junction design for gradiometers is also presented implementing the same principle using asymmetric junctions while avoiding any large YBCO film weak links on the substrate grain boundaries.

**II.1.16 STEP-EDGE STRUCTURE DEPENDENCE OF 1/F NOISE IN rfSQUIDs AND THE EFFECT OF HIE PARA-JEETERS. J. Schubert, Y. Zhang, W. Zander, M. Fardmanesh, Institut fuer Schicht- und Ionentechnik [ISI], Forschungszentrum Juelich, Juelich, GERMANY.**

Step-edge junction rfSQUID gradiometers and magnetometers are made using PLD YBCO films on LaAlO$_3$ [100] and SrTiO$_3$ [100] substrates. Effects of the step-edge structure, prepared by different HIE processes, as well as the influence of re-deposited material on the 1/f noise and signal of the SQUIDs have been investigated. For the normal incident ion beam etched steps, a hard layer of re-deposited material is found to form on the side-walls of the steps standing up to e.g. one hundred nm deep steps on LaAlO$_3$. This is found to be much less for the steps on SrTiO$_3$ substrates. The re-deposited layer is found to strongly reduce the yield of the SQUIDs on LaAlO$_3$ substrates and drastically increase the 1/f noise for the working devices. The SQUID signal on SrTiO$_3$ substrates is found to be less sensitive to the re-deposited material at the edge of the steps. SQUIDs made on LaAlO$_3$ with steps etched using different angle incident ion beam, having similar re-deposited structure and lower 1/f noise behavior, contradicting re-deposition of the material at the steps. The detailed effects of the step etching parameters on the noise and signal of the devices are presented in this work.
H10.15 MICROJET AND DC FLUXON DYNAMICS IN BICRYSTAL YBCO GRAIN-BOUNDARY JOSEPHSON JUNCTIONS. H. Xu, 1,2,3 D.E. Oates, 1,2 G. Dresselhaus, 1,2 M.S. Dresselhaus 1,2
1 Massachusetts Inst of Tech, Cambridge, MA; 2 Lincoln Laboratory, Lexington, MA; 3 AFRL, Hanscom AFB, Bedford, MA.

Measurements of the power-dependent and dc-magnetic-field-dependent microwave (rf) impedance $Z(H, H_A, H_B)$ of a YBCO suspended microstrip resonator with an engineered bicrystal grain-boundary junction were made at this conference. By measuring the fundamental and first overtone modes of the resonator, effects of the junction can be separated from those of the background film, and we can infer the microwave impedance $Z(H, H_A, H_B)$ of the junction. The suspended microstrip geometry has no superconducting ground plane so that small calibrated magnetic fields can be applied to the junction. Both rf power dependence and dc magnetic field dependence of $Z$ were studied at frequencies 4 to 8 GHz for temperatures ranging from 5 to 70 K. The second to third harmonics generated by the grain boundary junctions were also measured as functions of rf power and external dc magnetic field. The applied dc magnetic field ranged from 0 to 1000 Oe. The measurements show that the dc magnetic field reduces the rf critical current of the junction. Additionally, the dc magnetic field was scanned in fine steps at fields of the order of 1 to 10 Oe, with constant rf input power (constant $H_A$), changes of the microwave impedance by greater than a factor of two were observed. This behavior is consistent with single dc fluxons entering and interacting with the rf in the junctions. Experimental results will be compared with various model predictions. The results will be related to the origins of the non-linear microwave impedance of epitaxial thin films. This work is supported by Air Force Office of Scientific Research, account number F49620-96-1-0021.


Several different kinds of YBCO grain boundary Josephson junctions, all characterized by a 45° misalignment of the c-axes have been obtained by employing a recently implemented bi-epitaxial technique. Different kinds of boundaries are obtained by using either MgO or CeO$_2$ as seed layers. Junctions on these grain boundaries exhibit good Josephson properties, which could be fruitfully used for applications. High values of the $R_0$ product and a Fraunhofer-like dependence of critical current on the magnetic field, differently from traditional bi-epitaxial junctions, have been obtained by using an MgO seed layer. The temperature dependence of these properties has been investigated by high resolution electron microscopy, which was also performed on previously measured junctions, and by X-ray Diffraction. The presence of atomically clean bond plane face in the junction boundaries, among other types of interfaces, has been shown. The possibility of selecting these kind of boundaries by controlling film growth, and their possible advantage in terms of reproducibility and uniformity of the junction properties, are presented. The possibility of employing these junctions to explore the symmetry of the order parameter is also discussed.

H10.17 FABRICATION AND PROPERTIES OF YBCO COATED CONDUCTOR ON METALLIC TAPE SUBSTRATE. Guowei Yuan, Jian Yang, Dongqi Shi, Superconducting Materials Research Center, General Research Institute for Nonferrous Metals, Beijing, CHINA.

The YBCO coated conductor as a second generation of high-$T_c$ superconducting tape has widely attracted people's attention. We have deposited YBCO superconducting film on Cu-O$_2$ and YSZ buffer layers on rolling Ni substrates by pulsed laser deposition (PLD) system. The metal tape substrates were formed by progressive rolling of high pure nickel at room temperature and recrystallized by high temperature annealing. After well-polished nickel substrates, the YBCO film was deposited on the substrate by PLD or sub-textured Ni surface at temperatures in the range 200°C to 400°C with Ar; $H_2$ = 9 : 1. The deposition temperature of 400–500°C yield epitaxial films, higher temperatures lead to both formation of NiO and decomposition of La$_2$O$_3$ phase. From the X-ray $θ-2θ$, $ω$, $ϕ$ scans, pole figure analyses, all the films show good in-plane and out-of-plane orientations. In addition, smooth, dense, and pinhole-free as-deposited structures were observed for all the films deposited on bi-axially textured nickel substrates. We also describe the development of new buffer layer configurations directed towards the implementation of high temperature superconducting conductors. The works are in progress on the conductive films for this and other potential applications will be discussed.

H10.20 MAGNETIC FIELD SCANNER OF HIGH-T$_c$ SUPERCONDUCTORS. H.J. Kroemer, and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

A mutual inductor is used to scan high-$T_c$ superconductive tapes to show spatially varying critical current densities. A superconductor conductive plane placed below the tape is biased through a current source. A second secondary windings shields the secondary from a small ac magnetic field of current supplied to the primary. No secondary signal is induced until the primary ac current and the induced magnetic field, enhanced by a ferrite core, induces critical current density in the superconductive material. Similarly multi-filmyrate tape reduces the mutual inductive coupling roughly to the volume fraction of the matrix. By varying the primary ac current both the critical current density and the carrier density of the superconductor. Such scans at liquid-nitrogen temperature, $T$ at 77 K, have been carried out for multi-filmyrate Bi2223 tape and for YBCO film grown on a rolled, annealed, bi-axially textured substrate (RA-BETS).


Intermetallics is developing a wide range of HTS conductors for electronic power applications such as superconducting fault current limiters, Transmission cables and Generators. In addition to manufacturing kilometer-lengths of Bi-2223 Co/Co for these applications, Intermetallics has been developing Coated YBCO Superconductor as a high performance, low cost alternative HTS conductor. A Metal Organic Chemical Vapor Deposition (MOCVD) process is being developed at Intermetallics to fabricate Coated YBCO Superconductor using metal substrates. MOCVD is a viable alternative to Physical Vapor Deposition techniques as an industrial process for large-scale YBCO conductor manufacturing. Nickel alloy substrates with a bi-axially-textured buffer layer of silicon were used for YBCO deposition. The bi-axially-textured buffer layers were fabricated at Argonne and Los Alamos National Labs by Ion Beam Assisted Deposition (IBAD). The in-plane texture of the IBAD buffer layers used for YBCO deposition ranged from 15 to 25 degrees FWHM. MOCVD reactor design, precursor delivery scheme and process conditions were optimized to deposit epitaxial YBCO films with high Transition Temperature ($T_\text{c}$) and high Critical Current Densities ($J_c$). YBCO films with in-plane texture less than 4 degrees FWHM, $T_\text{c}$ of 80 K and $J_c$ greater than 1 MA/cm$^2$ at 77 K have been fabricated by MOCVD using the IBAD substrates. Detailed analysis of texture, composition, film coverage, transition temperature and current density of the YBCO films will be discussed in the presentation.

H10.22 EPITAXIAL GROWTH OF CONDUCTIVE BUFFER LAYERS FOR THE DEVELOPED CONDUCTOR WIRE SYSTEM. Arnaud Joly, Judy Z. Wu, Univ. of Kansas, Dept. of Physics and Astronomy; Lawrence, KS; Claudia Contoni, Darren T. Verheghe, David P. Norton, David K. Christen, Oak Ridge National Laboratory, Solid State Div., Oak Ridge, TN; Amit Goyal, Elise D. Specht, Oak Ridge National Laboratory, Metals and Ceramics Div., Oak Ridge, TN; Muniwigga Paranthaman, Oak Ridge National Laboratory, Chemical and Analytical Sciences Div., Oak Ridge, TN.

Epitaxial buffer layers of electrically conductive TiN, Zn and LaNiO$_3$ have been deposited on bi-axially textured nickel tapes by sputter deposition. Epitaxial deposition of TiN and Zn films were achieved reactively by dc-magnetron sputtering techniques at substrate temperatures ranging between 600–800°C, at rates of 3A/sec. For the growth of LaNiO$_3$ films, rf magnetron sputtering techniques were employed. While the deposition temperatures of 400–500°C yield epitaxial films, higher temperatures lead to both formation of NiO and decomposition of La$_2$O$_3$ phase. From the X-ray $θ-2θ$, $ω$, $ϕ$ scans, pole figure analyses, all the films show good in-plane and out-of-plane orientations. In addition, smooth, dense, and pinhole-free as-deposited structures were observed for all the films deposited on bi-axially textured nickel substrates. We also describe the development of new buffer layer configurations directed towards the implementation of high temperature superconducting conductors. The works are in progress on the conductive films for this and other potential applications will be discussed.
H10.20 DEVELOPMENT AND USE OF CUBE TEXTURED NON- MAGNETIC NaAlO₂ TAPES AS SUBSTRATES FOR YBCO FILM GROWTH. Norman Roger, Laura Fernandez, Bernd de Boer, Jörg Eichler, Reinhold Hoffmann, Helmut Schubert, Institute for Solid State and Materials Research, Dresden, GERMANY.

A promising technique for producing long lengths of high current carrying HTS wires is the RABITS approach. High current densities are achieved on Ni-tapes in which a strong cube texture develops after rolling and recrystallisation, but one disadvantage of Ni-substrates is the ferromagnetism by which losses due to magnetisation processes are expected. The approach described here is a non-magnetic substrate. The ferromagnetism of Ni is suppressed by alloying it with 13 staunch 5Cr or 9Cr 5V. In these alloys very strong cube textures with a FWHM of 6° and less than 1% highly misoriented grains (detected by electron backscattering diffraction EBSD) are achieved by rolling and recrystallisation. On these tapes, with a thickness of 0.1mm, different systems of buffer and YBCO films were deposited by pulsed laser deposition. The texture of the substrates and the films was compared with the aid of X-ray texture analysis and EBSD. Their microstructure was investigated by SEM and AFM. In addition the suitability of the buffers to prevent diffusion of Ni and Cr or V into the YBCO was verified by measuring MS-profiles through the buffer layer. Furthermore the YBCO layer was characterized by Tc and jc measurements.

H10.20 TEM ANALYSIS OF SILVER METAL CONTACTS ON YBCO HIGH Tc: SUPERCONDUCTING FILMS. Ron Anderson, Mark Hudson, IBM Analytical Services, Hopewell Jct., NY; Claire Pettiette-Hall, John Burch, TRW Space and Electronics Group, Redondo Beach, CA.

There are very few references to TEM analytical studies of metal contacts to high temperature superconducting (HTSC) materials because most metal contacts are complex oxide multilayers and attempts to prepare cross section TEM specimens fail because of delamination problems. Conventional TEM preparation methods, where the cross section specimen is mechanically thinned to 30 to 50 microns in thickness and then ion milled to electron transparency, are difficult to prepare because HTSC materials, in our case YBCO, may not be mechanically polished in water, and even very short ion milling steps cause severe radiation damage to the HTSC and considerable thickness variations in the final cross section due to differential milling rates of the various layers. The use of a focused ion beam (FIB) tool is problematical because of electronic discharge problems and severe radiation and implantation effects from the 30 to 50 KeV ion beam.

We have developed specimen preparation methods for preparing Ag contacts on YBCO films that are free of water etching artifacts and can be performed with either no, or at most one or two minutes, of ion mill thinning. These methods are based on the tripod polishing technique invented by the IBM Analytical Services facility. Mechanical tripod polishing is performed in propylene glycol to a thickness of less than 0.5 micron. Ion milling, when needed, is performed in a very low angle of incidence directed from the substrate to the film stock only for no more than one or two minutes. The entire process was optimized greatly by the design and implementation of a special TEM device macro on the HTSC chip. The method yields large area, near-faultless specimens with no differential thinning of the silver metal, YBCO HTSC, the strontium titanate insulator, or the chip substrate. The specimens are thin enough for atomic resolution TEM analysis over several hundred thousand square microns. Numerous examples will be shown with emphasis on the Ag to YBCO interface structure.

H10.31 REEL-TO-REEL CONTINUOUS DEPOSITION OF OXIDE BUFFER LAYERS ON BIAxially TEXTURED Ni TAPES BY RF SPUTTERING. P.A. List, D.F. Lee, D.M. Kroeger X. Cui, A. Goyal, M. Paranthaman and P.M. Martin, Oak Ridge National Laboratory, Oak Ridge, TN.

A reel-to-reel, RF sputtering system has been developed to continuously deposit epitaxial oxide buffer layers on meter long lengths of biaxially textured Ni substrates. The deposition system consists of two interconnected sputtering chambers for YSZ and CeO₂ buffer layer deposition. The most commonly used buffer layer architecture consists of a CeO₂ layer deposited by electron-beam evaporation or sputtering, followed by sputtering of YSZ and CeO₂ layers. X-ray diffraction measurements using Cu Kα irradiation indicate that the deposition of YSZ deposited by RF sputtering under moving tape condition is associated with loss of oxygen in the initial e-beam or sputtering deposited CeO₂ film. By supplying sufficient water vapor during the deposition process, a layer of delamination-free YSZ and CeO₂ films can grow. Two previously e-beam or previously sputtered CeO₂-buffered Ni tapes used under moving tape condition. The microstructures of these buffer layers will be presented. We will also report the results of YBCO films deposited on short segments of these buffer substrates. This work was conducted in part under a CRADA with 3M/Southwire/LANL funded by the U.S. Department of Energy, the Office of Energy Efficiency and Renewable Energy, and the Office of Energy Research. Oak Ridge National Laboratory is managed by Lockheed Martin Energy Research Corporation for the U.S. Department of Energy under contract # DE-AC05-96OR22644.

H10.32 REEL-TO-REEL DEPOSITION OF STOICHIOMETRIC Y-Ba₂Cu₃O₇ PRECURSOR FILMS ON EPITAXIALLY OXIDE BUFFERED Ni TAPES BY ELECTRON BEAM EVAPORATION. X. Cui, P.A. List, D.M. Kroeger, E.D. Specht, L.P. Thomas, Oak Ridge National Laboratory, Oak Ridge, TN.

One of the steps to grow a high current density YBCO film on epitaxially textured, oxide buffered, Ni tape (RABITS) is to deposit high quality Y-Ba₂Cu₃O₇ precursor film. This film should be stoichiometric in cation composition (i.e., Y₂BaCuO₅) for the entire precursor deposition process. Rutherford backscattering spectroscopy studies indicate that partial pressure of water vapor during deposition has a significant effect on cation deposition rates and the oxygen content in the precursor film. Oxygen content of the precursor is increased and its stability upon exposure to air is improved by the introduction of water vapor during precursor deposition. High quality precursor films with a thickness of 300 nm were deposited on lengths of RABITS in a reel-to-reel, electron beam evaporation system. Properties of post-annealed YBCO films will also be presented. This work was conducted in part under a CRADA with 3M/Southwire/LANL funded by the U.S. Department of Energy, the Office of Energy Efficiency and Renewable Energy, and the Office of Energy Research. Oak Ridge National Laboratory is managed by Lockheed Martin Energy Research Corporation for the U.S. Department of Energy under contract # DE-AC05-96OR22644.


One of the challenges in making commercially useful coated conductors is the fabrication of a high performance superconductor layer that serves as the structural template for subsequent superconductor growth. This biaxial texture is necessary to maximize both the critical current and magnetic field performance of the superconductor. La₁−ₓSrₓAlO₃ (LSAO) single crystal layers have been shown to have several advantages for the growth of Tl-based superconducting films especially with regard to proper phase formation. LSAO single crystals have also supported high-quality films of YBa₂Cu₃O₇ (YBCO). We will describe the development of an epitaxial tri-layer buffer scheme consisting of LAO/LNO/Co₃O₄ deposited in-situ on biaxially textured Ni (100) substrates using pulsed laser deposition (PLD). The full-width-at-half-maximum of the (110) LaO phi scan peak is ~1.5°. The use of LaNiO₃ (LNO) as the middle layer overcomes problems with previous tri-layer buffer configurations. Specifically, LNO has both a more consistent growth orientation and a lower deposition temperature on Co₃O₄. However, the proper phase formation of LNO requires the deposition of O₃ during growth. This can cause problems in the surface morphology due to the reaction of Ni with O₃, if the PLD parameters are not properly controlled. Issues concerning both the crystal orientation and surface morphology will be discussed. We will also present the results of superconducting films grown on the LAO/LNO/Co₃O₄/Ni substrates.


Substrate finishing plays a big role in determining the superconducting properties of YBCO films and both for electronic devices and second generation coated conductors. We have
investigated the effect of extreme substrate surface roughness on the structural and superconducting properties of YBCO films. Results are compared to those obtained on high-purity strontium titanate substrates. The YBCO films on unpolished substrates showed reduced critical transition temperature and current density. The crystallographic misalignment, both out-of-plane and in-plane, for the films on unpolished substrates increases compared to the films on polished substrates. Scanning and transmission electron microscopes show poor connectivity in the films on extremely rough substrates.

H10.35
EVALUATION OF NICKEL BASE ALLOYS AS METALLIC SUBSTRATES FOR GENERATION II TEXTURED YBCO COATED Conductors. Rama M. Nekkanti, Lyle B. Brunke, Venkat Seetharaman, USE, Inc., Dayton, OH, John A. McDaniels, Imam Maatene, Gerry Landis, Dave Dempsey, University of Dayton Research Institute, Dayton, OH, Gregory K. Kozlowski, Wright State University, Dayton, OH, Dave Tomich, Rand Biggers, Timothy Peterson, Materials and Manufacturing Directorate, Paul N. Barnes, and Charles E. Oberly, Propulsion Directorate, Air Force Research Laboratories, Wright-Patterson Air Force Base, OH.

High current densities (~10^5 A/cm^2) were recently demonstrated in short specimens by epitaxial growth of YBCO on bimetallic textured polycrystalline nickel substrates. The process has the potential for long-length processing of the conductors and is being pursued for practical applications. Unfortunately, the low strength of the recrystallized nickel requires delicate handling and its low electrical resistivity and high magnetic permeability (leading to eddy current losses) pose a problem for AC applications. There is a definite need for substrate materials characterized by high strength, good oxidation resistance, high electrical resistivity and low magnetic permeability. The proposed work is focused on binary nickel base alloys as alternate metallic substrates. High purity Ni-Cr binary alloys were melted and hot-rolled/welded to obtain thin sheets/stripes. Results to date on the development of texture by thermo-mechanical processing of these alloys will be presented and compared with those from nickel.

H10.36
FABRICATION OF 3mm LONG Y-123 COATED CONDUCTORS BY IBAD METHOD. Yoshio Higata, Miki Kimura, Takashi Suhit, Fujiura Ltd., Material Technology Lab., Tokyo, JAPAN; Knoru Takeda, Super-GM, Osaka, JAPAN.

YBa2Cu3Ox-x (x=1-2) (Y-123) conductors is a hopeful candidate for practical use of superconducting wires in liquid nitrogen temperature. In order to derive the intrinsic pinning properties of Y-123, biaxially aligned structure is so far the only way, which can avoid its severe weak-link problems. Ion-beam-assisted deposition (IBAD) method offers an ideal tape-shaped substrate for Y-123 conductors which have both biaxially aligned surface structure and sufficient mechanical strength and flexibility. YSZ films of 0.7-1.0 mm thick were continuously deposited by IBAD on Ni-based alloy tapes with lengths of 1.2-5.0 m and tape shifting speeds of 2.5 - 25 cm/h. The YSZ films were uniformly coated with the in-plane mosaic spread of 1-7 8. Y-123 films were deposited by pulsed laser deposition (PLD) on the YSZ tape-like layers with the mosaic spread of 1-7 8. The minimum in-plane mosaic spread for the Y-123 films was 98 degrees. I. of 19.7 A and Jc of 2.3x10^3 A/cm^2 were obtained at 77K, 9T, in a sample with 0.85 mm thick at the length of 1.9 m. In order to avoid slight interdiffusion and lattice mismatch between Y-123 and YSZ thin Y3O5 films were deposited on several YSZ templates by PLD. Ic of 44 A and Jc of 3.8x10^3 A/cm^2 were obtained at the length of 0.9 m, in a sample which had a 1.2 mm thick Y-123 film with a 0.1 mm thick Y3O5 layer. The longitudinal uniformity of Jc values was deeply improved by the intercalation of the Y3O5 layer. The trial for further length are in progress and the scheme of practical scale-up for both YSZ and Y-123 films will be discussed. This work was carried out as a part of R&D on superconducting power apparatus for electric transport systems, the New Sunshine Project of AIST, MITI, being consigned to NEDO.

H10.37
CRITICAL CURRENT DENSITY MEASUREMENTS OF ONE METER LONG, HIGH CURRENT, YBCO COATED CONDUCTOR TAPES. Raymond F. DePaoli, J. Yates Coulter, Michael J. Demekovski, Eric J. Peterson and Fred M. Mueller, Los Alamos National Laboratory Superconductivity Technology Center, Los Alamos, NM.

Meter-long lengths of high current superconducting tape are now being manufactured on a regular basis at Los Alamos National Laboratory. The best one meter end-to-end measurement to date has been 122 Amps. While end-to-end measurements define the ultimate performance of the tapes, critical current measurements on a shorter length may be more useful in order to examine tape uniformity. The single tape exhibits very high (>200 Amps) current-carrying regions that are interspersed with lower critical current regions. Complete characterization of these tapes becomes problematic, in the process of characterizing high critical current sections, low critical current sections often degrade before they can be located and characterized. One approach to this problem is to use a multi-channel multiplexing system to simultaneously measure I-V curves for all of the sections of the tape. Properly performing such scans can then be subtracted or removed and the high critical current sections can then be characterized. This poster will present the results and details of this and other techniques used to address this problem.

H10.38

Yttria-stabilized Zirconia and Ceria have been widely used as epitaxial buffer layers between roll-textured Ni tapes and the YBCO layer in superconducting tapes. In this talk, we describe results on an alternate buffer layer, TiN. TiN has the advantages of high electrical conductivity, potentially allowing shifting of current into the Ni tape, better mechanical toughness than oxides, and relative ease of high-rate deposition by reactive sputtering. While nitride growth works well for NiO formation at the substrate, there is a potential problem with oxidation of the nitride during YBCO growth. Epitaxial TiN films with thickness ~300nm were grown on Ni RABITS by reactive magnetron sputtering at ~500-650°C. X-ray diffraction (XRD) phi-scan of the TiN (111) reflection showed cubic-on-cube epitaxy, despite large lattice mismatch (18.5%). Typical full width half maximum (FWHM) values were 7°, less than that of the textured Ni substrate, ~8°. Films were also grown on MgO substrates, which has much smaller lattice mismatch. Initial studies of YBCO growth and superconducting properties. Pulsed laser deposition was used to grow a thin (~<100 nm thick) layer of ceria on the TiN-coated MgO followed by an ~300nm thick YBCO layer. The XRD phi-scan showed a YBCO [200] reflection with a FWHM of ~3° and XRD rocking curve showed FWHM of ~1° on (001) reflection, indicating excellent in-plane and out-of-plane texture. A superconducting critical transition temperature (Tc) of 89 K was measured by AC susceptibility. A critical current density (Jc) of 6x10^5 A/cm^2 was reproducibly obtained at 77 K by whole body transport current measurement in self field using a 1 mV/cm criterion.

H10.39
THE ROLE OF GRAIN BOUNDARY PLANE AND FACETING IN THE CURRENT CARRYING CAPABILITY OF HIGH-ANGLE GRAIN BOUNDARIES IN YBCO. M. Mirnov, S. Stolbov, and K. Ahrends, Univ of Houston, Texas Center for Superconductivity, Houston, TX.

We have studied 3 grain boundaries in YBCO with measured Jc using TEM, followed by simulation of their microstructure and estimation of superconducting order parameter. For these grain boundaries, misorientation characteristics and grain boundary planes as well as facetting planes have been determined. Our results suggest that for high angle boundaries, this conventionally should be considered as weakly coupled, the high critical current densities can be stipulated by certain grain boundary planes. In particular, if in at least one growing grain boundary plane is (001), the grain boundary has high chances to be strongly coupled. The (001) plane that is favorable energetically as well can be maintained by (001) or (110) facets of different length. However, in the bulk material, grain boundary with the same misorientation characteristics changes planes many times. When inevitably changing to other planes, facetting still can present to reduce the energy of the new orientation. We estimated the contribution of grain boundary planes and facets to the current carrying capability of these grain boundaries and discuss a role of these factors for the current carrying capability of high-angle grain boundaries.

H10.40
SUPERCONDUCTING PROPERTIES OF [001] TILT YBCO THICK FILM GRAIN BOUNDARIES ON SrTiO3 AND TEXTURED Ni-SUBSTRATE USING THE BaF2 EX-SITU POST REACTION PROCESS. Qing Li, V.P. Solovyov, H.J. Wiesemann, Y. Zhu and M. Stuenag, Department of Applied Science, Brockhaven National Laboratory, Upton, NY.

We present a study of superconducting properties of [001] tilt YBCO thick film boundaries on SrTiO3 bicrystal substrates and textured Ni substrates. The YBCO films with thickness ranging from 0.5 to 5 micrometer were prepared using BaF2 ex-situ post reaction process. The substrate temperature during the precursor by high rate oxygenation process at the misorientation angles of the grain boundaries ranged from 80 to 240°. 2.5 micrometer wide strips containing a single [001] tilt grain
boundary can be isolated by either photolithographic or laser patterning. An electric configuration, with up to 5 voltage points, located along the grains, was used to measure resistance and the voltage-current (V-I) characteristics of the grain boundary at various section. The microstructure of the grain boundaries was examined using advanced TEM. We shall discuss the relation between the local microstructure and transport properties of these grain boundaries at micrometer length scale.

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I11.41
SUPERCONDUCTING YBCO THIN FILM ON MULTI CRYSTAL SILICON. AS FABRICATED OR AGED ON MgO SUBSTRATE. Jacob Azoulay, Artem Verdyaev, Igoi Laponog, Center for Technological Education Holon affiliated with Tel-Aviv University, Dept of Science, Holon, ISRAEL.

Superconducting YBa2Cu3O7 films were grown by reactive evaporation on multocrystalline silver film which was evaporated on MgO substrate. A simple inexpensive vacuum system equipped with resistively heated boat was used for the whole process. Silver film was first evaporated on MgO substrate kept at 400°C during the evaporation after which with no further annealing a precursor mixture of yttrium small grains and Cu and BaF2 in powder form weighed in the atomic proportion to yield stoichiometric YBa2Cu3O7 was evaporated.

The films thus obtained were annealed at 740°C under low oxygen partial pressure of about 1 Pa for 30 minutes to form the superconducting phase. X-ray diffraction and scanning electron microscopy techniques were used for texture and surface analysis. Electrical properties were determined using a standard dc four-probe method for electrical measurements.

The physical and electrical properties of the YBCO films are discussed in light of the fact that X-ray diffraction measurements done on the silver film have revealed a multicrystalline structure.

I11.42
FABRICATION OF DIELECTRIC/NBCO MULTI-LAYER STRUCTURES. Michitomo Yama, Osami Horibe, Yasuo Oshikubo, Yoichi Enomoto, ISTEC, Superconductivity Research Laboratory, Chiba, JAPAN.

NdBaCuO (NBCO) has outstanding characteristics among the 90K class superconductors. Particularly NBCO films have smooth and particle-free surfaces, suitable for multi-layer structures. Therefore ramp-edge type Josephson junctions utilizing NBCO films have been extensively studied. Superconducting circuits require large area NBCO films with uniform characteristics to accomplish their superconducting operation. NBCO films have been deposited by either sputtering or pulsed laser deposition methods. We already reported that the deviation of the critical temperature was suppressed less than 5% over 4 cm diameter area by the 90K-off-axis RF sputtering method.

The 3 mm-diameter Si/SiO2/SiC/PHN heterostructure with flat heating bend was revealed to be effective to minimize the random distribution of the specimen temperature mentioned above. Multi-layer structures with NBCO layers, either device structure or multi-layer interconnection, can also be deposited sequentially by this sputtering apparatus attacking multiple targets. In this work, we report on the adjustment of fabrication conditions for dielectric/NBCO multilayer structures. We utilized either L-Si or G-O as dielectric material. We would also like to describe about the in situ process to suppress the deviation of the critical current of NBCO layers, effective to enlarge the operation margin of the superconducting circuits. This work was supported by New Energy and Industrial Technology Development Organization.

I11.43
COMPETITION BETWEEN A-AXIS AND C-AXIS GROWTH IN SUPERCONDUCTING REBCO THIN FILMS: A NEW MODEL. F. Miletto, M. Soci, G. Botti, A. Guglielmetti, Dipartimento di Fisica, Scienze Fisiche, Università di Napoli Federico II, Napoli, ITALY.

The competition between a-axis and c-axis growth in REBa2Cu3O7_δ (RE = rare earth or Y) superconducting thin films has been analysed. Stoichiometric and Nd-rich NdBaCuO thin films have been deposited by DC sputtering. The relative a/c-axis content has been estimated by X-ray diffraction and plotted as a function of deposition temperature. Comparison of observed results with the experimental data available for other compounds yields clear evidence that a universal behavior is followed for REBa2Cu3O7_δ oxides, a behavior which can be described as a function of a normalised deposition temperature. A model for the nucleation of supercritical clusters from a multicomponent adsorbed phase, allowing to face the problem of phase competition in complex compounds, is presented. As a particular case, the competition between different phases of a single compound is analysed as a function of supersaturation. A general behaviour for the orientation dependence as a function of a normalised deposition temperature is thus obtained. Our experimental results on the relative a/c axis content fall on the theoretical curve. The same procedure is also successfully applied to all the data derived from the literature regarding the relative a/c axis content of other REBa2Cu3O7_δ oxides.

I11.44
STRAIN, OXIDATION AND DOPING EFFECTS ON SUPERCONDUCTIVITY OF La2−xSrxCuO4 Thin Films. Weidong Si and Xiaojing Xi, Department of Physics, The Pennsylvania State University, University Park, PA.

We have measured normal-state and superconducting properties of La2−xSrxCuO4 thin films as a function of Sr content x, lattice strain and oxygen content. Strain was controlled by depositing SrAl2O4 buffer layer of different thickness on SrTiO3, SrLaCuO4 and SrLaAlO4 substrates. An oxygen/molecular oxygen mixture was used during cooling to achieve better oxygen uptake. We found that both full oxygenation and compressive in-plane strain are critical for the properties of the La2−xSrxCuO4 thin films, and perhaps the compressive strain makes the oxygenation much easier. Also, we found samples with x=0.5 is superconducting while the bulk counterpart is insulating.

I11.45

Applications using High Temperature Superconducting (HTS) thin films are expected to reach the market very early in this century. Success applications will include low-loss narrow microwave filters for cellular phone repeater stations, microwave delay lines, and SQUIDs. Furthermore, YBCO films deposited onto metal tapes have applications in high-field superconducting magnets and motors. To date, the devices requiring the highest quality YBCO materials are still fabricated using Pulsed Laser Deposition (PLD). PLD has been proposed for production-oriented HTS applications as it was felt early on that this industrial scale-up would not be possible for this process. Deposition equipment exists today that can handle up to 5 inch diameter substrates (or three 3 inch substrates, simultaneously). Furthermore, production PLD systems that can handle sixteen 3-inch, or twenty-four 3 inch diameter substrates are currently under development. Prototype reel-to-reel PLD equipment is also under development for metal tape applications as it is felt that the PLD plume shape and other unique features are ideal for this purpose. However, several other deposition techniques also work well, and co-evaporation is actively being pursued. PLD will readily compete with co-evaporation in terms of film quality, uniformity, and deposition rate. In order to make PLD a more mainstream production tool, improved rate monitors will be required for this process. We have developed a rate monitor for PLD based on Atomic Absorption (AA). A prototype AA rate monitor for copper-vapor sensing has been built and incorporated into a 5 inch PLD system. The design of the AA monitor and its performance characteristics will be presented along with associated YBCO film qualities.

I11.46
SUPERCONDUCTING PROPERTIES OF YBa2Cu3O7 Thin Films on La2−xSrxCuO4 THIN FILMS ON Lso−xcsO3 FEHRROMAGNETIC TEFLONATE LAYERS. S. Freiheer, J. Alem, F.H. Kes, Kamerlingh Onnes Laboratory, University Leiden, THE NETHERLANDS; T. Nojima, Center for Low Temperature Science, Tohoku University, JAPAN; H.W. Zindeliserg, Material Science Department, Delft University of Technology, THE NETHERLANDS.

In view of the interest in spin injection from a ferromagnet into a superconductor, we have investigated the superconducting properties of bilayers of YBa2Cu3O7 / La2−xSrxCuO4 (YBCO/LCMO) grown by laser deposition on SrTiO3 (STO) substrates. The LCMO bottom film grows very smooth, leading to a high quality interface with the YBCO on top. For YBCO films in a thickness range of 12 nm to 48 nm, the superconducting transition temperature lies in the range 79 K–91 K, with no discernible influence of the ferromagnet. However, critical currents in fields up to 8 T proved much lower for the YBCO / LCMO bilayers than for single layers of YBCO on STO, accompanied with lower critical annealing temperatures. A possible explanation is that the good lattice match of the pseudo-cubic lattice parameter of LCMO (0.386 nm) with the a, b-axes of YBCO (0.382 nm and 0.389 nm) leads to less twinning of the YBCO layer than when grown on STO (lattice parameter 0.391 nm).
CONTROL OF THE IN-PLANE EPITAXY OF YBCO FILMS DEPOSITED ON [100] MgO FOR MICROWAVE APPLICATIONS. Kyoko Kawayashi, Kazunori Komori, Misono Fukutomi, Kazunari Togano, National Research Inst for Metals, 1st Research Group, Ibaraki, JAPAN.

Recently, technological progress in the deposition and characterization of high-\textit{T}_c superconducting (HTS) films has enabled the design and fabrication of various high-\textit{T}_c, microwave devices. For the HTS film deposition, microwave applications are widely used as substrates because of its low dielectric constant, low cost, and availability in large sizes. However, YBa\textsubscript{2}Cu\textsubscript{3}O\textsubscript{7-\textdelta} (YBCO) deposition on MgO often causes mixtures of 0\textdegree and 45\textdegree in-plane rotations, resulting in degradation of electrical properties, such as critical current density (\textit{J}_c) and microwave surface resistance (\textit{R}_s). In order to overcome this problem, we used yttrium-stabilized zirconia (YSZ) and CeO\textsubscript{2} buffer layers between MgO and YBCO to control the in-plane epitaxy of YBCO films. Buffer layers were deposited on MgO [100] using off-axis magnetron sputtering and pulsed laser deposition (XRD \{\theta, \phi \text{ and } \phi_s\} scans) reveals an excellent in-plane epitaxy with no evidence of misoriented grains in a YBCO/CeO\textsubscript{2}/YSZ/MgO structure. The dielectric rod resonator method was used for \textit{R}_s measurements of HTS films obtained. Various other methods such as \textit{dc} susceptibility, scanning electron microscopy, atomic force microscopy and inductively coupled plasma spectrometry have been used to analyze the film properties. An attempt was made to correlate these film properties with the measured \textit{R}_s values. The possibility of new buffer layer structures will also be discussed.

ELECTROSTATIC MODULATION OF SUPERCONDUCTIVITY IN EPITAXIAL Pb/\textsubscript{123}Zr/\textsubscript{123}Ti/\textsubscript{123}O\textsubscript{7-\textdelta} HETEROSTRUCTURES. C.H. Ahn, S. Girgilo, P. Paruch, T. Tybell, L. Antognazza, J.-M. Triscone, DPMC University of Geneva, Geneva, SWITZERLAND.

Using the polarization field of the ferroelectric oxide Pb/\textsubscript{123}Zr/\textsubscript{123}Ti/\textsubscript{123}O\textsubscript{7-\textdelta} in epitaxial Pb/\textsubscript{123}Zr/\textsubscript{123}Ti/\textsubscript{123}O\textsubscript{7-\textdelta} heterostructures, we have electrostatically modulated superconductivity in the high temperature superconductor \textsubscript{123}Ba\textsubscript{2}Cu\textsubscript{3}O\textsubscript{7-\textdelta}, shifting the doping level without introducing or structural disorder. For slightly underdoped samples, a change in the normal state resistivity of 50 percent and a uniform shift of the superconducting transition of 7 K were observed. In more underdoped samples, an insulating state was induced. This approach allows one to switch between superconducting and insulating behavior in a reversible and nonvolatile fashion. C.H. Ahn, S. Girgilo, P. Paruch, T. Tybell, L. Antognazza, J.-M. Triscone, Science 284, 1152 (1999).

THE EFFECT OF SUBSTRATE ON THE GROWTH UP SUPERCONDUCTING THIN FILMS. Claudio Lue Carvalho, Universidade Estadual Paulista, Departamento de Fisica, Ilha Solteira, SP, BRAZIL; Josef Anna Varela, Universidade Estadual Paulista, Dept of Chemistry, Aracajuara, SP, BRAZIL; Paulo N.L. Filho, Universidade Federal de Sao Carlos, SP, BRAZIL.

Differents techniques have been used to obtain thin superconducting films as molecular beam epitaxy, magnetron sputtering, etc, but all of them are expensive and the substrates play an important role in the growth of the films. In this work, we propose to obtain high quality films as cheap as possible with favorable orientation of the Bi\textsubscript{2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{3}O\textsubscript{7+\textdelta} system. A new method has been used to deposit thin film on polycrystalline substrates like silver (Ag). A polycrystalline precursor solution obtained by Pechini method has been used to obtain thin film after rheology characterization. Multiple layer were applied on substrate before to heat treated in a cylindrical furnace at 810°C during 1 hour in ambient atmosphere. The best quality films were obtained high quality films deposited on Ag substrate and the presence of the 2212 and 2223 phases that are in agreement with electric and magnetic characterization. Both technique showed transitions temperature around 85K and 110K that correspond to 2212 and 2223 phases, respectively.


An optimized magnet-flux \textsubscript{MOCVD} reactor for the deposition of epitaxial YBCO thin films will be described. The reactor design is the result of extensive numerical simulation, and is able to deposit highly uniform films on 5 cm MgO substrates. The reactor incorporates advanced on-line sensors and diagnostics, including in-situ polarized FTIR ellipsometry for real-time temperature sensing, and ultrafast alternating sensor flow control for precursor design and operation will be described, along with characterization of the deposited films.

SESSION III: Bi, Tl, AND Hg CONTAINING SUPERCONDUCTORS: PROCESSING AND PROPERTIES.

Chair: Lawrence P. Cook
Friday Morning, December 3, 1999
Room 208 (H)

EVOLUTION OF PHASES AND MICROSTRUCTURES OF BPSO-FILM FABRICATED DURING HEATING AND COOLING OF 2223-BPSO TAPES. Klaus Fischer, Tonnetz Fahr, Institut fuer Festkoepfer und Werkstoffkunde, Dresden, GERMANY; Peter Mijewski, Max-Planck-Institut fuer Metallforschung, Stuttgart, GERMANY.

In the past the high-temperature reactions of the precursor in BPSO/Ag tapes in air were extensively studied. We have investigated the precursor conversion in an atmosphere of a reduced oxygen content (N\textsubscript{2}/O\textsubscript{2}). The investigations were performed in situ using high temperature XRD and SEM/EDX measurements on tapes after annealing at different temperatures and subsequent rapid cooling. During heating up the Bi-rich phases such as Pb\textsubscript{12}Bi\textsubscript{4}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{4}O\textsubscript{8} could not be detected above 700\textdegree Cr. This temperature is slightly lower than that published for the precursor conversion inside of BPSO/Ag tapes heated up in air but is substantially lower than expected from phase diagram studies in the Bi-Pb-Sr-Ca-Cu-O system. It was found that the evolution of the degree of texture of the 2212 phase observed during the heating up process depends on the rolling parameters used for the preparation of the samples by flat rolling of round wires to tapes. The maximum texture degree (Logtening factor \textit{F}) of the 2212 as well as of the 2223 phase observed during the conversion of 2212 in to 2223 depends on the reaction temperature. Since the temperature dependence of \textit{F} is nearly the same for both phases at temperatures above 800\textdegree C, it is concluded that the texture of the growing 2233 phase is determined by that of the decomposing 2212 phase. Our assumption, that this texture correlation is caused by the heterogeneous nucleation of the 2223 phase at 2212 grain boundaries, could be supported by a kinetic analysis of the 2233 phase formation. Finally, we studied the phase composition and its influence on the critical current density of tapes cooled down with different cooling rates after the reaction annealing.

LOCAL OXYGEN AND POINT DEFECTS PROBING IN Hg-1211 HTS SUPERCONDUCTORS. J.G. Carrera, CERN, Div. EP, Geneva, SWITZERLAND; J.P. Aragno, Porto University, Dept of Physics, Porto, PORTUGAL; S.J. Loureiro, Princeton University, Dept of Chemistry, Princeton, NJ; P. Touloucan, L. Flech, P. Bocquet, J.J. Cappo, CNRS, Laboratoire de Physique de Grenoble, FRANCE; W. Troger, B. Stortkein, T. Butz, Leipzig University, Dept of Physics, Leipzig, GERMANY; H. Hase, Hahn-Meitner-Institut, Dept of Physics, Berlin, GERMANY; R. Gutt, Wisconsin University, Dept of Physics, Madison, WI; J.G. Mieres, Nuclear and Technological Institute, Dept of Physics, Salamanca, PORTUGAL; J.C. Soares, Lisbon University, Nuclear Physics Centre, Lisbon, PORTUGAL.

The family of mercury-based copper oxides, Hg\textsubscript{1211}Cu\textsubscript{3-x}O\textsubscript{y} with a tetragonal lattice and at 30 GPa a high critical temperature of 1.6 K can be achieved in the Hg-1212 (n=3) compound. These facts create the expectation that precise measurements of the structural properties would allow a better understanding of the charge transfer mechanisms that generate carriers in the superconducting CuO\textsubscript{2} planes. However, the Hg planes are particularly disordered and it is not understood how Hg interacts with point defects like the non-stoichiometric oxygen that regulates \textit{T}_c, \textit{O}_1, and with impurities such as Cu\textsubscript{2-\textdelta}. Many studies using crystallographic techniques as well more local studies such as EXAFS suggested the existence of local distortions in these compounds. So far, it is not known if such effects are linked to the superconductivity mechanism or are simply due to crystal-chemical effects. In this work the Perturbed Angular Correlation (PAC) local technique was applied to measure the electric field gradients (\textit{EFG}) at Hg sites in the Hg-1201 (n=1) high-\textit{T}_c superconductor. High quality pellets were implanted at ISOLDE/CEHIN with very low doses of the \textsuperscript{199}Hg radioactive isotope. The PAC experiments were performed in a 4.2 K, under \textit{Ar} and \textit{O}_2 flow, to change the \textit{O}_2 doping.
concentration. The results show that the EFG parameters are highly sensitive to the O content, that is clearly distinguished from other probe elements in complex oxides. The [FLAPW] of the EFGs were performed in undoped and doped Hg1201 that show an excellent agreement with the experimental results. The local behaviour of Hg atoms in highly oxygen doped Hg1201 was further investigated by the high-resolution X-ray photoelectron spectroscopy. Below 110 K the asymmetry parameter of the EFG becomes non-zero showing that the charge distribution near the O(2)-Hg(2)-O(2) apical chain becomes non-axially symmetric at low temperature in these samples.

9:15 AM H1.3 CONTROLLING TEXTURE IN Ti-1223/Ag COMPOSITES

The irreversibility lines of YBCO and Ti-1223 show that these phases are able to sustain significant critical currents in large fields near 77 K. However, control of grain boundary orientation by texturing biaxially textured films is essential to produce a large area, strongly linked percolative path and therefore high transport critical currents. Research is currently focused on the fabrication of long lengths of textured substrate materials using methods such as IBAD, RABITS and inclined substrate deposition which require the deposition and growth of textured buffer layers. Ti-1223/Ag tape may provide a simpler alternative system which does not require the additional complexity of buffer layer fabrication. The emphasis of our work has been to investigate how the macrotexture of the Ti-1223 phase, determined using X-ray techniques, can be controlled on silver tape and silver film substrates. We have studied the effects of thermomechanical processing conditions and alloying elements on texture development in silver tape, and will discuss their influence on both the recrystallisation texture obtained and the reduction of minor texture components. Ti-1223 thick films have been fabricated on these substrates using spray pyrolysis to deposit a precursor film which is subsequently sintered using a crucible method. We have studied inter and intra-column grain misorientation using EBSD and will report on the relative fractions of epitaxial and misoriented colonies obtained on different textured silver substrates and discuss whether these observations are consistent with CSL theory.

9:30 AM H1.4 SYNTHESIS OF Hg-Ba_{2}Cu_{3}O_{6.44} UNDER CONTROLLED OXYGEN AND MERCURY PARTIAL Pressures
Emmanuel G. Vincz-Dyck, Wesleyan Univ, Dept of Physics, Middletown, CT, John L. Wagner, Univ of North Dakota, Dept of Physics, Grand Forks, ND.

We have investigated the structure, defect concentration, and superconducting properties of Hg-Ba_{2}Cu_{3}O_{6.44} (0.7c<1.1) by resistivity, X-ray and neutron diffraction measurements. With the possibility of more than one defect in Hg-Ba_{2}Cu_{3}O_{6.44}, the role of individual defects on the stability and superconducting properties is obscured by the variations and correlations of multiple defects. To isolate the effect of a single defect, other defects must be held constant. Numerous studies have succeeded in correlating T_c with the variable oxygen defect, δ, through low temperature measurements. However, little is known concerning the interrelation of different defects, specifically ones associated with disorder on the Hg-site. In this work, we demonstrate variations in defects associated with Hg stoichiometry. As the disorder of the Hg-site increases, T_c is found to decrease. Attempts to minimize or eliminate this metastable defect resulted in a decrease in stability in this system. These results indicate that defect associated with Hg stoichiometry is required to form and stabilize the compound with the effect of suppression in T_c.

9:45 AM H1.5 POROSITY AND CONNECTIVITY IN HIGH-Jc AgClAD
(Bi,Pb)Zr_{0.2}Ca_{0.8}Ox TAPES

The porosity and its effects on the connectivity of several series of critical current density Jc optimized Ag-clad multifilament (Bi,Pb)Zr_{0.2}Ca_{0.8}Ox (Bi-2223) tapes through important steps in their thermomechanical treatment was investigated by mass density measurement, microstructural observation, and extensive superconducting property characterization. The filament mass density, measured by digital image analysis of transverse cross sections of the silver and filament materials, could retain relative values normalized to fully 2223 phase density of 85-90% before heat treatment (HT), decreasing to ~70% after the first HT, rising again to about 90% after intermediate annealing, then decreasing to values of 80% after final HT. Consistent with these large porosity and crack fraction, the limiting low temperature moment of samples measured by SQUID magnetometry was almost constant, irrespective of process steps, indicating a high level of sample coherence. After the HT step, extensive cracks were observed by magnetooptical imaging, and these cracks did not heal properly through subsequent HT, showing that the densification during the HT step is obtained at the expense of cracking. Our study, made on a variety of high Jc tapes [Jc(0,77K) values from ~20-60 kA/cm^2], suggest that the performance of 2223 tapes could be significantly improved by raising the mass density if this can be done without simultaneously introducing detrimental cracks.

10:00 AM H1.6 POLYCRYSTALLINE HTC MATERIALS WITH HIGH CURRENT CARRYING CAPACITY
L.J. Gruenker, D. Schneider, S. Koehler, ETH Zurich, Department Materials, Nonmetallic Materials, Zurich, SWITZERLAND.

Bi-2212 and Bi-2223 are the only HTC materials that show sufficient power carrying capacities for industrial applications. In Bi-2212/Ag conductors the growth of the HTS phase can be controlled more easily than in Bi-2223/Ag conductors since the phase development of Bi-2212 is well understood [1]. Bi-2223/Ag tape conductors and bulk materials today are used in current limiters and current leads. Outstanding issues are the limitations of critical currents in polycrystals and the effects of surface barriers in single crystals. Oriented Bi-2212 thick films exhibit much higher critical currents than expected if transport is limited by j_c, of the a-b plane and if it would be obstructed by large grain boundaries. Therefore macroscopic transport is thought to occur differently in Bi-2212 than in the Bi-2223 and Y-123 [2]. This has led to several models for the current transport in Bi-2212 polycrystals [3,4]. We give an overview on the processing/property relations of Bi-2212 thick film and bulk materials on Ag and MgO substrates and present results from Magnetic Force Microscopy [MFM] measurements of oriented Bi-2212. An overview of polycrystalline bulk and single crystals in the temperature range 40-70 K. The current paths in Bi-2212 differ from Y-123 and Bi-2223 by the absence of wehneltics [4,5]. Magnetically measured j_c in Bi-2212 were comparable to direct current transport in polycrystalline material. Therefore this material shows no weak link behavior. On the edges of Bi-2212 single crystals, our MFM measurements showed the current to be highly confined in a layer close to the grain boundaries parallel to the a-axis. In polycrystalline material their percolating network allows appreciable current densities (>8000 A/cm^2), 72 K, 0 T) even in non-textured bulk Bi-2212. The MFM method makes it possible to localize such type of current distributions with a precision almost on the 10-100 nm scale.

properties (phase assemblages, orientations, and defect sizes) were examined with XRD and SEM/EDS. Reactions of nanoscale Al$_2$O$_3$ with Bi-Sr-Ca-Cu-O melts show promise for producing chemically stable solid-solution Sr$_{2-}$CaAl$_2$O$_6$ defects, where the Sr:Ca ratio is close to the ratio of the precursor powder. Carbon nanotubes had large effects on phase assemblages observed, and an addition caused a high degree of 2212 misorientation in the film. In general, addition of Al$_2$O$_3$ and carbon nanotube defects improved fluxpinning in the range 20 - 60 K, however worsened the 2212 c-axis orientation and reduced transport Jc. The problem of nanoscale defect commingling will be presented. Fluxpinning and critical current density $J_c$ was studied in 0 - 5 T magnetic fields from 5 - 60 K using magnetic and transport methods.

11:30 AM III.1.8
ABNORMAL ANNEALING EFFECT ON (Cu,Tl)-1223 AND (Cu,Tl)-1234 AND ACHIEVING $T_c > 130 K$. K. Tanaka, Y. Tanaka, A. Iyo, N. Terada, M. Takimoto, H. Ikeda, Electrotechnical Laboratory, Tsukuba, JAPAN and CREST of JST; T. Tsukamoto, Science Univ of Tokyo, Dept of Applied Physics, Tokyo, JAPAN; M. Ariyama, Science Univ of Tokyo, Dept of Physics, Noda, JAPAN; K. Tokiwa, S. Miyashita, T. Watanabe, Science Univ of Tokyo, Dept of Applied Electronics, Noda, JAPAN and CREST of JST.

Annealing in flowing nitrogen gas caused the abnormal behaviors on the electronic properties of (Cu,Tl)-1223 and (Cu,Tl)-1234. These abnormal effects were observed in resistivity and Hall number. The resistivity and Hall number decreased with increasing the annealing temperature below 400 °C. On the contrary they decreased above 400 °C. These behaviors relate to the valence change of the thallium element. The XPS measurement on $4f_{7/2}$ core-level binding energy of thallium atom indicated that the valence of the thallium is about +3 below 400 °C. On the contrary valence of the thallium gradually decreased above 400 °C. This change of the valence supplies the holes in the charge reservoir layer instead of the removed oxygen. Moreover we found a drastic improvement of $T_c$ being higher than 130K on (Cu,Tl)-1223 annealing around 550 °C. It can be considered that the Fermi level crosses the band consisting from 6s of thallium atom after the changing of the valence. We discuss the origin of the enhancement of $T_c$ considering the contribution of the 6s orbital of the thallium atom to the band structure.

11:45 AM III.1.9
LARGE AREA Bi-2212 THICK FILMS ON CERAMIC SUBSTRATES. Steffen Kübel, D. Schneider, L. Fäll, P. Stüttelin, L.J. Grueniger, Swiss Federal Institute of Technology Zürich, Dept of Materials, Zürich, SWITZERLAND.

Superconducting films may be the core element of a resistive type fault current limiter. High critical currents in the superconducting state as well as high resistance of the composite structure in normal state are required for this application. The aim of this study was to develop a large area ceramic substrate and to adopt process technology from partial melt processing on silver substrates to achieve similar high critical current capacities in Bi-2212 thick films on ceramic substrates. Thick films of up to 20*20 cm$^2$ were produced by tape-casting and subsequent partial melting. We found that, beside the maximum temperature during partial melting, silver content strongly influences the critical current density of the processed film. Current densities in untextured thick films were up to 2400 A/cm$^2$. These results show that Bi-2212 Ag / MgO composite conductors are a suitable candidate for switching applications in power engineering.