

## SYMPOSIUM II

# High-Temperature Superconductors—Crystal Chemistry, Processing, and Properties

November 27 – December 1, 2000

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\* Invited paper

SESSION III: RECENT PROGRESS IN COATED  
CONDUCTOR SCIENCE AND TECHNOLOGY

Chairs: Masaki Suenaga and Tomio Izumi  
Monday Morning, November 27, 2000  
Room 306 (Hynes)

**8:30 AM \*III.1**

RECENT PROGRESS IN R&D OF COATED CONDUCTORS IN JAPAN. Yuh Shiohara, ISTEC-SRL, Tokyo, JAPAN.

The present state of coated conductor development in Japan is reviewed. Three-dimensional orientation is required in order to obtain high  $J_c$ . Additionally,  $I_c$ ,  $J_e$  and low cost processing are also important factors for the tape applications. In Japan, the development has been proceeded by following three groups. Two of them are responsible for realizing 3-dimensional orientation due to "Substrate Texturing" and "Buffer Layer Alignment" respectively. "Substrate Texturing" group is applying rolling-assisted-biaxially-texturing technique to Ni- and Ag-alloys. Additionally, SOE (Surface Oxidation Epitaxy) process was developed to get an aligned NiO layer on the textured Ni tape by appropriate oxygen heat treatment. The deposited YBCO layer by PLD (Pulsed Laser Deposition) on the SOE tape showed over  $3 \times 10^5$  A/cm<sup>2</sup>. On the other hand, "Buffer Layer Alignment" group has made effort to make a long tape using ISD (Inclined Substrate Deposition) and IBAD (Ion Beam Assisted Deposition) techniques, which both can realize to attain the aligned buffer layer even on a non-textured high strength substrate and were originally developed in Japan. Recently, 6m-long coated conducting tape was fabricated at 0.2m/h using ISD process. For aiming thicker superconducting layer with low cost, the third group has been trying MOD (Metal Organic Deposition) and the LPE (Liquid Phase Epitaxy) techniques. The advantages of the LPE process are mainly to achieve high growth rate and high crystallinity, which can realize high  $I_c$ . However, high growth temperature and reaction between liquid and metal substrate are problems for proceeding development. The problems is going to be solved by using buffer layer and solution saturated by buffer layer material elements. Recently, we succeeded in realizing a feasible construction for the saturation technique which is  $YBa_2Cu_3O_y$  (LPE)/ $YBa_2(CuMg)3O_y$  (LPE)/MgO/Hastelloy. This work was supported by New Energy and Industrial Technology Development Organization (NEDO) through International Superconductivity Technology Center (ISTEC).

**9:00 AM \*III.2**

YBCO COATED CONDUCTOR DEVELOPMENT AT IGC-SUPERPOWER. V. Selvamanickam, C. Park, G. Carota, N. Vo, C. Trautwein, M. Funk, P. Haldar, IGC-SuperPower, Schenectady, NY; U. Balachandran, M. Chudzik, Argonne National Lab, Argonne, IL; P. Arendt, S. Foltyn, J.Y. Coulter, B. Newnam, Los Alamos National Lab, NM.

Intermagnetics is developing YBCO Coated Conductor using flexible metal tapes for electric power applications. These tapes consist of biaxially-textured oxide buffer layers deposited by Ion Beam Assisted Deposition (IBAD). IBAD has been used to deposit buffer layers of yttria-stabilized-zirconia (YSZ) on flexible nickel alloy substrates. Using optimized IBAD conditions, biaxially-textured YSZ has been obtained with a spread in the in-plane texture ranging from 15 to 20 degrees FWHM. YBCO superconducting films have been deposited over the IBAD layer using vapor deposition techniques. One of the techniques that has been successfully used to fabricate high-quality YBCO is Metal Organic Chemical Vapor Deposition (MOCVD). A MOCVD facility with a liquid precursor delivery system is being used to deposit YBCO films on the buffered metal substrates. Optimization of MOCVD process conditions has yielded critical currents up to almost 100 A in 1 micron thick YBCO films deposited on a single buffer layer of IBAD-YSZ, which corresponds to a current density greater than 1 MA/cm<sup>2</sup> at 77 K in self-field conditions. These films exhibit high current densities in the presence of a magnetic field too, with values greater than 200 kA/cm<sup>2</sup> at 75 K and 600 kA/cm<sup>2</sup> at 64 K in a field of 1 T applied perpendicular to the film plane. This work was partly supported by the Air Force Office of Scientific Research, U.S. Department of Energy, and the New York State Energy Research and Development Authority.

**9:30 AM \*III.3**

FABRICATION OF RABiTS-BASED CONDUCTOR BY CONTINUOUS PROCESSES. D.M. Kroeger, F.A. List, D.F. Lee and Ed Specht, Oak Ridge National Laboratory, Oak Ridge, TN.

Our effort to define and address problems associated with the manufacture of conductors by the RABiTS approach will be discussed. We have constructed laboratory scale reel-to-reel systems for performing each of the steps in the fabrication of meter lengths of conductor. A modular approach in which the various steps are carried out in separate systems has been pursued, to permit unconstrained development and study of individual processes. Reel-to-reel systems

for annealing of rolled metal tapes, deposition of buffer layers by electron beam evaporation and rf sputtering, electron beam co-evaporation of Y-BaF<sub>2</sub>-Cu precursor and precursor conversion have been developed. In the precursor conversion system gas flow is transverse to the tape axis to minimize boundary effects and facilitate rapid removal of HF. In addition, a reel-to-reel x-ray diffraction system has been developed such that lengths of tape can be examined at any step of the fabrication process. Meter lengths of conductor, processed in these systems and in collaboration with industrial partners, have shown high  $J_c$ . Local variations in  $J_c$  are thought to be associated with compositional variations occurring in the precursor deposition process as well as with variations in the completeness of precursor conversion as affected by sample geometry and local gas flow conditions.

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**10:15 AM \*III.4**

ENHANCING THE CURRENT TRANSPORT IN EX SITU PROCESSED YBCO COATED CONDUCTORS. R. Feenstra, D.K. Christen, D.T. Verebelyi, D.F. Lee, M. Paranthaman, A. Goyal, B.W. Kang, S.W. Lu, P.M. Martin and D.M. Kroeger, Oak Ridge National Laboratory, Oak Ridge, TN.

Processes featuring BaF<sub>2</sub> as one of the reactants to form YBCO during ex situ annealing are attractive for coated conductor technology because of inherent process simplification, absence of rate limiting effects during HTS deposition, as well as a demonstrated compatibility with basic coated conductor substrates. Starting from a precursor layer which may be conveniently produced by vacuum evaporation or solution chemistry, superconducting YBCO is formed by annealing at 700-800°C, controlled by a decomposition reaction of BaF<sub>2</sub> with water, supplied at a specified flow rate and partial pressure. Thus the conversion reaction has similarities with CVD, however, because the YBCO epitaxial growth occurs beneath a layer of residual precursor, there is also a strong resemblance to bulk processing. In research directed at enhancing the total critical current  $I_c$  by increasing the precursor thickness, it is found that the thickness has a pronounced effect on the initial nucleation and growth at the substrate interface. Increasing the thickness also increases the probability of random nucleation away from the substrate. Using countermeasures to reduce these effects, high- $J_c$  YBCO coatings with thickness  $\geq 1 \mu\text{m}$  were produced. The current transport in these conductors exceeds 100 A/cm width at 77 K.

**10:45 AM \*III.5**

GROWTH OF THICK YBCO FILMS WITH HIGH CRITICAL CURRENT DENSITY BY PULSED LASER DEPOSITION. S.R. Foltyn, Q.X. Jia, P.N. Arendt, T.G. Holessinger, H. Kung, J.F. Smith, Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM.

In the development of YBCO coated conductors based on ion beam assisted deposition (IBAD) and pulsed laser deposition, we have fabricated meter lengths of centimeter-wide tape with critical current ( $I_c$ ) values (75 K, self field) of 100 A and above. On shorter lengths,  $I_c$  values of over 200 A have been reached. In principal, arbitrarily high  $I_c$  levels are achievable by simply increasing the thickness of the YBCO, which we have demonstrated with  $> 6 \mu\text{m}$  thick YBCO on single crystal substrates with an  $I_c$  equivalent of 700 A/cm width. Similarly, using IBAD-on-metal substrates, we found that as the superconductor thickness was increased to about 1.5  $\mu\text{m}$  the product of critical current density ( $J_c$ ) and thickness increased to a maximum value of about 220 A/cm. Above this thickness, however, additional YBCO produced no increase in  $I_c$ . We investigated this phenomenon by ion-milling the YBCO, remeasuring  $I_c$ , and repeating, thereby allowing us to determine the distribution of  $J_c$  through the entire film thickness. We discovered two problems. First, many samples with thick YBCO had a 0.25  $\mu\text{m}$  thick nonsuperconducting layer at the bottom interface; the cause of this "dead layer" is presently unknown. The second problem was that essentially no supercurrent was carried at thickness levels above 1.5 - 2  $\mu\text{m}$ . Through electron microscopy, we found that this was directly attributable to a transition from a dense YBCO microstructure to a porous, disconnected one at about 1.5  $\mu\text{m}$ . By modifying the superconductor deposition process, we have been able to overcome this problem and make dense coatings up to 5  $\mu\text{m}$  thick. The performance has improved accordingly, with a best result for an IBAD-on-metal substrate of 1.4 MA/cm<sup>2</sup> at a thickness of 3.7  $\mu\text{m}$ : the equivalent  $I_c$  is over 500 A/cm.

**11:15 AM \*III.6**

YBCO FILMS BY THE INSITU METHOD: PROCESSING AND PROPERTIES. Robert H. Hammond, William Jo, Tsuyoshi Ohnishi, Luke S.-J. Peng, Weizhi Wang, Ann Marshall and M. R. Beasley,

Stanford University, Laboratory for Advanced Materials, Stanford, CA; Eric Peterson, Los Alamos National Labs, Los Alamos, NM.

In order to achieve a high rate and large area process, overall system oxygen pressures lower than is required for thermodynamic stability is required. The use of activated oxygen is being investigated. Low fluxes of atomic oxygen apparently does stabilize YBCO growth over a range of flux and temperature. Unexpectedly the TEM and SEM suggest the growth is from a liquid flux, which has allowed ordered growth at rates approaching 10 nm/sec so far. Critical currents up to a MA/cm<sup>2</sup> at 77 K and 0T are found so far.

#### 11:45 AM II1.7

**ELECTROMAGNETIC PROPERTIES OF DOPED AND UNDOPED YBCO BICRYSTALS.** George Daniels, Alex Gurevich, David Larbalestier Applied Superconductivity Center, University of Wisconsin Madison, WI.

We have made thin film bicrystals of both pure YBCO and those in which Y has been partially replaced by Ca (0.7Y 0.3Ca). Concentrating on low angle bicrystals having [001] tilt misorientations lying in the range 3-7°, we find domains of temperature and field well above 4K in which both the intergranular (J<sub>b</sub>) and the intragranular current densities (J<sub>c</sub>) are higher than in pure YBCO. This is in spite of the fact that the transition temperature of Ca-doped YBCO is depressed from 91 to 71K. For example, for 5° [001] tilt bicrystals tested at 44K, that is T/T<sub>c</sub> = 0.48 for pure YBCO, T/T<sub>c</sub> = 0.56 for as grown Ca-doped YBCO and T/T<sub>c</sub> = 0.62 for oxygen-overdoped Ca-doped YBCO, there is a marked improvement in both intergranular properties on doping. J<sub>b</sub>(1T) is 0.51, 0.34 and 2.3 A/cm<sup>2</sup>, respectively. There is a broad separation between J<sub>b</sub> and J<sub>c</sub> for the pure YBCO and the as-grown Ca-doped sample, but this separation almost disappears for the lowest T<sub>c</sub>, oxygen overdoped sample. A broad study of the low angle properties of both monolithic and multilayer samples of varying misorientation angle will be presented. The results obtained appear to show that the excellent results of overdoping observed on 24° boundaries in small fields are also obtained in fields of several Tesla for low angle grain boundaries.

#### SESSION II2: RECENT PROGRESS ON BI-BASED SYSTEMS – SYNTHESIS, PROPERTIES, AND PERFORMANCE

Chairs: Victor A. Maroni and Martin W. Rupich  
Monday Afternoon, November 27, 2000  
Room 306 (Hynes)

#### 1:30 PM \*II2.1

**PHASE FORMATION IN BI BASED HIGH T<sub>c</sub> SUPERCONDUCTORS.** R. Flükiger, E. Walker, M. Lomello-Tafin, E. Giannini.

The Bi,Pb(2223) tapes with the highest critical current density values contain around 20% voids, which is directly correlated to the commonly used non-equilibrium reaction, leading from the initial Bi(2212) phase to the final, textured Bi,Pb(2223) platelets. A marked enhancement of the critical current density can thus only be obtained by forming denser samples, thus strongly enhancing the number of percolation paths. Since the formation of Bi,Pb(2223) inside the Ag sheath involves the presence of small quantities of liquid (transient liquid: <10%), the question is open whether new reaction paths can be found involving larger quantities of liquid phase. The possibility of the equilibrium reformation of Bi,Pb(2223) after heating well above its decomposition temperature has been investigated on partially melted samples. Precursor rods of Bi,Pb(2223) and Pb free Bi(2223) were zone melted crucible-free close to 1'100°C in air, and the kinetics of the reactions during prolonged heat treatments between 850 and 880°C was studied. So far, we did not succeed in reforming the Pb free Bi(2223) phase. In contrast to earlier reformation experiments, however, Bi,Pb(2223) was formed in our highly dense samples (>95%) after prolonged heat treatments, even without intermediate crushing. A surprising result is that no major Pb losses were observed: our results suggest that the reformation process is influenced by the presence of crucible material, showing a more or less important reaction with the transient liquid. Possible consequences of this reaction behavior for the formation of highly dense Bi,Pb(2223) tapes with higher critical current density are discussed.

#### 2:00 PM II2.2

**MULTIPLY SATURATED LIQUIDS IN PORTIONS OF THE BSCCO SYSTEM.** V.J. Styve, J.K. Meen and D. Elthon, Department of Chemistry and Texas Center for Superconductivity, University of Houston, Houston, TX.

This study synthesizes our completed work on the BiO<sub>1.5</sub>-SrO-CaO-CuO (BSCCO) quaternary system that includes 3 known super-

conductors belonging to the homologous series Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>(n-1)</sub>Cu<sub>n</sub>O<sub>(2n-4)</sub>. The BSCCO quaternary is quite complicated, containing several binary, ternary and quaternary phases many of which exhibit solid solutions or variable alkaline earth ratios. The majority of the quaternary phase volume is occupied by primary phase volumes (PPVs) of AeO (CaO with dissolved SrO) and CuO. Separating these two volumes is a thin region of PPVs for the alkaline earth cuprates. The dominance of these volumes in the quaternary results in the confinement of PPVs of Bismuth bearing phases to a small portion of the quaternary. Moreover, Bi-AeO's have PPVs between Bi<sub>2</sub>O<sub>3</sub> and AeO PPVs. Thus, liquidus relations of all ternary and quaternary phases exist in a small region of interest (ROI). Within this ROI, phases relations very rapidly with temperature and composition. Our study has determined the location of this ROI and we have studied the reaction relationships in great detail concentrating on the composition of the liquids and crystalline phases in equilibrium at each temperature and composition. Over 1000 experiments have been conducted within the system and analyzed by electron microprobe to determine the composition of both quenched liquids and crystalline solids within each experiment. This presentation will discuss the location of the ROI that emanates from a small area on the face of the BiO<sub>1.5</sub>-SrO-CuO ternary projecting towards the analogous point on the BiO<sub>1.5</sub>-CaO-CuO face. The region is bounded by PPVs of AeO, AeCuO's, CuO and Bi-LMCs (Low temperature Melting bismuth based Compounds).

#### 2:15 PM II2.3

**SINGLE CRYSTALS OF Bi-2223 PHASE: FORMATION, GROWING MECHANISM AND SUPERCONDUCTING PROPERTIES.** Sergey Lee, Ayako Yamamoto and Setsuko Tajima, Superconductivity Research Laboratory, ISTEC, Koto-ku, Tokyo, JAPAN.

Bi-based 2223 phase with the superconducting transition temperature of 110 K has been considered as one of the most attractive compound for basic research and application. However, fundamental studies of this phase seriously restricted due to the lack of high-quality single crystals. In the present work single crystals of 2223 phase have been grown using KCl as flux. Influence of many factors (crucibles materials, chemical and phase composition of the precursor powder, temperature and duration of heat treatment, precursor/flux ratio and evaporation rate of the flux) on the formation and crystal growth were studied. By optimization of these parameters, single crystals of Pb-doped and Pb-free Bi-2223 phase with the size of 0.5 mm were grown in MgO crucibles by isothermal heat treatment in the temperature range of 850-870 C for 100 hours. At the first stage of heat treatment (<15 h.) rapid formation of almost single-phase Bi-2223 in the KCl flux for both leaded and unleaded compositions was established and detailed investigation of the microstructure and kinetic of 2212 to 2223 phase transformation was performed. It was proposed that fast formation of Bi-2223 in the KCl is limited by dissolution of 2212 in the flux and further two-dimensional nucleation processes, in contrast to the conventional solid-state reactions techniques, where formation of 2223 phase mainly controlled by diffusion. The most plausible mechanism of crystal growth during isothermal heat treatment of single- phase sample is material transport from small to large crystals, so-called Ostwald ripening. Quality of as-grown single crystals, effect of Pb-doping and post-annealing in different atmospheres on the oxygen content and superconducting properties were studied by XRD, SEM/EDX, ISP/AES, susceptibility and resistivity measurements, thermal and wet chemical analyses.

#### 2:30 PM II2.4

**PHASE RELATIONS AROUND THE Bi-2221 AND Bi-2201 PRIMARY PHASE VOLUMES.** James K. Meen, Binh Nguyen, Vance J. Styve, Karoline Müller, and Don Elthon Department of Chemistry and Texas Center for Superconductivity, University of Houston, Houston, TX.

Near-stoichiometric Bi-2212 melts incompatibly at all values of p(O<sub>2</sub>). Liquids in the primary phase volume of Bi-2212 have more Bi than Bi-2212. Our best estimate of the composition of the most Bi-depleted liquid in equilibrium with Bi-2212 crystallizes a Bi-2212 solid solution (Bi:Sr:Ca:Cu=2.2:2.0:0.8:2.0) at 880°C in oxygen. The liquidus-solidus interval is only 10°C and the second phase to crystallize is a Raveau quaternary solid solution (Sr:Ca ranges from 3 to 6; Bi excess). These two phases change composition only slightly (low-T Bi-2212 is 2.2:1.9:0.9:2.1) until liquid is exhausted. Liquids in this system contain Cu<sub>2</sub>O and the p(O<sub>2</sub>)=1 atm isobar lies within the Bi<sub>2</sub>O<sub>3</sub>-SrO-CaO-CuO-Cu<sub>2</sub>O quinary, thus truncating some of the phase relations. Furthermore, tie-lines between Bi-2212 solid solutions and coexisting liquids can only sensibly be interpreted in the quinary system; they do not show a simple trend toward Bi. A pseudobinary section from Bi-2201 towards CuO in pure oxygen also demonstrates a complex change in composition of the solid solution with change in the liquid composition. Increase in Cu in the bulk composition results in an increase in both Bi and Cu in the Bi-2201 solid solution as the

liquidus phase changes from  $\text{Sr}_{14}\text{Cu}_{24}\text{O}_x$  to  $\text{CuO}$ . Tying together the phase relations of Bi-2201 and Bi-2221 (and Bi-2223) remains a major problem in phase relations of Bi superconductors. The portion of the quinary space in which these complex phase relations are contained is small and the temperature interval over which they occur is limited. This requires careful characterization of the products of experiments in this field.

### 3:15 PM \*II2.5

RECENT PROGRESSES IN THE DEVELOPMENTS OF Bi-2212 SUPERCONDUCTORS. Kazumasa Togano, National Research Institute for Metals, Tsukuba, Ibaraki, JAPAN.

Among the many high temperature superconductors,  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$  (Bi-2212) is one of the most promising materials for the practical application of magnetic field generation because of its extremely high transport critical current density  $J_c$  at low temperatures even in strong applied magnetic fields. Although several companies have produced 100m-class long Bi-2212/Ag superconductors, they have not yet been deployed by industry in commercial basis. One of the obstacles for commercialization is the high cost of conductor (KA-m). In order to reduce the conductor cost, it is essential to increase the engineering  $J_c$  and/or to reduce the amount of expensive Ag as much as possible. This paper reports our recent progresses toward these objectives. We have been carrying out an extensive work to optimize the processing parameters, investigating the relationship between the microstructure and transport  $J_c$ . We have found that the improvement of c-axis grain alignment for the whole oxide layer is the key to achieve larger transport  $J_c$ . In addition to this c-axis alignment, we have succeeded to achieve in-plane (a,b-axis) alignment by carrying out the melt process in temperature gradient. The tape showed a transport  $J_c$  value of well over 500kA/square-cm at 4.2K and 10T. The paper also proposes a new approach to reduce the amount of Ag in the conductor, while keeping the advantage of Ag. The technique involves the preparation of Ag/Ni clad tape as a cost-effective substrate instead of expensive Ag. Bi-2212 layer with high  $J_c$  comparable to that of conventionally processed Bi-2212/Ag tape has been successfully produced on the Ag/Ni substrate. The present status of scale-up production of Bi-2212/Ag conductors and the constructions and the tests of prototype superconducting magnets are also presented.

### 3:45 PM \*II2.6

COMPETITIVENESS OF PROCESSING GOALS FOR ATTAINING IDEAL MICROSTRUCTURE IN MELT PROCESSED Bi2212/Ag CONDUCTORS. M.O. Rikel, Y. Yuan, S. Rogers, N.A. Scarborough, D. Wesolowski, E.E. Hellstrom, Applied Superconductivity Center, Univ. Wisconsin-Madison, Madison, WI.

Single-phase, 100% dense, strongly textured microstructure with nanometer-scale inhomogeneities is the ultimate processing goal for most HTS materials. In melt processed Bi2212/Ag conductors, the reported microstructures do not yet meet the above criteria. In this work, we focus on possible restrictions of the melt-processing route for Bi2212/Ag conductors. We show that almost all processing goals are to a certain extent competitive: *Porosity-Phase Purity* The soak time in the melt state necessary to attain a certain level of porosity depends on conductor geometry being longer for mechanically stiffer shapes (round wires, multifilamentary tapes). Longer soak times lead to coarser second-phase particles and difficulties in their consumption during solidification. *Texture-Phase Purity* Highly textured 2212 layers that are formed during solidification become obstacles for mass transfer in the system, because the diffusion along the c-axis is much slower than in the ab-planes. *Texture-Porosity* The smaller surface energy of BSCCO-melt/Ag pair as compared to the surface energy of Ag with other phases (including  $\text{O}_2$ ) is a very probable reason for a well aligned 2212 layer close to BSCCO/Ag interface but is also an obstacle for eliminating porosity. Strategies to overcome the competitiveness of processing goals are discussed.

### 4:15 PM \*II2.7

RECENT DEVELOPMENTS OF Bi-2212 WIRE AND CABLE. L.R. Motowidlo, R.S. Sokolowski, IGC Advanced Superconductors, Waterbury, CT; T. Hasegawa, Y. Aoki, T. Koizumi, N. Ohtani, Showa Electric Wire & Cable, Kanagawa, JAPAN.

Recent Progress in the development of high performance  $\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_x$  multifilament round wire and cable is presented. The critical current and bend strain properties of a 6x1 cable design as function of applied magnetic field up to 8 Teslas will be presented. Cable designs and results including silver, silver alloy, and nickel cores will be discussed. The cables are fabricated with six active strands each 0.8 mm diameter. The conductor utilized in the cables was a double stack design (7x91) comprising of 637 filaments. The critical current ( $I_c$ ) of the cable was measured at liquid helium in applied magnetic fields up to 8T. The self-field  $I_c$  of the strand is over 500 Amps. The  $J_c$  is over 400,000 A/cm<sup>2</sup>.

### 4:45 PM II2.8

REDUCTION IN AC TRANSPORT SELF-FIELD LOSSES OF POWDER-IN-TUBED (Bi,Pb)-2223 TAPE-FORM CONDUCTOR BY RESISTIVE BARRIERS. A. Oota, R. Inada, T. Uno, Y. Takatori and P.X. Zhang, Toyohashi University of Technology, Toyohashi, Aichi, JAPAN; H. Fujimoto, Railway Technical Research Institute, Kokubunji, Tokyo, JAPAN; L. Zhou, Northwest Institute for Nonferrous Metal Research, Shaanxi, CHINA.

We succeeded in reducing the AC transport self-field losses at 77 K and 50 Hz in (Bi,Pb)-2223 tape-form conductor fabricated using a four-roller machine, by introduction of Bi-2201 sheets as resistive barriers in an arrangement parallel to a wide surface of the conductor. Numerical calculations based on the theory of Norris show that the origin for this loss reduction is ascribed to the division of field-free-core under AC current transmission, caused by introduction of resistive barriers. There is, however, some deterioration in current capacity of the conductor ascribed to some reactions of Bi-2201 sheets with (Bi,Pb)-2223 filaments during heat treatments. Improvements for fabrication process to suppress the reaction, together with some experiments using other barrier materials than Bi-2201, will be presented.

## SESSION II3: COATED CONDUCTOR SUBSTRATES AND BUFFER LAYERS

Chairs: Ron Feenstra and Yuh Shiohara  
Tuesday Morning, November 28, 2000  
Room 306 (Hynes)

### 8:30 AM \*II3.1

BIAXIAL TEXTURE CONTROL OF YBCO FILMS ON METALLIC SUBSTRATE BY SURFACE-OXIDATION EPITAXY METHOD. Kaname Matsumoto, Kyoto Univ, Dept of Materials Science, Kyoto, JAPAN; Tomonori Watanabe, Furukawa Electric, Nikko, JAPAN; Toshihiko Maeda, SeokBeom Kim, Izumi Hirabayashi, SRL/ISTEC, Nagoya, JAPAN.

Surface-oxidation epitaxy (SOE) technique has been investigated for controlling biaxially textured YBCO films on metallic tapes. Highly biaxially textured NiO layers are grown on cube textured Ni tapes by SOE. High Jc YBCO films with about 0.5MA/cm<sup>2</sup> (0T, 77K) has been obtained on the SOE grown NiO/Ni tapes. Grain boundary thermal grooves in Ni tapes, however, produce grooves in NiO. The NiO grooves become a cause of large angle grain boundaries and Ni contamination in YBCO films, resulting in low Jc values. To reduce the number of large angle grain boundaries in YBCO films, oxide cap layer for flattening NiO surface is very effective. MgO, YSZ, CeO<sub>2</sub>, etc. are suitable for the cap layer material. These materials are epitaxially grown on NiO/Ni tapes by physical vapor deposition techniques such as the pulsed laser deposition, the sputtering, the electron beam deposition, etc. For example, a thin MgO film with the thickness of 50-100 nm caps the grooves of NiO surface and suppresses the Ni contamination completely. We have successfully fabricated the biaxially textured 50m-long NiO/Ni tape by SOE. The in-plane texture FWHM of NiO layer was about 10-14 degrees throughout the length. An effort is underway to deposit the cap layer on the moving NiO/Ni tape by physical vapor deposition system. In this paper, the microstructure of YBCO films deposited NiO/Ni tapes with cap layer and their superconducting properties will be reported..

### 9:00 AM \*II3.2

MECHANISMS OF ION-BEAM-ASSISTED TEXTURING OF YSZ LAYERS. Jürgen Dzick<sup>1,2</sup>, Sibylle Sievers<sup>2</sup>, Jörg Hoffmann<sup>2</sup>, Lars-Oliver Kautschor<sup>2</sup>, Herbert C. Freyhardt<sup>1,2</sup>. <sup>1</sup> Zentrum für Funktionswerkstoffe GmbH, Göttingen, GERMANY. <sup>2</sup> Institut für Materialphysik, Universität Göttingen, GERMANY.

Biaxially textured yttria-stabilized zirconia (YSZ) buffer layers deposited by an ion-beam-assisted deposition process (IBAD) serve as templates for high-current carrying  $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{7-x}$  films. To scale-up the IBAD process for the deposition of buffer layers on large-area substrates it is important to understand the dominant mechanisms involved in the texture development. Furthermore, deposition parameters, e.g. the substrate temperature or the intrinsic divergence of the assisting ion beam, must be taken into account. Our present investigations (TEM and x-ray diffraction analyses) of the texture evolution of IBAD-YSZ films during film growth indicate a sequence of at least three different characteristic growth stages. During an initial step, wire-textured (011)-oriented grains nucleate. This preferential orientation is stress induced due to the ion bombardment. In a second step, (001)-oriented grains nucleate epitaxially on (011)-grains which are favourably oriented with respect to the ion beam. Therefore, a preferential in-plane orientation of these (001)-oriented grains is induced. In the final stage a relatively slow improvement of the in-plane texture occurs due to a growth selection

of those (001)-oriented grains with their (111) axis parallel to the assisting beam. The relevant underlying mechanisms have been identified. Internal film stresses may influence the orientation of nuclei. Ion-beam-induced homoepitaxy generates epitaxial relations between nuclei and underlying film layers. Finally, anisotropic sputter rates cause alterations in growth rates of differently oriented grains that lead to an overgrowth of misoriented grains. Accordingly, the distinct growth steps are dominated by different ion beam induced texturing processes. The results will be summarized in the form of a schematic growth model for IBAD-YSZ films. Furthermore, the influence of different substrate temperatures and of the divergence of the assisting ion beam will be discussed.

Work in part supported by Alcatel and Siemens, within the frame of projects of the BMBF and European Union, respectively.

### 9:30 AM II3.3

**FORMATION OF BIAXIALLY TEXTURED MgO BUFFER LAYERS USING ION-BEAM ASSISTED PULSED LASER DEPOSITION.** R. Hühne, Ch. Beyer, C.-G. Oertel, W. Skrotzki, Technische Universität Dresden, Institut fuer Kristallographie und Festkörperphysik, GERMANY; B. Holzapfel, L. Schultz, Institut fuer Festkörper- und Werkstofforschung, Dresden, GERMANY.

MgO thin films were deposited on amorphous substrates using pulsed laser deposition. The texture formation was investigated in-situ with the help of RHEED patterns. Furthermore the microstructure of the films was observed by AFM. Without an assisting ion beam strong fibre textures are observed changing with temperature. Using an ion beam under  $55^\circ$  with respect to the substrate normal biaxially textured films are grown. Under certain conditions a cube texture is observed in the nucleation state below 10 nm. During further growth this nucleation texture changes to a texture with the (200) direction parallel to the ion beam. This behaviour can be explained with an anisotropic sputter rate of MgO found with sputter experiments on single crystals. Furthermore MgO films were grown homoepitaxially on MgO single crystals. The films are stressed depending on the deposition parameters. On the basis of these results the influence of the laser deposition process as well as the influence of the assisting ion beam on the texture formation will be discussed.

### 10:15 AM II3.4

**TEXTURE CONTROL DURING THE ION BEAM ASSISTED DEPOSITION OF MgO.** Luis A. Zepeda-Ruiz, Princeton Materials Institute and Department of Mechanical and Aerospace Engineering, Princeton University, Princeton, NJ; Liang Dong, Cadence Corporation, San Jose, CA; David J. Srolovitz, Princeton Materials Institute and Department of Mechanical and Aerospace Engineering, Princeton University, Princeton, NJ.

MgO thin films deposited on amorphous substrates are used as structural templates for the deposition of superconducting thin films. In order to achieve the necessary degree of in-plane texture, ion beams have been employed to control the texture evolution during the growth of these films. In this presentation, we examine the early stages of film growth (up to and including island coalescence), where the film texture is primarily established. Several types of three-dimensional molecular dynamics (MD) simulations were performed. In the first, we examined sputter yield as a function of crystal orientation relative to the ion beam. This establishes the strength of the channeling effect that gives rise to texture and the angular width of the channels. Additional simulations were performed with the ion beam impinging on small islands (over a range of island sizes) to determine whether the channeling effect observed in crystals also operated for islands. Although all very small islands are destroyed when hit by high energy ions, the ions only destroyed larger islands not oriented for channeling. This shows that island nucleation rates are orientation dependent, even on an amorphous substrate. A third set of simulations was performed to investigate whether island rotation during coalescence is possible and to assess its impact on texture development. These results basic input for the development of models for texture development during the early stages of ion beam assisted deposition.

### 10:30 AM II3.5

**CONDUCTIVE-OXIDE BUFFER LAYERS FOR HIGH CRITICAL CURRENT DENSITY YBCO COATED CONDUCTORS.** Tolga Aytug, J.Z. Wu, Univ of Kansas, Dept of Physics and Astronomy, Lawrence, KS; C. Cantoni, D. Verebelyi, D.K. Christen, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN; B.W. Kang, A. Goyal, E.D. Specht, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; M. Paranthaman, Chemical and Analytical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN.

The ultimate development of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO)-based coated conductors for electric power applications will require electrical stabilization of the HTS coating. Epitaxial buffer layers of electrically

conductive  $\text{LaNiO}_3$  (LNO) and  $\text{SrRuO}_3$  (SRO) have been deposited on biaxially textured nickel substrates (RABiTS) by *rf*-sputter deposition and/or pulsed laser ablation. In the present case, we establish electrical contact between the thin HTS layer and the thick metal, Ni, substrate, improving electrical stability in the event of a transient to the dissipative regime. The new conductive buffer layer structure comprises the layer sequence of SRO/LNO/Ni. Due to its significance as a base layer, we have investigated the optimum deposition conditions for the epitaxial growth of LNO on Ni. High quality LNO layers were achieved in oxidizing conditions at temperatures between 450-500°C. In contrast to the out-of-plane texture, the in-plane texture of the LNO layers was found to be sensitive to the oxygen partial pressure during deposition. Conductive SRO films were then deposited to yield a chemically compatible structure for the subsequent growth of YBCO films, since it was observed that the superconducting properties of YBCO coatings suffer degradation from the significant Ni contamination through the single LNO layers. Epitaxial deposition of YBCO films by PLD on the SRO/LNO/Ni multilayer structure yielded self-field  $J_c$  values as high as  $1.4 \times 10^6$  A/cm<sup>2</sup> at 77K. In addition to an excellent structural and chemical compatibility, SRO/LNO bilayers show good electrical contact with YBCO. Extension of conductive-oxide buffer layers to other metal systems, i.e. Ni-alloy, has also been explored.

### 10:45 AM II3.6

**NEW NITRIDE BUFFER LAYERS ON TEXTURED Ni FOR YBCO SUPERCONDUCTING TAPE.** I.W. Kim, S. Sambasivan and S.A. Barnett, Applied Thin Films, Inc. A. Goyal - Metals and Ceramic Division, Oak Ridge National Laboratory, C.E. Oberly, C. Varanasi, P. Barnes, R. Biggers, Air Force Research Laboratory, Dayton OH.

Compared with more conventional oxide buffer such as Yttria-stabilized Zirconia and Ceria, nitride buffers offer the advantages of high electrical conductivity, potentially allowing shunting of current into the Ni tape, better mechanical toughness than oxides, and relative ease of high-rate deposition by reactive sputtering. Although, nitride growth avoids any difficulty with NiO formation at the substrate, oxidation of the nitride surface during YBCO growth was main difficulties in implementing this material for YBCO superconducting tape. In this talk, we describe results on a new transition metal nitride based buffer. Unlike nitrides buffer previously studied, such as TiN, newly developed composition showed a great amenity to the subsequent oxide growth, including epitaxial YBCO. Buffers of thicknesses  $\sim 200$ nm were grown on Ni RABiTS by reactive magnetron sputtering at  $\sim 500$ - $600^\circ\text{C}$ . X-ray diffraction (XRD) phi-scans of the nitride (111) reflection showed cube-on-cube epitaxy, despite large lattice mismatch. Typical full width half maximum (FWHM) values were  $10^\circ$ , while that of the textured Ni substrate was  $\sim 8^\circ$ . A thin ( $<< 100$  nm thick) layer of YSZ was coated as an intermediate layer on the nitride buffer coated Ni, followed by an  $\sim 300$ nm thick YBCO layer. The XRD phi scan showed a YBCO (226) reflection with a FWHM of  $< 13^\circ$ , and XRD rocking curve showed FWHM of  $< 6^\circ$  on (005) reflection, indicating relatively good in-plane and out-of-plane texture. Simple, easily-scalable, very low cost method was used for YSZ deposition. The reason for amenity of buffer and result of on-going superconductivity measurement will be discussed.

### 11:00 AM II3.7

**DETERMINATION OF THE FREE ENERGY BARRIER TO NUCLEATION IN SOL-GEL DERIVED RARE EARTH OXIDE BUFFER LAYERS ON BIAXIALLY TEXTURED NICKEL.** Harald Dobberstein, Nisanart Navapan, Robert W. Schwartz, Clemson University, Dept. of Ceramic and Materials Engineering, Clemson, SC; P.G. Clem, Sandia National Laboratories, Materials and Process Sciences Directorate, Albuquerque, NM.

For the fabrication of 2nd generation high temperature superconductor tape products, chemical solution deposition routes are recently under considerable investigation for both the buffer layer and the superconductor. While in physical and chemical vapor deposition techniques, primarily surface and interface energy considerations dictate the nucleation and growth characteristics of the thin films, in sol-gel routes, the nucleation and growth behavior is defined by the difference in free energy between the amorphous and crystalline states. For a further fundamental understanding of the nucleation mechanism, it is essential to determine the free energy barrier to nucleation in these films independent to the barrier to growth. A direct, non-Arrhenius method was used to determine the free energy of nucleation by measuring the isothermal nucleation and growth rates. Because this non-Arrhenius method reveals the activation energy rather than the enthalpy, entropic contributions, such as the differences between in short-range order, can be obtained. We have also extended this method, which was first introduced for a-silicon by Frank G. Shi, to include surface and interface energy considerations into the free energy barrier. In this presentation, results will be shown for the free energy barrier to nucleation of rare earth oxide films on

biaxially textured nickel, and compared to free energy barriers of more commonly encountered oxides. Interface and surface energy data were obtained by a theoretical model based on the nearest neighbor broken bond (NNBB) model, as well as by calculations, which employ a molecular dynamic modeling approach. Film and powder samples were characterized by X-ray diffraction, DSC/DTA, SEM and TEM.

#### 11:15 AM II3.8

ISSUES RELATED TO THE FABRICATION OF LONG LENGTHS OF SOLUTION DERIVED YBCO COATED CONDUCTORS.

M. Paranthaman, T.G. Chirayil, S. Sathyamurthy, D.B. Beach, F.A. List, A. Goyal, D.F. Lee, S.Y. Lu, D.M. Kroeger, C. Cantoni, and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

A low-cost, non-vacuum, solution precursor route has been developed to fabricate epitaxial buffer layers and superconductors on biaxially textured-Ni (100) substrates. Meter lengths of sol-gel buffered-Ni tapes were produced. The sol-gel seed layers were dense, continuous and crack-free. YBCO films with a  $J_c$  of over 140 KA/cm<sup>2</sup> at 77 K were grown on all solution buffers using Pulsed Laser Deposition. In addition, YBCO films with a  $J_c$  of over 1 MA/cm<sup>2</sup> at 77 K and self-field were also grown solution based cap layers using TFA precursors. We will discuss in detail about issues related to the scaling up of solution based YBCO coated conductors.

Supported by the U.S. DOE, Division of Materials Sciences, Office of Science, Office of Energy Efficiency and Renewable Energy, and Office of Power Technologies-Superconductivity Program. The research was performed at the Oak Ridge National Laboratory, Oak Ridge, TN; managed by UT-Battelle, LLC for the USDOE under contract DE-AC05-00OR22725

#### 11:30 AM II3.9

COMPARISON BETWEEN THE EFFECTS OF SUBSTRATE SURFACE CONDITIONS AND ION IRRADIATION DEFECTS ON FLUX-PINNING PROPERTIES OF YBCO EPITAXIAL THIN FILMS. C. Cantoni, D.K. Christen, D.T. Verebelyi, E.D. Specht and D. Norton, Oak Ridge National Laboratory, Oak Ridge, TN; D.J. Miller, Argonne National Laboratory, Argonne, IL.

Understanding the origin of strong flux-pinning in epitaxial YBCO films is very important for coated-conductor applications that require high critical currents in magnetic field. Although several types of defects (dislocations, grain-boundaries, anti-phase boundaries (APB), precipitates etc.) are commonly observed in YBCO films, it is still unclear which is mainly responsible for the high  $J_c$ 's measured. Extended edge and screw dislocations originating during YBCO islands growth and coalescence can be strong pinning sites, but their density ( $B_0=0.02-0.1$  T) is too low to account for typical irreversibility fields of 6-8 T. Our approach is to study pinning properties of samples in which a particular type of defect has been enhanced with respect to the others. We focused on high-quality YBCO thin films deposited by PLD on STO substrates and have conducted careful transport  $J_c$  measurements in applied magnetic fields up to 14 T for three cases. 1. Columnar defects with a density corresponding to matching fields as high as 5 T produced in YBCO films by ion irradiation. 2. YBCO films grown on reconstructed vicinal surfaces characterized by a miscut angle in the range 0.4 to 8 degrees to influence the formation of APB's and other structures, and minimize island growth-related dislocations. 3. Defects created on the STO surface by ion irradiation prior to YBCO deposition to provide nucleation sites for additional defects in the superconductor. The existence of  $J_c$  anisotropy, and the difference in transport, structural, and morphological properties of these samples provide interesting insights into the role of different defects on flux-pinning. Research sponsored by the U.S. Department of Energy under contract DE-AC05-00OR22725 with the Oak Ridge National Laboratory, managed by UT-Battelle, LLC.

#### SESSION II4: CHEMICAL METHODS FOR DEPOSITING COATED CONDUCTORS

Chairs: Venkat Selvamanickam and Rene Flukiger  
Tuesday Afternoon, November 28, 2000  
Room 306 (Hynes)

#### 1:30 PM \*II4.1

APPLICATION OF SOLUTION DEPOSITION TECHNIQUES TO COATED CONDUCTORS. Kamel Salama and Srivatasan Sathyamurthy, Texas Center for Superconductivity and Department of Mechanical Engineering, University of Houston, Houston, TX.

Coated conductors offer a viable alternative to the BSCCO Powder-in-tube tapes. Currently, results are being reported for conductors with buffer and superconductor layers processed using conventional thin film deposition techniques which are vacuum based. These aspects of fabrication routes makes it difficult to scale up at

reasonable cost. This presentation describes a research program directed at the development of fabrication methods for metallorganic decomposition (MOD) routes for buffer and Y123 layers using simple solution based techniques which can be readily scaled. Processing of oriented buffer layers of barium zirconate and strontium titanate using simple metallorganic decomposition routes have been achieved [1]. These processes, which use precursor solutions made by dissolution of simple acetates in common solvents like acetic acid and methanol, produce highly oriented buffer layers even when processed in a partial reducing atmosphere. This route, under optimized process conditions, yields Y123 films with  $J_c$  of the order of  $10^6$  A/cm<sup>2</sup> on single crystal substrates. These results give promise to the applicability of these scaleable solution deposition techniques to coated conductor fabrication.

[1] S. Sathyamurthy and K. Salama, Physica C, 329 (2000) 58.

#### 2:00 PM II4.2

METALORGANIC DEPOSITION OF REBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> FILMS FROM METAL TRIFLUOROACETATE PRECURSORS.

Tetsuji Honjo, Hiroshi Fuji, Yuichi Nakamura, Teruo Izumi, Yuh Shiohara; Superconductivity Research Laboratory, ISTEK, Koto-ku, Tokyo, JAPAN.

Metalorganic deposition(MOD) is expected as one of attractive processes for tape conductor applications because of a non-vacuum process, a rapid deposition rate and compositional controllability. Especially, the MOD process of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> (Y123) using metal trifluoroacetate(TFA) precursors is of great interest for the development of coated conductors since high quality Y123 films with the high  $J_c$  can be fabricated by this process. Actually, this process has been started to apply for coated conductors on metal and realized high  $J_c$ . From the material point of view, the RE-Ba-Cu-O system(RE=Nd or Sm) is one of the most promising materials, since NdBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> (Nd123) and SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> (Sm123) have demonstrated higher  $T_c$  than Y123 as well as stronger vortex-flux pinning, which results in the high irreversibility field of the materials and the high  $J_c$ . However, both REBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> phases have the RE<sub>1-x</sub>Ba<sub>2-x</sub>Cu<sub>3</sub>O<sub>y</sub> (RE123) type solid solution, in which RE substitutions for Ba site considerably suppress  $T_c$ . Therefore, the control of x values in the RE123 phase is a very important factor. In this study, the TFA process was applied to the Nd-system and the dependence of experimental conditions such as starting composition, P(O<sub>2</sub>) and P(H<sub>2</sub>O) for annealing were investigated. As a result, we confirmed that the Nd123 single phase was clearly observed by the XRD in the Nd123 films fabricated by the same processing conditions of Y123. However, the  $T_c$  value of the Nd123 films so produced exhibited lower than that of the Y123 films. The growth mechanism and the effect of processing parameters on superconducting properties in RE123 will be discussed.

This work is supported by the New Energy and Industrial Technology Development Organization(NEDO) as Collaborative Research and Development of Fundamental Technologies for Superconductivity Applications.

#### 2:15 PM II4.3

DEVELOPMENT OF HIGH CRITICAL CURRENT YBCO COATED CONDUCTORS BY PHOTO-ASSISTED MOCVD.

Alex Ignatiev, Yimin Chen, PenChu Chou, Xin Zhang, Xin Chen and Zhongjia Tang, Space Vacuum Epitaxy Center and Texas Center for Superconductivity, University of Houston, Houston, TX.

The fabrication of second-generation YBCO high temperature superconducting wire for electric power applications requires the rapid fabrication of thick atomically-ordered YBCO films on flexible metal substrates with appropriate buffer layers. We have applied photo-assisted MOCVD to the fabrication of YBCO thick films and buffer layers on rolling-assisted biaxially textured Ni-based alloy substrates. Because of its fast growth rate and excellent atomic ordering, photo-assisted MOCVD is a promising method for thick YBCO film processing. YBCO films of ~ 3 micron thick have been deposited on oxide substrates by photo-assisted MOCVD in ~ 5 minutes, and are seen to be atomically ordered and with  $J_c \geq 1$  MA/cm<sup>2</sup>. Several buffer layer systems have also been developed by photo-assisted MOCVD. These include both the YSZ/CeO<sub>2</sub> and the YSZ/(Ce,Sm)O<sub>2</sub> systems. Of these buffer layers (Ce,Sm)O<sub>2</sub> is new, and has been found to be crack-free on nickel even above 1 micron thicknesses. All these epitaxial layers exhibited crystal orientation and in-plane alignment similar to that of the atomically textured Ni-based alloy substrates. YBCO growth on the buffer layer systems on nickel-based alloy substrates has showed promising results with  $J_c \sim 6 \times 10^5$  A/cm<sup>2</sup>.

#### 2:30 PM II4.4

CONVERSION KINETICS OF OXYFLUORIDE DERIVED YBCO FILMS. Igor Seleznev, Mani Gopal and Michael Cima, Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA.

YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> (YBCO) thin films were prepared on lanthanum aluminate (LAO) single crystals and buffered (5000 Y<sub>2</sub>O<sub>3</sub>, 500 CeO<sub>2</sub>) Ni-single crystal substrates by metal organic deposition (MOD) process. Glassy oxyfluoride films were converted to epitaxial YBCO films, by heating in wet atmosphere of N<sub>2</sub>/O<sub>2</sub> mixture. A novel technique was used to determine the rate of conversion of films from glassy state to crystalline YBCO films. In this approach, the concentration of residual fluoride in the partially converted films was examined using fluoride ion selective electrode. Results obtained by this method were compared to results obtained by such methods as X-ray analysis and in-situ resistivity measurements. Analysis of data obtained by different methods showed that fluoride concentration measurement method is a fast and accurate method. The paper will also discuss the influence of different factors on the conversion rate of the oxyfluoride films, including high and low vapor pressure of water, temperature and substrate material. A simple model will be presented that will describe the growth of crystalline YBCO film for diffusion controlled and reaction controlled growth kinetics. Results obtained by in situ resistivity measurements will be compared to results predicted by the model. The obtained data fits the model that describes linear growth kinetics.

### 3:15 PM \*II4.5

DEVELOPMENT OF NEXT GENERATION HTS CONDUCTORS. M.W. Rupich, Q. Li, S. Annavarapu, C. Thieme, W. Zhang, S. Cui, S. Lu, E. Siegal, J. Lynch, S. Hancock, E. Thompson, American Superconductor, Westborough, MA.

The first generation HTS conductor, based on the Bi-2223 oxide-powder-in-tube technology, is currently moving from the laboratory into commercial applications. As a result, R&D efforts are increasingly focused towards development of Second Generation HTS conductors with improved price-performance values that will broaden the commercial market for HTS applications. One approach to the development of the Second Generation HTS conductor is based on the deposition of YBCO films on oxide buffered, deformation-textured metal substrates with the architecture YBCO/CeO<sub>2</sub>/YSZ/RE<sub>2</sub>O<sub>3</sub>/Ni. One particularly attractive path to the manufacture of this YBCO coated-conductor architecture is the low-cost solution-based deposition of both epitaxial oxide buffer layers and the YBCO layer. Previously we have shown the performance of solution-deposited YBCO films is comparable to that of YBCO films deposited by vacuum-based deposition techniques, with critical current densities on oxide buffered, metal substrates approaching 2 MA/cm<sup>2</sup> (77 K and self-field). Recent efforts have focused on extending these deposition processes to longer length samples. In this presentation, we will review progress on the continued development of the Bi-2223 oxide-powder-in-tube conductors, then present improvements in the performance of the solution deposited YBCO films and progress on the development of reel-to-reel processing of the YBCO conductors.

### 3:45 PM II4.6

DEVELOPMENT OF LPE PROCESS FOR FABRICATION OF COATED CONDUCTORS. Teruo Izumi, Natsuro Hobara, Toru Izumi, Katsuya Hasegawa, Masahiko Kai, Hiroshi Fuji, Tetsuji Honjo, Yao Xin, Christian Krauns, Maxim Kuznetsov, Yuichi Nakamura and Yuh Shiohara; Superconductivity Research Laboratory, ISTEC, Koto-ku, Tokyo, JAPAN.

Recent progress of the development of the Liquid Phase Epitaxy (LPE) process for the RE123 coated conductors is reviewed. The LPE is one of the expected processing for fabricating the coated conductor, since it can realize thick film by high growth rate with maintaining high crystallinity. However, the reaction between the solvent and metal substrate has to firstly be prevented, because the solvent for RE123 growth is very reactive with most metals except with silver. Therefore, the buffer layer to prevent the reaction should be prepared between the metal tape substrate and the LPE layer. Several different kinds of oxide materials were tried for the buffer layer. Consequently, MgO and/or NiO were selected because of their relatively high stability for the solvent. However, both materials have some solubility in the solvent and dissolve into the solvent. Then, the new concept to prevent the dissolution was developed. The solvent was saturated by the buffer materials by means of adding them into the solvent before growth. In these cases, the dissolution of buffer materials and the reaction between metal and the solvent were prevented. Then, the LPE 123 layer substituted by the buffer materials was grown from the saturated solvent. This layer revealed lower T<sub>c</sub> than 90K because of its substitution. Therefore, the LPE layer without substitution was coated further on the substituted LPE layer to obtain high superconducting properties such as T<sub>c</sub> and J<sub>c</sub>. Actually, the double layered LPE films was successfully grown on the hastelloy tape. The detail influence of the saturation in the solvent for the LPE growth and superconducting characteristics of the final structure after annealing for oxydization will also be discussed. This work is supported by the New Energy and Industrial Technology

Development Organization (NEDO) as Collaborative Research and Development of Fundamental Technologies for Superconductivity Applications.

### 4:00 PM II4.7

MAGNETIC FIELD DEPENDENCE OF CRITICAL CURRENT DENSITY OF MOD-TFA AND LPE-PROCESSED YBCO COATED CONDUCTORS. Yutaka Yamada, Seokbeom Kim, Takeshi Araki, Yasuo Takahashi, Toshihiro Suga, Toshihiko Maeda, Yasuji Yamada, Izumi Hirabayashi, ISTEC-SRL, Nagoya, JAPAN; Hisashi Yoshino, Mutsuki Yamazaki, Truon Dinh Thanh, Toshiba R&D Center, Kawasaki, JAPAN; Tomonori Watanabe, Kaname Matsumoto, The Furukawa Electric Co., Ltd., Nikko, JAPAN.

It is well known that critical current densities, J<sub>c</sub>, of MOD (Metal Organic Deposition)-TFA (Trifluoroacetate) and LPE (Liquid Phase Epitaxy) processed YBCO coated conductors were high. Moreover, both samples have other merits for the industrialization. The process of MOD-TFA sample is relatively simple using only heat-treatment without complicated vacuum processes such as physical vapor deposition. LPE process can fabricate thick films over 5 micron, which is difficult by other methods. This will result in high current carrying capacity, which is crucial for the application. We present magnetic field dependence of J<sub>c</sub> for MOD-TFA and LPE-processed YBCO coated conductors and discuss the results combined with their microstructures peculiar to the fabrication processes. Especially, the MOD-TFA sample with high J<sub>c</sub> over 1MA/cm<sup>2</sup> is investigated in detail. From these results, the possibility is also discussed for the high magnetic-field applications.

### 4:15 PM II4.8

PHASE EQUILIBRIA IN THE SYSTEM BaF<sub>2</sub>-BaO-Y<sub>2</sub>O<sub>3</sub>-CuO-H<sub>2</sub>O. Lawrence Cook, Winnie Wong-Ng, Joseph Ritter, and Ralph Klein, Ceramics Division, NIST, Gaithersburg, MD.

The barium fluoride process for the production of YBCO superconducting films depends upon the reaction: 2 BaF<sub>2</sub> 1/2 Y<sub>2</sub>O<sub>3</sub> 3 CuO 2 H<sub>2</sub>O = YBa<sub>2</sub>Cu<sub>3</sub>O<sub>6.5</sub> 4 HF. In this process the reactants are deposited as an amorphous film and water vapor is then introduced at high temperature, resulting in formation of the YBCO 123 superconductor. This process is kinetically limited by the rate of removal of HF on the product side. The partial pressure of HF must be kept to a low value in order for the reaction to proceed. Because defluorination results in a substantial mass loss, it is ideally suited to study by thermogravimetric analysis. The thermodynamics of this reaction and relevant phase equilibria will be discussed, including data on fluorine entry into YBCO eutectic liquids, and the effect of water pressure on the stability of the 123 superconductor. Also, our recent calorimetric studies will be discussed, as appropriate.

### 4:30 PM II4.9

GROWTH MECHANISM OF Re-Ba-Cu-O CRYSTAL BY LIQUID PHASE EPITAXY PROCESS. Katsumi Nomura, Saburo Hoshi, Xin Yao, Yuichi Nakamura, Teruo Izumi, Yuh Shiohara, Superconductivity Research Lab, ISTEC, Tokyo, JAPAN.

Liquid phase epitaxy (LPE) has been expected to prepare high quality and large single crystalline films, and has been applied to grow the REBa<sub>2</sub>Cu<sub>3</sub>O<sub>y</sub> (RE=Y, Nd, Sm; REBCO) crystal films on the MgO single crystalline substrates. It was interesting that the pseudo-single crystalline REBCO films can be grown even from the polycrystalline seed films with c-axis orientation (without in-plane alignment). It was also interesting that the preferential direction of in-plane orientation in the LPE crystalline film depends on kinds of REs for the REBCO system. In this paper, we have investigated the growth mechanism of the LPE crystalline films and the difference due to the kind of RE for the REBCO systems. The c-axis oriented polycrystalline seed films of both YBCO and NdBCO were prepared by the vapor deposition method on the MgO substrates. The REBCO LPE films were grown by making a contact of the seed film with the RE supersaturated Ba-Cu-O solution. The microstructures of the films were observed by optical microscopy and atomic force microscopy. The crystal orientation of the films was analyzed by X-ray diffraction. It was found that the crystallinity with in-plane alignment was improved due to the preferential dissolution/growth of the seed islands during the LPE growth, and that the difference of the preferential growth orientations between the YBCO and the NdBCO systems were realized. This phenomenon of the preferential dissolution/growth from the seed to the LPE film could be explained by the coarsening mechanism, which was considered by not only the Gibbs-Thomson effect but also the undercooling due to the difference in the bonding energies between the REBCO grains and the MgO substrate. This work was supported by the New Energy and Industrial Technology Development Organization (NEDO) as Collaborative Research and Development of Fundamental Technologies for Superconductivity Applications.

**4:45 PM II4.10**

SYNTHESIS OF SINGLE CRYSTALLINE  $\text{Nd}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-y}$  THIN FILM BY TRI-PHASE EPITAXY. Kyung Sung Yun, Jeong Hwan Song, Byeong Dae Choi, Yuji Matsumoto, Takanori Itoh, Tokyo Institute of Technology, Toyohiro Chikyow, COMET-NRIM, Ibaraki, JAPAN, Masashi Kawasaki, Hideomi Koinuma, Tokyo Institute of Technology, Yokohama, JAPAN.

We report a novel method of depositing  $\text{Nd}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-y}$  thin films satisfying both requirements of single crystal quality as well as atomic scale surface smoothness in the fabrication of high  $T_c$  superconducting devices. This new approach involves the solid thin film growth on appropriate substrate via gas-liquid phase epitaxy within a vacuum chamber.

This method combines film growth from a liquid phase and pulsed laser deposition. A liquid phase film (near-eutectic BaO-CuO mixture) is intentionally formed on an ultrathin  $\text{Nd}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-y}$  seed layer prior to the growth of the rest of the film by PLD. The precursors i.e. the laser ablated Nd, Ba, Cu and their oxides dissolve into a liquid BaO-CuO layer on the film/substrate surface and penetrate through it reaching the liquid-solid interface where condensation into the solid phase occurs. Thermodynamic equilibrium can be maintained at the liquid-solid film boundary while preserving the benefit of intrinsic stoichiometry control offered by the PLD technique. Since all three phases, gas, liquid, and solid, are involved in this form of film growth, we have termed it "Tri-Phase Epitaxy" (TPE). Microstructural characterization by cross-sectional transmission electron microscopy (TEM) performed on several TPE films reveal high epitaxial quality of these films without any grain boundaries over the widest observable area available in the TEM machine. The  $\text{Nd}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-y}$  TPE films fabricated on  $\text{SrTiO}_3$  substrates shows the same structure as bulk single crystals over a large area.

SESSION II5: CHARACTERIZATION OF COATED CONDUCTORS I – PHASE CHEMISTRY AND MICROSTRUCTURE

Chairs: Roger Woerdenweber and Kazumasa Togano  
Wednesday Morning, November 29, 2000  
Room 306 (Hynes)

**8:30 AM \*II5.1**

MICROSTRUCTURAL DEVELOPMENT IN  $\text{YBa}_2\text{Cu}_3\text{O}_y$  COATED CONDUCTORS BASED ON ION-BEAM-ASSISTED DEPOSITION (IBAD) OF YSZ BUFFER LAYERS. Terry Holesinger, Steve Foltyn, Paul Arendt, Quanxi Jia, Harriet Kung, Jim Smith, James Groves, Paul Dowden, Ray DePaula and E.J. Peterson, Los Alamos National Laboratory, Los Alamos, NM.

A detailed summary of the relevant microstructural features of Y-123 coated conductors fabricated with the IBAD YSZ process on nickel alloy substrates with or without additional buffer layers is presented. Each process used to build the bi-axially textured template or deposit the superconductor creates an interface along which defects or interfacial reactions may result. These defects can be additive and propagate through the entire film structure to affect the growth and properties of the superconducting film. Each interface in these samples has been carefully analyzed and the observed defects correlated with the transport properties. Modifications have been made to the overall structure of these coated conductors based on the microstructural work. It will be shown how these changes have minimized defect generation and optimized the performance of these coated conductors.

**9:00 AM \*II5.2**

$\text{YBa}_2\text{Cu}_3\text{O}_7$  GROWTH KINETICS AND MICROSTRUCTURES IN  $\text{BaF}_2$  PROCESSES. M. Suenaga, Division of Materials and Chemical Sciences, Brookhaven National Laboratory, Upton, NY.

The so-called  $\text{BaF}_2$  process is one of the major candidates for commercially viable processes in the fabrication of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  coated conductors for high-current applications. For this reason, significant research and development effort is being expended on this process. One of the key factors for successful application of this process for conductor production is the control of the c-axis texture of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  crystals on single-crystalline or textured substrates. Here, a review of the kinetics of the nucleation and the growth of the YBCO layers in this process is presented. Since two methods of preparing the precursor films, sol gel and physical vapor deposition, are used extensively in this process, the nucleation and growth kinetics are compared for the precursors which were derived from these different methods. In addition, the influence of the substrate materials for the formation of the c-axis textured YBCO layers is also discussed as a number of different substrate materials are being used for the development of the conductors.

This work was performed under auspices of U.S. Department of

Energy, Office of Science, Division of Materials Sciences and Office of Energy Efficiency and Renewable Energy, under contract DE-AC02-98CH10886.

**9:30 AM II5.3**

MANIPULATING THE NUCLEATION AND GROWTH OF  $\text{REBa}_2\text{Cu}_3\text{O}_7$ . Guus Rijnders, Dave H.A. Blank, Horst Rogalla, Low Temperature Division, MESA Research Institute, University of Twente, Enschede, THE NETHERLANDS and Sara Bals, Gustaf van Tendeloo, EMAT-RUCA, Antwerp, BELGIUM.

The nucleation and growth of the cuprate high  $T_c$  materials, especially the  $\text{REBa}_2\text{Cu}_3\text{O}_7$ -d compounds, have been subject to many studies in the last ten years. In the case of deposition techniques such as pulsed laser deposition (PLD) or sputter deposition mainly the growth mode was studied. Depending on the growth conditions, a multiple level 2D or spiral growth has been observed and the surface of the film is roughened. This will limit applications based on multi-layer technology. Starting with an atomically smooth substrate, i.e.,  $\text{TiO}_2$  terminated strontium-titanate, we studied the growth of  $\text{REBa}_2\text{Cu}_3\text{O}_7$ -d using high-pressure RHEED. With standard PLD conditions, the RHEED intensity drops significantly during the initial growth and nucleation of sub-unitcell blocks is observed by AFM and TEM. As a consequence, defects such as anti-phase-boundaries, are present in the film. In this study, growth manipulation, using pulsed laser interval deposition, is used to control and modify the nucleation and growth and, therefore, the surface morphology of the as-grown film. Here, the perovskite stacking sequence at the interface with the substrate and its impact on the film growth will be discussed.

**10:15 AM \*II5.4**

PHASE EQUILIBRIA OF THE Ba-Nd-Cu-O AND Ba-Nd-Yb-Cu-O SYSTEMS. Winnie Wong-Ng, Lawrence Cook, Brian Toby, MS&E Laboratory, NIST, Gaithersburg, MD; Julia Suh, Jeremy Dillingham and Rachel Coutts, University of Maryland, Geology Department, College Park, MD; James Kaduk, BP-Amoco Research Center, Naperville, IL; Terry Holesinger and Dean Peterson, LANL, Los Alamos, NM.

In response to the need for improved Ba-Nd-Cu-O phase equilibrium data for processing of second generation RABiTS and IBAD coated conductor tapes, phase diagrams of this system under air and reduced oxygen partial pressure (0.1%  $\text{O}_2$ ) were developed. A special experimental procedure was used for preparing BaO and for the handling and heat-treatment of samples. A discussion of the crystal chemistry and phase relationships of phases found in these systems will be presented. A comparison of the diagrams prepared under different atmospheric conditions will be summarized. By mixing a small lanthanide ion such as Yb with  $\text{Ba}_{2-x}\text{Nd}_x\text{Cu}_3\text{O}_6$  (Nd-123), both flux-pinning and melting properties of the resulting high  $T_c$  phases can be tailored and optimized. The solid solution range of  $\text{Ba}_{2-x}(\text{Nd,Yb})_x\text{Cu}_3\text{O}_6$  under 0.1%  $\text{O}_2$ , 2%  $\text{O}_2$  and air was determined using x-ray diffraction, and the superconducting properties were obtained using magnetic susceptibility measurements. Crystal chemistry of this solid solution was studied using x-ray, electron, and neutron diffraction. Results of the phase equilibria and phase characterization will be presented.

**10:45 AM \*II5.5**

CHARACTERIZATION OF COATED CONDUCTOR PHASE PURITY, OXYGEN STOICHIOMETRY, DEFECT STRUCTURES, AND IN-PLANE TEXTURE BY SPECTROSCOPIC AND SYNCHROTRON X-RAY METHODS. V.A. Maroni, A.K. Fischer, A.J. Kropf, H. You, Y. Jee, Argonne National Laboratory, Argonne, IL; K.T. Wu, State University of New York, Old Westbury, NY; M.W. Rupich, Q. Li, C. Thieme, W. Zhang, American Superconductor, Westborough, MA.

Spectroscopic and synchrotron x-ray methods are being used to interrogate the phase chemistry, oxygen stoichiometry, nature of defect structures, and quality of in-plane texture for YBCO-type coated conductor samples on single crystal and metallic substrates. Raman microspectroscopy and imaging Raman microscopy provide information about the YBCO film that relates to orthorhombic/tetragonal structure type, oxygen ordering, out-of-plane puckering, film morphology, and the presence (and location) of second phases, such as copper oxide, barium cuprate, and 211. Attenuated total reflectance mode infrared spectroscopy is especially useful for detecting carbonaceous impurity phases (including carbonate) and for monitoring the decomposition of carbon-based YBCO precursors. X-ray reflectivity and diffraction measurements (made using facilities and equipment now available to us at the Advanced Photon Source) allow the determination of precise values for the thickness and in-plane alignment of superconducting films, buffer layers, and textured substrates. We are able to correlate many of the measured parameters and microstructures with substrate features, precursor type, and film deposition method. An overview of results will be

discussed along with possible pathways to online monitoring methods for coated conductor manufacturing facilities.

Work at Argonne National Laboratory was sponsored by the U.S. Department of Energy, Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, under Contract W-31-109-ENG-38.

#### 11:15 AM II5.6

TEXTURE ANALYSIS OF COATED CONDUCTORS BY  $\mu$  RAMAN AND SYNCHROTRON X-RAY DIFFRACTION. T. Puig, X. Obradors, N. Mestres, F. Alsina, Institut de Ciència de Materials de Barcelona, SPAIN; A. Puig-Molina, H. Graafsma, European Synchrotron Radiation Facility, Grenoble, FRANCE; A. Usoskin, F. Garcia-Moreno, H.C. Freyhardt, Zentrum fuer Funktion Werkstoffe, Goettingen, GERMANY.

Two novel complementary techniques for texture analysis of YBCO coated conductors are presented. Conventional x-ray diffraction is the most used study for ex-situ texture determination of these materials, however the complicated epitaxial growth of coated conductors with the possible presence of undesired a-crystals and in-plane large angle grain boundaries may benefit from an additional complementary analysis. The non-destructive method of  $\mu$ -Raman spectroscopy enables an easy analysis of the film homogeneity by determining the distribution of a- and c-oriented grains within the tape with a 1  $\mu$ m spatial resolution and acquisition times of 5 min/spot. On the other hand, synchrotron x-ray diffraction analysis in transmission geometry and 2D detectors enables, with acquisition times of 100 ms, simultaneous observation of a- and c- crystallites and in-plane textures of substrates, buffers and superconducting layer without sample rotation. Thus, opening the field of this technique to in-situ analysis of epitaxial growth of coated conductors prepared by ex-situ processes (solution methods, sol gel, MOD, BaF<sub>2</sub> evaporation,...). Results from these two techniques for stainless steel tapes buffered with IBAD-YSZ and coated with PLD-YBCO are analyzed and compared with conventional x-ray diffraction studies. The potential benefits of combining the different methods are presented.

#### 11:30 AM II5.7

STRUCTURE-TRANSPORT CORRELATIONS IN PHOTODOPED YBCO CRYSTALS AND FILMS. T.A. Tyson, L. Dieng and C. Cui, NJIT, Newark, NJ.

Polarized x-ray absorption measurements have been used to determine the local structural changes in oriented crystals and thin films of YBCO accompanying photodoping. The photoresponse as a function of oxygen content and light frequency was examined. Changes in the local structure and transport with photodoping are compared to the local structural and transport changes found in high pressure measurement.

This research was supported by NSF Grant DMR-9733862.

### SESSION II6: CHARACTERIZATION OF COATED CONDUCTORS II - MICROWAVE, JUNCTION, AND GRAIN BOUNDARY

Chairs: Terry G. Holesinger and Juergen Dzick  
Wednesday Afternoon, November 29, 2000  
Room 306 (Hynes)

#### 1:30 PM II6.1

IMPACT OF MICROSCOPIC DEFECTS ON MECHANICAL AND MICROWAVE PROPERTIES OF YBCO THIN FILMS.

Roger Wördenweber, Jan Einfeld, Peter Lahl, Forschungszentrum Jülich, ISI, GERMANY.

In this contribution we demonstrate, that microscopic defects HTS films can be created by sputtering technique, which lead to a considerable improvement of the mechanical and microwave properties of the films. The improvement of the mechanical and microwave properties are explained in terms of heteroepitaxial growth models and the two-fluid model, respectively. YBCO films with different size and densities of Y<sub>2</sub>O<sub>3</sub> precipitates are grown on LaAlO<sub>3</sub> and sapphire by variation of the energy of the ions during sputter deposition. The temperature dependence of the microwave surface resistance R<sub>s</sub> does not depend on the type of substrate material but only on the density of the defects. Films with low density of defects show a characteristic shoulder in the R<sub>s</sub>(T) curve which shifts to higher temperature and decreases in size with increasing density of defects. Temperature dependence and reduction of R<sub>s</sub> with increasing density of defects are explained in terms of the two-fluid model with thermally excited quasiparticles characterised by a Drude-shaped conductivity spectrum. Values for the scattering rates can be derived from the measurements of the surface resistance, which agree with the classical Matthiesen rule and are comparable to results obtained from other experiments. The impurity scattering rate increases with increasing

defect density. Finally, the experimental data and the theoretical model demonstrate, that the surface resistance can be reduced by up to a factor of two over a wide temperature range. The reduction of the surface resistance is accompanied by an improvement of the mechanical properties of the HTS thin films which leads to an increased critical film thickness. Both properties, namely the increase of the critical thickness and the reduction of the microwave surface resistance, demonstrate the potential of microscopic defects for improvement of HTS films for applications.

#### 1:45 PM II6.2

EFFECT OF YBCO FILM CHARACTERISTICS ON RF PROPERTIES OF MSL RESONATORS WITH THESE FILMS. Kazunori Yamanaka, Akihiko Akasegawa, Teru Nakanishi, Fujitsu Laboratories Ltd., Atsugi, Kanagawa, JAPAN.

Oxide-superconducting films such as YBCO have received much attention due to their application potentials in high-Q RF devices including resonators and filters. In these devices, the unloaded-Q (Q<sub>u</sub>) would depend on the RF properties of the thin-films that are mainly governed by the weak-links of the films. Generally, high quality films (well textured with strong-link grains) were reported to be effective in high-Q<sub>u</sub> devices. However, so far the relationship between film characteristics and weak-links is not well-understood in actual devices, irrespective of device geometry. In this study, the effect of the film characteristics (prepared with different parameters) on the weak-links was investigated. Here, resonators were fabricated with YBCO films with a film-thickness in the range of 200 - 1000 nm. These films were characterized for microstructures and crystallinity. The RF properties of the resonators were measured at frequency of about 2 GHz. From the experimental results it was understood that the films with Q<sub>u</sub> value of 1E4 - 5E4 at 70 K exhibited strong C-axis orientation, Y211 segregation and porous structures. On the other hand, the films with high-Q<sub>u</sub> values (5E4 - 8E4) had much stronger c-axis orientation and twin boundaries. From comparative studies on these two types of films, it was understood that the films with Y211 segregation had more disorientated Y123 grains. It was also found that in similar type of films the influence of the weak-links decrease with increasing the film thickness. Moreover, it was observed that high-Q<sub>u</sub> films possessed fewer weak-links compared with low-Q<sub>u</sub> films irrespective of the film thickness, suggesting that the existing pores and Y123 disorientation in low-Q<sub>u</sub> films result weak-links. Also, twin boundaries are considered one of the weak-link origins in high-Q<sub>u</sub> films.

#### 2:00 PM II6.3

THE FINE-SCALE MICROSTRUCTURE OF YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>  INTERFACE-ENGINEERED JUNCTIONS. Marica Gustafsson, Dept. of Experimental Physics, Chalmers University of Technology, Göteborg, SWEDEN; Gunnar Svensson, Dept. of Structural Chemistry, Stockholm University, Stockholm, SWEDEN; Brian Moeckly, Conductus Inc., Sunnyvale, CA; Eva Olsson, Analytical Materials Physics, Angström Laboratory, Uppsala University, Uppsala, SWEDEN.

The performance of Josephson junctions is partially determined by their fine-scale microstructure. In this work, the microstructure of the barrier layer in interface-engineered ramp-edge and planar Josephson junctions (IEJs) has been studied. One advantage of the interface-engineered junctions is that the problems of epitaxial growth and introduction of interface defects on an inclined surface are avoided compared to junctions made by depositing a separate layer of a different phase. The IEJs are fabricated by an annealing, a plasma treatment and a second annealing of an ion milled YBCO ramp edge or planar surface followed by a deposition of the top YBCO layer. The microstructure of both ramp edge and planar IEJs has been characterized using high-resolution transmission electron microscopy (HREM) in combination with energy dispersive X-ray (EDX) analysis and electron energy loss spectroscopy (EELS). The planar junctions were made in order to allow the junctions to be studied using different incident angles of the incoming electron beam. The ramp edge junction geometry did not allow this. Two different types of barriers were investigated for which the difference between them was the thickness of the barrier layer. The thickness of the barrier layer varied between 4.0-6.0 nm and 1.7-3.5 nm for the specimens fabricated with parameters corresponding to high R<sub>n</sub> and low R<sub>n</sub> junctions. HREM in combination with EDS, EELS and convergent beam electron diffraction indicated a modified barrier structure, which is believed to be Y<sub>2</sub>O<sub>3</sub>, with the orientation relations [110]<sub>Y2O3</sub> \ \ [100]<sub>YBCO</sub> and [001]<sub>Y2O3</sub> \ \ [001]<sub>Y2O3</sub>.

#### 2:15 PM II6.4

EPITAXIAL GROWTH OF YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> FILM ON SINGLE DOMAIN YBCO SUBSTRATE FOR RF COMPONENT DEVELOPMENT. Donglu Shi, Yongli Xu and Shaun McClellan, Dept of Materials Science and Engineering, University of Cincinnati, Cincinnati, OH; Altan Ferendeci, Dept of Electrical and Computer Engineering, University of Cincinnati, Cincinnati, OH.

In our previous experiments we have developed a high quality single domain YBCO for rf component development. The material exhibits excellent physical properties including extremely sharp T<sub>c</sub>'s, well-controlled crystal orientation, and low surface resistance, R<sub>s</sub>. However, we believe that R<sub>s</sub> can be further reduced if the surface of the single domain can be modified. As the major rf losses may arise from the dissipation at the 211 particles, we have developed a new method to cover the 211 particles by depositing a YBCO thin film on the polished single domain surface. In this method, the single domain YBCO serves as a substrate that has a perfect lattice matching with the YBCO film. We will report the experimental results of film synthesis, physical property characterization, and microwave behavior.

### 2:30 PM II6.5

**SUPERCONDUCTING CARRIER DENSITY IN ULTRATHIN YBCO FILMS.** Lyuba Delimova, Igor Grekhov, Ivan Liniichuk, Ivan Veselovsky, Ioffe Physical Technical Inst, Dept of Solid State Electronics, St. Petersburg, RUSSIA.

The study of ultrathin (<10nm) YBaCuO films is of great interest due to its possible application in such promising devices as field effect transistors, sensitive bolometers, etc. This paper considers the transition of a laser ablated ultrathin YBaCuO film from the superconducting to resistive state due to the temperature or transport current increase. Films of 8nm thickness were deposited on both a SrTiO<sub>3</sub> substrate (YBCO/STO) and a YBa<sub>2</sub>Cu<sub>3-x</sub>Nb<sub>x</sub>O<sub>7</sub> buffer (YBCO/buffer/STO) which we have found to raise the critical temperature of ultrathin YBaCuO films. The Kosterlitz-Thouless model was used to analyze the measurements, since Y. Matsuda *et al.* [Phys.Rev.B48,10498,(1993)] have shown that the transition in ultrathin YBaCuO films can be regarded as a 2D-fluctuation dominated transition involving vortex-string pairs. Both types of film manifested a power law for current-voltage characteristics, and the  $\alpha$ -power was shown to decrease linearly with a subsequent drop to unit at higher temperatures, in agreement with the model. The model parameters were found from the temperature dependence  $\alpha(T)$  to be  $T_{kT} = 75K$ ,  $T_{c0} = 79K$  for YBCO/buffer/STO, and  $T_{kT} = 39K$ ,  $T_{c0} = 66K$  for YBCO/STO and were used to calculate the temperature dependence of the resistance, which is consistent with the measured resistive transition. The superfluid density  $n_s$  at  $T_{kT}$  was found from the  $T_{kT}$  value for YBCO/STO to be 2% of the total carrier density in the film and showed an anomalously slow rise just below  $T_{kT}$ , an order of magnitude lower than that in single crystals. In contrast, the superfluid density at  $T_{kT}$  for YBaCuO deposited on the buffer was 3% of the total density in the film and increased, just below  $T_{kT}$ , only by a factor of three slower than  $n_s$  in single crystals. The possible reason for the anomalous increase of  $n_s$  in ultrathin YBaCuO films is discussed.

The work was supported by RMS Grant \$30696.

### 3:15 PM II6.6

**MICROWAVE INTERMODULATION DISTORTION IN BICRYSTAL YBCO GRAIN BOUNDARY JUNCTIONS.** H. Xin<sup>1,2,3</sup>, D.E. Oates<sup>1,2</sup>, G. Dresselhaus<sup>2,3</sup> and M.S. Dresselhaus<sup>2</sup>. <sup>1</sup>MIT, Dept of Physics/Lincoln Lab, Cambridge, MA. <sup>2</sup>MIT, Cambridge, MA. <sup>3</sup>AFRL, Hanscom AFB, MA.

Measurements of the microwave intermodulation distortion (IMD) in engineered bicrystal YBCO grain-boundary junctions are presented. The IMD measurement is a very sensitive probe to study the origin of the nonlinear microwave properties of high-temperature superconducting thin films. The two-tone IMD at 4.4 GHz of thin YBCO films and films with engineered bicrystal grain-boundary junctions of misorientation angles from 2 to 24 degrees was measured as a function of microwave power and temperature in a suspended microstrip resonator configuration. The IMD of the films with grain boundary junctions increases as the misorientation angle increases. The long Josephson junction effects in the high-angle grain boundaries were studied. Differences in experimental results of high and low misorientation angles are discussed. The correlation between the microwave impedance of the YBCO grain boundaries and the intermodulation distortion were investigated. Moedling of the IMD including the long-junction effects of Josephson vortices will be presented. This work was supported by the Air Force Office of Scientific Research.

### 3:30 PM II6.7

**SUPERCONDUCTING AND MICROSTRUCTURAL PROPERTIES OF [001] TILT YBCO THICK FILM GRAIN BOUNDARIES ON STO BICRYSTAL AND TEXTURED Ni-SUBSTRATE.** Qiang Li, V.F. Solovoyov, H.J. Wiesmann, Y. Zhu and M. Suenaga, Brookhaven National Laboratory, Upton, NY.

A study is presented of superconducting and microstructural properties of [001] tilt YBCO thick film grain boundaries on SrTiO<sub>3</sub>

bicrystal substrates and textured Ni substrates, prepared using BaF<sub>2</sub> ex-situ post reaction process. The misorientation angles of the grain boundaries ranged from 0 to 24 degree. In addition, columnar defects were introduced by heavy ion-irradiation along the common c-axis in some of the bicrystal films to enhance the flux pinning. 2-18 micrometer wide bridge containing a single [001] tilt grain boundary can be isolated by either photolithographic or laser patterning. An electric configuration, with up to 5 voltage pairs located along the grain boundaries, was used to measure the resistance and the voltage-current (V-I) characteristics of the grain boundary in broad H-T plane. The microstructure of the grain boundaries at the patterned bridge was examined using advanced TEM. We shall discuss the relation between the transport properties and the exact local microstructure of both unirradiated and irradiated grain boundaries. This work was supported by the U.S. Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under contract No. DE-AC02-98CH10886.

### 3:45 PM II6.8

**COMPARISONS OF MICROSTRUCTURE IN PURE AND Ca-DOPED YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>  EPITAXIAL THIN-FILM BICRYSTAL GRAIN BOUNDARIES.** Xueyan Song, G.A. Daniels, J.L. Reeves, D.C. Larbalestier and S.E. Babcock, MS&E and Applied Superconductivity Center, University of Wisconsin-Madison, WI.

Ca substitution for Y has been shown to improve the superconducting properties of epitaxial thin film bicrystals of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>  for the case of both low-angle and high-angle [001] tilt boundaries. For this work pulsed laser deposition was used to grow ~250 nm thick Y<sub>0.7</sub>Ca<sub>0.3</sub>Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>  and of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>  epitaxial films on 5° and 24° SrTiO<sub>3</sub> bicrystals. The superconducting properties of the bicrystal improved in the Ca-doped samples relative to the undoped sample in each case. TEM samples that show the magnitude and wavelength of the meander of the (YCa)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>  over very long lengths of the boundary (1 mm in one case) have been prepared and will be studied in detail. The microstructure of both 24° and 5° boundaries will be studied in detail and compared with a view toward understanding the change in superconducting properties. Furthermore, microstructural differences in the single crystal part of the film already have been identified. For example, the density of a-axis grains is increased in Ca-doped samples grown under the same deposition conditions, and their morphology differs from that in the pure YBCO film.

### 4:00 PM II6.9

**THE ORIGIN OF REDUCED CRITICAL CURRENTS AT GRAIN BOUNDARIES IN YBCO.** M. Kim<sup>1,2</sup>, G. Duscher<sup>1,3</sup>, N.D. Browning<sup>2</sup>, K. Sohlberg<sup>1</sup>, S.T. Pantelides<sup>1,3</sup>, S.J. Pennycook<sup>1,3</sup>. <sup>1</sup>Oak Ridge National Lab, Solid State Division, Oak Ridge, TN. <sup>2</sup>University of Illinois at Chicago, Dept of Physics, Chicago, IL. <sup>3</sup>Vanderbilt University, Dept of Physics and Astronomy, Nashville, TN.

A combination of atomic-resolution Z-contrast microscopy, electron energy loss spectroscopy and first-principles theory has been used to investigate the origins of electrical activity at grain boundaries in the model perovskite SrTiO<sub>3</sub> as well as in YBCO thin films. We show that grain boundaries are intrinsically non-stoichiometric. Total-energy calculations reveal that the introduction of nonstoichiometry into the grain boundaries is energetically favorable and results in structures that are consistent with atomic-resolution Z-contrast micrographs. Electron energy-loss spectra provide direct evidence of oxygen deficiency. These results and calculations for model non-stoichiometric grain boundaries provide a consistent microscopic explanation for the band bending that has been proposed to explain the low critical currents across grain boundaries in the high-temperature superconductors. It further explains the exponential reduction in critical currents with grain boundary misorientation, and suggests means for correcting the problem.

### 4:15 PM II6.10

**NICKEL GRAIN BOUNDARY INDUCED CURRENT BARRIERS IN YBCO COATED CONDUCTORS.** J.L. Reeves, D.M. Feldmann, S.E. Babcock, D.C. Larbalestier, University of Wisconsin-Madison, Applied Superconductivity Center, Madison, WI; T.L. Peterson, Air Force Research Laboratory, Wright-Patterson Air Force Base, OH; A. Goyal, Oak Ridge National Laboratory, TN.

Current-limiting defects in coated conductors were investigated using focused ion beam cross-sectioning, scanning electron microscopy, backscattered electron Kikuchi pattern analysis, and magneto-optical imaging. These combined techniques reveal current barriers in the YBCO above grain boundaries in the biaxially textured nickel substrate that influence the connectivity of the YBCO, even in coated conductors with high critical current density (J<sub>c</sub>) and processed by different methods (PLD and BaF<sub>2</sub>). The focused ion beam was used to produce cross-sections at specific grain boundary defects visible on the YBCO surface to provide direct evidence that the boundaries seen

in the YBCO layer are copied from the nickel substrate below. Current barriers appear in the magneto-optic image above all nickel boundaries with misorientation angles greater than 4 degrees as determined by backscattered electron Kikuchi pattern analysis. These results suggest that the properties of the grain boundaries in the YBCO layer that are inherited from the nickel substrate are influenced by the sample history and can be improved through process optimization.

#### SESSION II7: CURRENT FLOW AND FLUX PINNING IN YBCO

Chairs: Robert H. Hammond and Donglu Shi  
Thursday Morning, November 30, 2000  
Room 306 (Hynes)

##### 8:30 AM \*II7.1

MAGNETO-OPTICAL STUDY OF GRAIN BOUNDARIES, INTERFACES AND GRAIN BOUNDARY NETWORKS IN YBaCuO. Ch. Jooss, K. Thiele, B. Bringmann, M.P. Delamare, K. Guth, L.-O. Kautschor, S. Sievers, J. Hoffmann, H.C. Freyhardt\*, Institut für Materialphysik, University of Göttingen, Göttingen, GERMANY; H. Walter, J. Dzick \*Zentrum für Funktionswerkstoffe, Göttingen, GERMANY; F. Sandiumenge, Institut de Ciencia de Materials, Campus de la Universitat Autònoma de Barcelona, Bellaterra, SPAIN; B. de Boer and B. Holtzapfel, IFW Dresden, Institute of Metallic Materials, Dresden, GERMANY.

Using quantitative magneto-optics and inversion of Biot-Savart's law for determining current density distributions, we investigated the local current carrying capability of various types of grain boundaries and interfaces in YBaCuO thin films and bulk material: (i) Grain boundaries formed by multi-top seeding melt-textured growth, (ii) interfaces which are formed by bonding of monoliths with and without chemical additions, (iii) thin film bicrystals on Ni and SrTiO<sub>3</sub> substrates, (iv) Ca-doped YBaCuO thin film grain boundaries, and (v) networks of grain boundaries in RABiTS and IBAD coated conductors. We investigate the dependence of the local critical current density on the local magnetic flux density depending on the magnetic history of the bicrystals and grain boundary networks in coated conductors. By comparing systematically microstructure and local current density of differently processed grain boundaries, the current limiting properties are analyzed which are specific for differently processed grain boundaries, such as superconducting coupling, flux pinning, self-field effects, and normal phases.

##### 9:00 AM II7.2

CHARACTERIZATION OF CURRENT FLOW IN HIGH-J<sub>c</sub> YBCO COATED CONDUCTORS USING RABiTS. D.M. Feldmann, J.L. Reeves, A.A. Polyanskii, S.E. Babcock, D.C. Larbalestier, Univ of Wisconsin, Madison, WI; A. Goyal, R. Feenstra, D.F. Lee, M. Paranthaman, D.M. Kroeger, D.K. Christen, Oak Ridge National Lab, Oak Ridge, TN.

We have isolated small systems of grain boundaries (GBs) on high-J<sub>c</sub> coated conductor tapes using magneto-optical (MO) imaging to identify areas of interest. Our previous work has shown that percolative current flow is induced by many YBCO GBs that have propagated through from the Ni substrate. Specific regions were isolated by laser cutting, and characterized by transport, MO imaging of both magnetization currents and transport currents, and local orientation mapping. Transport studies revealed a spread in J<sub>c</sub> of 0.7-2.7MA/cm<sup>2</sup> (0T,77K) about the value of 2.3MA/cm<sup>2</sup> (0T,77K) for the full width of the tape. This means that current percolates, and combined MO imaging and local orientation mapping demonstrate that Ni GBs are the major source of this percolation. In a previous study, all Ni GBs with an angle greater than 4° were found to initiate percolative current flow, but this threshold may be higher and more diffuse in the present study. Improvement of the local substrate texture or low-angle GB properties should lead to higher J<sub>c</sub> values.

##### 9:15 AM II7.3

PENETRATION BEHAVIOR OF CURRENT INDUCED MAGNETIC FIELDS IN YBCO-THIN FILMS. M. Kuhn, B. Schey, W. Biegel, B. Stritzker; Universitaet Augsburg, Institut fuer Physik, Augsburg, GERMANY; A. Heinrich, K. Numsen, H. Kinder; Technische Universitaet Muenchen, Physik Department E10, Garching, GERMANY.

Flux penetration of external and current induced magnetic fields can be studied by magneto-optics. With a specially designed magneto-optical apparatus it is possible to investigate YBCO-thin films as large as 20 x 20 cm<sup>2</sup>. In the present work we studied the penetration behavior of current induced fields in patterned HTS-thin films of 8 x 0,7 cm<sup>2</sup> dimensions at 77 K. At an applied alternating current the image integration was triggered to distinguish between current

increase and decrease stage as well as the remanent state of the superconductor. A threshold value of the applied current of  $I/I_c \geq 0.3$  for flux penetration could be observed. The influence of the bridge geometry and of inhomogeneities on flux penetration has been investigated. Flux and therefore current distributions change drastically with increasing angle of the YBCO-path both in external as well as current induced fields. A magnetic interaction between adjacent bridges with parallel and antiparallel current directions could be observed, where in the antiparallel configuration the magnetic field is enhanced. The investigations were carried out up to currents of  $I/I_c \approx 3$  finally resulting in a quench of the YBCO-path. This enables us to observe the thermal front spreading out from the quench area. This work was performed within the Joint Project "HTS for Power Engineering" cofunded by the German ministry BMBF.

##### 9:30 AM II7.4

MAGNETIC IMAGING OF SUPERCONDUCTING TAPES TO DETERMINE CURRENT FLOW. G.W. Brown, M.E. Hawley, Materials Science & Technology Division, D.J. Brown, Theoretical Division and F.M. Mueller, Materials Science & Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

Magnetic field data at the surface of a superconductor can be inverted to current density maps that are useful for further development of High T<sub>c</sub> coated conductors. We have developed a magnetic imaging system that uses magnetoresistive read heads from computer hard disk drives to map the transport-current-induced magnetic field at the surface of superconducting tapes. The system operates while immersed in liquid nitrogen. The transport current pathways are determined from the 2-dimensional maps of the surface-normal magnetic field component using established inversion schemes implemented with the data acquisition software on a personal computer. The advantages of such a system are its simplicity and the possibility of high resolution imaging due to the small active areas of these magnetoresistive devices. In this work we examined the transport current flow in pulsed-laser-deposited YBCO films. The films were patterned into bridge structures on single crystal SrTiO<sub>3</sub> substrates and on a textured yttria-stabilized-zirconia layer deposited on an Inconel ribbon by ion beam assisted deposition. In each case, YBCO dimensions were chosen to provide samples with ~10 A critical current (I<sub>c</sub>), based on prior experience. In all samples, for  $I < 6A$ , current was confined along the edges of the bridge, consistent with the Critical State Model (CSM). For high J<sub>c</sub> samples, as the supply current was increased, current began to flow down the center of the tape. As the supply current approached I<sub>c</sub> we observed a roughly constant J across the width of the sample, also consistent with the CSM. In a sample with low J<sub>c</sub>, we observed deviations in the expected current pathways and have been able to correlate some of these with defects observed in optical, atomic force, and scanning electron microscopies.

##### 10:15 AM II7.5

IMPROVED INVERSION TECHNIQUES FOR OBTAINING TRANSPORT CURRENT PATHWAYS FROM MAGNETIC FIELD DATA. F.M. Mueller, D.J. Brown, G.W. Brown and M.E. Hawley, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

Transport current pathway information is useful for improving high temperature superconducting tapes because it can be correlated with data from other characterization techniques to optimize production methods. The current pathways can be determined by inverting magnetic field data measured at the surface of the superconductor, but care must be taken to avoid subtle errors in the inversion process. We have developed improved inversion techniques that allow us to compare the spatial dependence of the current density pathways with a variety of metallurgical defects present in superconducting tapes. In particular, our methods more adequately treat the high wavenumber noise inherent in these inversion processes. We will demonstrate our techniques with magnetic field data from simulated test cases and with data from a magnetic imaging instrument we have recently developed. This improved inversion process should lead to improvement in YBCO coated conductor tapes made at Los Alamos, which are already at the cutting edge in terms of current carrying capacity and length.

##### 10:30 AM II7.6

TAILORING MICROSTRUCTURES UNDER STRONG NON-EQUILIBRIUM CONDITIONS: A FEASIBLE PATH TOWARDS HIGH J<sub>c</sub> IN MELT TEXTURED YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>. F. Sandiumenge, Institut de Ciencia de Materials de Barcelona, CSIC, Campus de la UAB, SPAIN; J. Plain, Institut de Ciencia de Materials de Barcelona, Campus de la UAB, SPAIN, and Laboratoire de Metallurgie Physique, Universite de Poitiers, FRANCE; T. Puig, Institut de Ciencia de Materials de Barcelona, CSIC, Campus de la UAB, SPAIN; J. Rabier, Laboratoire de Metallurgie Physique, Universite de Poitiers, FRANCE; X. Obradors, Institut de Ciencia de Materials de Barcelona, CSIC, Campus de la UAB, SPAIN.

Under high oxygen pressure conditions,  $\text{YBa}_2\text{Cu}_3\text{O}_7$  (123) is brought far from its stability field into a region of the  $p\text{O}_2$  - T phase diagram where 124 is stable against a 123 CuO mixture. Under such non-equilibrium conditions, the nucleation of 124-like stacking faults results from a subtle interplay of plastic deformation and phase instability. Motivated by the high potential of this mechanism to increase the density of dislocations acting as flux pinning centers ( $J_c$  is enhanced by 150%, up to  $\sim 2.8 \times 10^9$  A/cm<sup>2</sup> at 5K, H parallel to c), in this report we explore the evolution of the microstructure under a range of thermodynamic conditions. In the low temperature regime, about 400°C, the evolution of the microstructure with exposure ( $p\text{O}_2 \times t$ ) is smooth and is governed by the heterogeneous nucleation of small stacking faults at 211 particles. In the high temperature regime a decomposition of 123 to 211 and a Ba - Cu oxide is preferentially induced at the 123 - 211 interfaces, the stacking faults grow beyond neighboring 211 particles, and the homogeneous nucleation of stacking faults within the matrix becomes a relevant mechanism of generation of new dislocations. Processing of transmission electron microscopy images was applied to quantify the partial dislocation and stacking fault surface density. Inductively determined  $J_c$  and  $L_{irr}$  clearly reveal that the pinning efficiency of the different microstructures is optimized when the total length of partial dislocations bounding the stacking faults is maximized relative to their surface area. The behavior of  $J_c(H,T)$  is consistent with a point-like pinning mechanism of the c-oriented flux line lattice by dislocation lines lying on the (001) plane.

#### 10:45 AM II7.7

TWIN REFINEMENT FOR A HIGH CRITICAL CURRENT DENSITY IN YBCO. Linfeng Mei, Siu-Wai Chan, Mater. Sci. & Eng. Program, Columbia University, New York, NY.

Twin boundaries in YBCO have been proved to be anisotropic flux pinning centers, and samples with fine twin structure can have high critical current densities. In this work, twin boundaries in melt-textured growth (MTG) YBCO are refined by both  $\text{PtO}_2$  doping and increasing the isothermal annealing temperature from 450 ° to 680 °. The twin structure is studied by TEM, and the superconducting properties are measured by SQUID magnetometer. Our work shows that by increasing the annealing temperature, the twin boundaries in MTG YBCO are greatly refined, therefore the superconducting properties can be significantly improved.  $J_c(77\text{K}, 1\text{T})$  can be enhanced by 20,000 A/cm<sup>2</sup>, while the maximum flux pinning force increased by 138%. Twin boundary engineering for a high critical current density in melt-textured YBCO has been achieved.

#### 11:00 AM II7.8

MODIFICATION OF STRUCTURE AND PROPERTIES OF SUPERCONDUCTING YBCO CRYSTALS BY COMBINED IRRADIATIONS. Elvira M. Ibragimova, Inst of Nuclear Physics, Tashkent, UZBEKISTAN; Marquis A. Kirk, Materials Science Division, Argonne National Laboratory, Argonne, IL.

Effects of electron and neutron irradiations in combination with  $^{60}\text{Co}$  gamma-quanta on magnetic properties of  $\text{YBaCuO}$  crystals have been studied to determine 1) the structure of radiation-induced pinning centers, 2) the contribution from oxygen defects to the pinning, and 3) the improvement in the critical parameters  $T_c$ ,  $J_c$ ,  $H_c$  of the crystals. The concentration of radiation-induced defects in oxygen sublattices of the layered structure was varied by means of combined and sequential irradiations with fast particles and ionizing gamma-rays at different temperatures, doses, dose rates, and by substituting Cu ions with non-isovalent diamagnetic impurities. Irradiation with 350 keV-electrons produced oxygen atom displacements both in CuO chains and planes. The defects responsible for the flux pinning at 77 K with a maximum at a magnetic field of 4 Tesla may be charged oxygen vacancies in CuO-planes, having local magnetic moments. It was shown that the oxygen defects also contribute substantially to the flux pinning at cation-anion defect clusters created by moderate fluences of 1 MeV-electrons and fast neutrons. Radiation annealing or regrouping of several oxygen defects is also possible under the combined irradiation. Moreover, both the maximum of flux pinning and the pinning force can either increase or decrease after the gamma irradiation. Cation defects can serve as centers for attracting oxygen defects. Being close to each other, the oxygen defects exert magnetic interactions causing the pinning force. These results and this mechanism may explain why charged particles like protons and ions are more effective for producing flux pinning centers.

#### 11:15 AM II7.9

$J_c$  ENHANCEMENT IN MELT TEXTURED YBCO BY FAST NEUTRON IRRADIATION AS A FUNCTION OF THE INITIAL MICROSTRUCTURE. A. Kohler, W. Seebock, H.W. Weber, Atomic Institute of the Austrian Universities, Vienna, AUSTRIA; J. Plain, T. Puig, A.E. Carrillo, X. Obradors, Institut de Ciencia de Materials de Barcelona, CSIC, Bellaterra, SPAIN.

Melt textured YBCO samples with and without 211 inclusions were irradiated by fast neutrons. The enhancement of the critical current density was studied as a function of the 211 content by means of SQUID magnetometry at temperatures from 5 to 77 K and magnetic fields from 0 to 8 T. The highest enhancement factor, found at 77 K and 1.5 T for a 211-free sample, is 27. The enhancement is less effective for the samples with 211 inclusions. However, after the irradiation the critical current densities show a similar temperature and field dependence for all samples. The role of the initial microstructure on the radiation effects will be discussed.

#### 11:30 AM II7.10

AC LOSS IN SUPERCONDUCTIVE CABLE: COMPARISON OF YBCO THIN FILMS AND COMPOSITE BSCCO/Ag TAPES. H.R. Kerchner and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

YBCO films grown on both  $\text{SrTiO}_3$  single crystal and rolling assisted biaxially textured (Ni) substrates (RABiTS) carry high critical current densities. New multi-filamentary BSCCO tapes also have quite high critical currents in the absence of applied magnetic fields. In each case ac current flow causes power loss [W.T. Norris, J. Phys. D 3 489 (1970)]. In the low-current regime, loss in a film strip much larger than that calculated by Norris is associated with the peak current density at the film edge arising from the barrier to vortex entry [H.R. Kerchner et al. Phys. Rev. B 60, 6878 (1999)]. The degree to which the observed power loss for multi-filamentary BSCCO tapes agrees with the calculated results depends primarily upon the quality of filament-to-filament coupling. The possible role of a barrier to vortex entry into a BSCCO filament will be discussed.

### SESSION IIS: PROPERTIES AND PERFORMANCE OF BULK 123-TYPE SUPERCONDUCTORS

Chairs: Winnie Wong-Ng and Christian Jooss  
Thursday Afternoon, November 30, 2000  
Room 306 (Hynes)

#### 1:30 PM II8.1

MICROSTRUCTURE CONTROL OF  $\text{Re-123}$  SUPERCONDUCTOR ON INTERACTION OF PINNING CENTERS AND GROWING CRYSTAL SURFACE. Masanobu Awano, Yoshinobu Fujishiro, Natl, Industr, Res, Inst of Nagoya, Nagoya, JAPAN; Fatih Dogan, Washington Univ, Dept of Materials Science and Engineering, Seattle, WA.

Crystal growth condition of  $\text{Re-123}$  superconductor in polycrystalline body under applied stress was studied for the microstructure control to improve superconducting current properties. Behavior of crystal growth front of superconducting grains through the solid state diffusion with or without the existence of a small amount of liquid phase was examined in various conditions of temperature and applied stress. Interfacial reaction of introduced pinning centers and growing surface of superconducting matrix grains was investigated to promote the uniform dispersion of nanoparticles in matrix for increasing  $J_c$ .

#### 1:45 PM II8.2

ENHANCEMENT IN TRANSPORT PROPERTIES OF MELT TEXTURED YBCO BY DOPING. Wai Lo<sup>1</sup>, Yu X. Zhou<sup>1,2</sup>, Tong B. Tang<sup>2</sup> and Kamel Salama<sup>2</sup>. <sup>1</sup>Texas Center for Superconductivity and Department of Mechanical Engineering, University of Houston, Houston, TX. <sup>2</sup>Department of Physics, Hong Kong Baptist University, Kowloon, HONG KONG.

Substantial effort worldwide has been focused in recent years on the incorporation of nanometer scale weak superconducting regions into melt textured superconductors so as to enhance their critical current density ( $J_c$ ), trapped fields and levitation force. The enhancement in these transport properties is derived from the fact that the size of the weak superconducting regions is close to the coherence length of the superconductors so that flux pinning effects can be maximized. An example of such materials is the  $\text{Nd}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-d}$ ; (NdBCO) solid solution, where substitution of Nd3 for Ba2 results in the formation of nm scale low transition temperature ( $T_c$ ) regions and raises the  $J_c$  in melt textured NdBCO. Making use of the properties of the charge reservoir layer as well as the  $\text{CuO}_2$  conduction planes in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ ; (YBCO) lattice, this study demonstrates that apart from Ba-site doping, transport properties of YBCO are very sensitive to Cu-site dopings on both CuO chain and  $\text{CuOZ}_2$  plane. It also explores the dimensionality on which each dopant suppresses local superconductivity. By optimizing the growth conditions and level of doping, as well as controlling the oxygen deficiency of melt textured YBCO, substantial improvement in the transport properties of these materials has been achieved. Due to the simplicity of the processing conditions, these doping-enhanced melt textured YBCO are

particularly suitable for large scale productions and hence can have important implications on the development of related engineering devices.

### 2:00 PM I18.3

INFLUENCE OF SUBMICRON 211 ADDITION ON FLUX PINNING IN MELT PROCESSED YBCO SUPERCONDUCTORS. Y. Feng, A.K. Pradhan, J.G. Wen and N. Koshizuka, ISTEC, Superconductivity Research Laboratory, Tokyo, JAPAN.

For high temperature superconductor applications, melt textured YBCO bulk materials are one of the most promising candidates especially in large scale system. The superconducting properties of YBCO are highly dependent on the microstructure, which is sensitive to the sample composition and processing technique. It has been established that the introduction of 211 particles to YBCO can improve the flux pinning feature and mechanical property. In this work, the YBCO samples with different amount of submicron 211 addition (0, 10wt%, 15wt%, 20wt% and 25wt%) were fabricated by a power melting process method. It is found that the maximum processing temperature is reduced very much by adopting the ultrafine 211 particles. The magnetization curves and magnetic relaxation were measured by a SQUID magnetometer at various temperatures. The results indicate that the irreversibility line and  $J_c$  increase with the increase of 211 addition, while  $T_c$  does not change much. A large irreversibility field can be obtained in the samples with high density of 211 addition. TEM observations show that many ultrafine 211 particles and a high density of dislocations are found in these samples, which may explain the high irreversibility line. Moreover, a peak effect in  $J_c(H)$  curves is observed in the sample with 10wt% 211 addition, whereas  $J_c$  drops with field according to a power law. In other samples. The temperature dependent  $J_c$  and flux pinning force are also discussed in this paper.

### 2:15 PM I18.4

MAGNETIC PROPERTIES OF MELT-TEXTURED DYBCO SINGLE-DOMAINS. Ph. Vanderbemden, M. Ausloos and R. Cloots, SUPRAS, University of Liege, BELGIUM.

This communication aims at reporting the superconducting properties measured on several bulk melt-processed  $\text{DyBa}_2\text{Cu}_3\text{O}_{7-x}$  samples by using various measuring techniques. The original single domain material was first cut into 9 similar cubic samples which have been characterized by AC susceptibility, flux profiles and DC magnetization. Several magnetic fields amplitudes and directions were used. The results are characteristic of good quality melt-processed (RE)BCO material with  $T_c = 89$  K. The properties of the 9 samples were found to coincide within 5% limit. Next, the effect of various oxygen treatments on these samples has been studied. The influence of the non-uniformity of the oxygen content on the magnetic property anisotropy has been discussed. Finally, additional magnetic measurements were carried out by using a couple of pick-up coils wound on the same sample. From the results we can conclude that the bending of flux lines has to be taken into account in order to extract the critical current density from the magnetic properties.

### 2:30 PM I18.5

THEORETICAL MODELING OF STRUCTURAL AND DEFECT PROPERTIES OF  $\text{RE}(\text{Ba}_{1-x}\text{Ln}_x)_2\text{Cu}_3\text{O}_y$ , WHERE  $\text{RE}=\text{Y},\text{Nd},\text{Eu}$  and  $\text{Ln}=\text{Eu},\text{Nd},\text{La},\text{Pr}$ . H.B. Su, Dept of Materials Science and Engineering, SUNY at Stony Brook; D. Welch, Energy Sciences and Technology Dept, Brookhaven National Laboratory, Upton, NY.

Compounds made by trivalent rare-earth substitutions and replacements in the high-temperature superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_7$ , (Y-123), are of interest for use in various bulk applications such as levitators, bearings, etc., and have been the subject of numerous experimental investigations. To aid in the understanding of such materials, we have developed interatomic potential models for RE-123 cuprates and used them to estimate various cohesive and structural properties, including heats of formation, elastic properties and the response of structural parameters, such as  $\text{CuO}_2$  plane buckling, to hydrostatic and uniaxial stress. The energy and structure of various point defects, such as vacancies, interstitials, and antisite defects were calculated. Trends with the type of RE-123 compound ( $\text{RE}=\text{Y},\text{Nd},\text{Eu}$ ) and with non-stoichiometry and doping will be discussed, and the implications for flux pinning and processing will be considered. This research was performed under the auspices of the U.S. Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under Contract No. DE-AC02-98CH10886.

### 3:15 PM I18.6

COMPARISON OF THE LOCAL AND LONG RANGE STRUCTURAL CHANGES ACCOMPANYING BROMINATION AND CHLORINATION OF UNDERDOPED SINGLE-CRYSTAL YBCO: RECOVERY OF SUPERCONDUCTIVITY. L.M. Dieng,

T.A. Tyson, NJIT, Dept. of Physics, Newark, NJ; M. Croft, Dept. of Physics, Rutgers University, Piscataway, NJ.

To understand the role of bromination and chlorination in the recovery of superconductivity in underdoped YBCO, the spatially resolved local structural changes in brominated and chlorinated single crystals have been measured and compared to those in normal crystals. The local structure about the Y, Ba, Cu and Br/Cl sites was determined. A comparison with normal YBCO crystals with a range of oxygen content was performed. While the high energy XAFS yielded local structural information, evidence for charge transfer was derived from the near edge spectra. We address the issue of the incorporation of Br/Cl into the lattice and develop structural models for the recovery of superconductivity in each case.

This research is supported by National Science Foundation Career Grant DMR-9733862.

### 3:30 PM I18.7

FEMTO-SECOND PHOTO INDUCED PAIR BREAKING SPECTROSCOPY OF HIGH  $T_c$  SUPERCONDUCTORS. Eric Li, Y.G. Zhao, R.P. Sharma, S.B. Ogale, T. Venkatesan, Center for Superconductivity Research, Department of Physics, University of Maryland at College Park, College Park, MD; J.J. Li, W.L. Cao, C.H. Lee, Department of Electrical Engineering, University of Maryland at College Park, College Park, MD.

Using 100 fsec laser pulses, photo induced kinetic inductance signals, which is related to the Cooper pair breaking rate, were measured as a function of the photon energy in High  $T_c$  cuprate based coplanar waveguides. Three very sharp features with FWHM of the order of 30 meV were seen at 1.5, 1.54 and 1.63 eV at 80K in YBCO. No such peaks were seen in LSCO in the photon energy range studied (1.40 eV - 1.70 eV). The amplitude of the peak at 1.63 eV diminished with decreasing temperature and the peak at 1.5 eV showed significant increase with decreasing temperature. The relative separations of the three peaks were 40 and 70 meV, close to the resonance seen in the neutron study of the spin DOS and the predicted optical magnon excitation energy. The Cooper pair breaking rate is also found to be extremely non-linear with the average laser power, indicating a highly coupled system. These observations suggest the presence of inhomogeneities even in the superconducting state of optimal doped YBCO thin films.

### 3:45 PM I18.8

EVIDENCE FOR ELECTRONIC PHASE SEPARATION IN HIGH  $T_c$  CUPRATES. R.P. Sharma, S.B. Ogale and T. Venkatesan, Center for Superconductivity Research, Physics Department, University of Maryland, College Park, MD; Z.H. Zhang, J.R. Liu and W.K. Chu, Texas Center for Superconductivity, University of Houston, TX; Boyd Veal, A. Paulikas and H. Zheng, Materials Science Division, Argonne National Laboratory, Argonne, IL.

Direct experimental evidence for electronic phase separation in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  as a function of oxygen doping has been observed by ion channeling (an ultrafast probe of small incoherent atomic displacements). The stripe dynamics in YBCO has been predicted to undergo three phase crossovers/transitions as a function of temperature at the stripe phase ( $T_1 \sim 200$  K), spin gap formation temperature ( $T_2 \sim 150$  K) and the global superconducting transition  $T_c$ . Three similar temperatures are seen in the form of non-anisotropies in the incoherent lattice fluctuations. A weak dependence of  $T_1$  and  $T_2$  on oxygen doping has been observed. Below  $T_3$  in the superconducting state there is a small increase in the incoherent lattice displacements, consistent with the notion of a slowing of boundary fluctuations, which the lattice could follow easily. In the non superconducting sample  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ ,  $x \sim 0.65$ , the rate of increase of atomic displacement  $u$  as a function of temperature is found to be higher than the calculated Debye value for the system. This increase in  $u$  is consistent with electronic phase separation, even in the non superconducting sample. However their temperature dependence largely reflects a classical character.

### 4:00 PM I18.9

LOW TEMPERATURE THERMAL CONDUCTIVITY OF CUPRATE SUPERCONDUCTORS. S. Nakamae, K. Behnia, ESPCI, Paris, FRANCE; N. Hussey, Dept of Physics, Loughborough University, Loughborough, UNITED KINGDOM; F. Rullier-Albenque, SPEC-CEA Saclay, Gif-sur-Yvette, FRANCE; T. Tamegai, Dept of Applied Physics, University of Tokyo, Tokyo, JAPAN; L. Forro, Ecole Polytechnique Federale de Lausanne, Lausanne, SWITZERLAND; C. Urano, H. Takagi, Dept of Advanced Materials Science, Graduate School of Frontier Sciences, University of Tokyo, Tokyo, JAPAN; S. Adachi, S. Tajima, Superconducting Research Laboratory, International Superconducting Research Center, Tokyo, JAPAN.

Thermal conductivity ( $\kappa$ ) measurements offer rich information on the inner workings of materials and thus have been used widely in both

experimental and theoretical investigations of high temperature superconductors (HTS). We have conducted two separate studies on the thermal conductivity on HTS in a wide temperature range (100 K - 100 mK) in order to verify the validity of some of the existing theories. First, the effect of point defects introduced by electron irradiation on the in-plane  $\kappa$  was investigated on several optimally doped Bi2212 crystals. It has been found that the absolute value of  $\kappa$  decreases with increased number of point defects at all temperature range, while the linear term in  $\kappa$  at very low temperatures shows little or no change. These observations are in quantitative agreement with theoretical predictions made on the nature of quasiparticles in HTS. Second, we have performed the very first measurements on  $\kappa$  along the crystal axis  $a$  and  $b$  of Y124 crystals, the only known compound that is naturally stoichiometric and under-doped. Surprisingly, we find no linear thermal conductivity term in this compound in either direction. Our finding provokes renewed debate on the currently accepted view of well defined quasiparticles in HTS at very low temperatures. Magnetic field induced effects on low temperature thermal conductivity will also be discussed.

#### 4:15 PM I18.10

LOCAL STRUCTURE ANALYSIS OF THE  $\text{Nd}_1\text{Ba}_2\text{Cu}_3\text{O}_x$  PHASE FOR PROCESSING ADVANCED SUPERCONDUCTING MATERIALS. E.A. Goodilin, D. Peryshkov, M. Makarova, A.P. Soloshenko, N.N. Oleynikov, Yu.D. Tretyakov, Department of Chemistry, Moscow State University, Moscow, RUSSIA; V.V. Petrykin, M. Kakihana, Materials and Structures Lab., Tokyo Institute of Technology, Yokohama, JAPAN; J. Hester, Australian Nuclear Science and Technology Organization, Lucas Heights, AUSTRALIA.

Phase stability, long-range and local structures of the famous  $\text{NdBa}_2\text{Cu}_3\text{O}_x$  superconductor were analyzed on the base of samples obtained using different chemical methods in order to suggest preparation conditions of phases with different cation ordering and superconductivity transition temperatures. Thermal stability was analyzed at 740-1100 in air using reactive, carbon-free and highly homogeneous spray-dried submicron precursors. It was found that the less-substituted solid solution ( $x=0$ ) with the best superconductivity transition temperature  $T_c \sim 94\text{K}$  and an "ideal" structure forms in air at 980-1030C only while  $T_c$ ,  $c$ -axis length and orthorhombicity parameters drop down significantly when preparing below or above this range. When choosing inappropriate oxygenation temperatures in air or pure oxygen, the  $\text{Nd}_{123}$  ( $x=0$ ) phase can undergo solid state decomposition into a solid solution ( $x>0$ ) and  $\text{BaCuO}_2$  with a drastic suppression of  $T_c$  whereas low- $T_c$   $\text{NdBa}_2\text{Cu}_3\text{O}_x$  samples decompose drastically faster compared to initially high- $T_c$  single phase samples. The most essential suppression of  $T_c$  in a fully-oxygenated state was found to occur after aging for 16-450 hrs nearby  $\sim 700\text{C}$ . Structural features of the low- $T_c$  nearly-stoichiometric  $\text{Nd}_1 x\text{Ba}_{2-x}\text{Cu}_3\text{O}_x$  phase ( $x<0.05$ ,  $z \sim 6.9$ ,  $a = 3.897 - 3.899 \text{ \AA}$ ,  $b = 3.902 - 3.908 \text{ \AA}$ ,  $c = 11.707 - 11.719 \text{ \AA}$ ) were investigated for the first time by XRD, EXAFS structure refinement techniques and Raman spectroscopy. It was found that the abnormal lattice parameters and the low superconductivity transition temperature (55-60K) of such a phase are related to structural disorder in the Ba and Nd sites (antisite defects) resulting in oxygen disordering which cannot be avoided by a standard oxidation procedure. It is shown that longer annealing at optimal high temperatures is an important factor for cation order that reduces oxygen disorder and enhances superconducting properties.

#### 4:30 PM I18.11

STRUCTURAL PARAMETERS EVOLUTION IN HTSP-CERAMICS. B.N. Kodess<sup>1,2</sup>, V.A. Sarin<sup>3</sup>, I.P. Zuravlev<sup>1</sup>.

<sup>1</sup>Department of Crystal Metrology VNIIMS, Moscow, RUSSIA.

<sup>2</sup>Material Science Department, ICS&E at Denver. <sup>3</sup>INR, Moscow, RUSSIA.

The time-dependencies of micro- and macrostructure parameters have been studied for the YBCO ceramics with different contents of oxygen. Using conventional x-ray source  $\text{CuK} \alpha$ -radiation data for single and double reflections have been collected in small-angle (15-25 degree in two-theta scale), middle (45-66) and high angle (100-145) regions. In each cycle of these measurements the variation in angle position of Bragg reflection and half-width connected with microstress level has been determined. Data have been collected at ambient temperature and constantly orientation of specimen. Earlier these phenomena were introduced as the results either occasional measurements in during several years or in turn three-specimens in during six weeks only. In addition these experiments have been carried continuously in during 900-1500 hours corresponding to life service following x-ray tube. For data adjust on the next tubes we have used our Standard Reference Materials of Lattice Parameters and Ratio of Intensity. This approach provide more detail investigations time-spatial structure forming and self-oscillations process on the  $a$ ,  $b$ ,  $c$  lattice parameters and orthorhombicity ( $b$ - $a$ ) degree, and phase separations on the become poor and enriched parts. The atomic positions, their occupancies as

the result of sorption and desorption of the oxygen from environmental have been evaluated from full diffraction pattern (5-165 degree) following Rietveld refinement procedures. New detail time-dependencies are in agreements with our hypothesis (MRS Spring Meeting, 1994) about the role of nonlinear phase transitions include self-organizations of the all of structural parameters in this superconductive multi-components system. Self-organization structural parameters in time and space are the result of anisotropic mass transfer and defect interactions with various lengths scales.

#### 4:45 PM I18.12

JOINING OF LARGE YBCO MONOLITHS BY MELT TEXTURED YBCO/Ag WELDING AGENT. P. Rodriguez Jr., A.E. Carrillo, T. Puig, J. Figueras, X. Obradors, Institut de Ciencia de Materials de Barcelona, CSIC, Campus UAB, Bellaterra, SPAIN.

The field of welding large melt textured monoliths by superconducting joints has recently attracted much attention, especially for applications like motors, fault current limiters and transmission lines. We present a novel welding agent, i.e. YBCO/Ag composites, which having the peritectic temperature below that of the YBCO material, enables the fabrication of arbitrary geometries without deterioration of the properties. The characteristics of the welding agent, YBCO/Ag composites are primarily analyzed. It is confirmed that the density of microcracks and porosity is reduced, as well as the critical current density is strongly increased. Second, the potential of this welding agent to join YBCO monoliths is strengthened. This welding methodology permits to avoid the segregation of the  $\text{Y}_2\text{BaCuO}_5$  phase, porosity formation and crack generation at the joint. Results on  $ab$  and  $ac$  joints regarding microstructure characterization, magnetoresistance measurements and inductive critical current densities are discussed.

### SESSION I19: POSTER SESSION SUPERCONDUCTING MATERIALS – CHARACTERIZATION, APPLICATIONS, AND PREPARATIVE METHODS

Chairs: Piotr W. Klamut and U. (Balu) Balachandran  
Thursday Evening, November 30, 2000  
8:00 PM

Exhibition Hall D (Hynes)

#### I19.1

MICROTEXTURE MAPPING OF ELECTRON BACKSCATTER DIFFRACTION IN  $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-x}$  SUPERCONDUCTOR TAPES. Sean Li, Thiam Teck Tan, Division of Materials Engineering, School of Materials Engineering, Nanyang Technological University, SINGAPORE.

It is believed that the grain boundaries act as weak-link to limit critical current density in bulk high- $T_c$  superconductors and the weak-link problem can be greatly reduced by elimination or minimization of the large-angle grain boundaries. It has been reported that the distribution of the current paths in  $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-x}$  ( $\text{Bi}2223$ ) superconductor tapes presents a parabolic relationship in the transverse cross-section of the tapes and it was proposed to strongly depend on the microtexture distribution of the  $\text{Bi}2223$  oxides. However, the microtexture distribution of  $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-x}$  crystals with three-dimensional orientations in  $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-x}$  superconductor tapes has not been experimentally determined yet. In present work, Electron Backscatter Diffraction technique was employed to map the crystallographic orientation distribution, determine the misorientation of grain boundaries and also map the misorientation distribution in  $\text{Bi}2223$  superconductor tapes. The results indicate that the  $c$ -axes of almost  $\text{Bi}2223$  grains were perpendicular to the rolling plan, while the 45% of  $a/b$  axes and 45% of  $[110]$  of  $\text{Bi}2223$  grains were aligned along the rolling direction of the  $\text{Bi}2223$  superconductor tapes after heavy mechanical deformation and heat treatment. From the map of the misorientation distribution, it can be observed that the low angle grain boundary are the main boundaries in  $\text{Bi}2223$  superconductor tapes even though there were a number of 45 misorientation boundaries. The low angle boundaries distributed on the side zones of the EBSD measurement areas while the boundaries with 45° misorientation gathered in the middle zone of the measurement areas along the rolling direction. To understand the relationship between the microtexture distribution, the boundary misorientation distribution and the fabrication parameters could be used to optimize the fabrication techniques, thus significantly increasing the critical current density.

#### I19.2

DEVELOPMENT OF LAB-SCALE MAGNETIC SEPARATION SYSTEM USING A CRYOCOOLER-COOLED  $\text{Bi}2223$  SUPERCONDUCTING MAGNET. Takeshi Ohara, Hiroaki

Kumakura, Hitoshi Kitaguchi, Hitoshi Wada, Kazumasa Togano, National Research Institute for Metals, Tsukuba, Ibaraki, JAPAN; Hideto Mukai, Kazuya Ohmatsu, Hiromi Takei, Sumitomo Electric Industries, Ltd., Osaka, JAPAN; Hidehiko Okada, Iwate Industrial Promotion Center, Morioka, Iwate, JAPAN.

We have developed a lab-scale magnetic separation system including a cryocooler-cooled Bi-2223 magnet. The purpose is to experimentally demonstrate the applicability of oxide superconductors to environment-related technologies, namely the separation of harmful substances from slurries. We report about the Bi-2223 magnet developed and experimental separation efficiency of weakly magnetized and very fine  $\alpha$ -hematite particles from water. We used  $\alpha$ -hematite particles as simulant of pollutant scavenger, and their average grain size and magnetic susceptibility are 1.0 micrometer and 1/500, respectively. The Bi-2223 magnet consists of 42 double pancake coils and has a 200mm room temperature bore. Each pancake coil was fabricated by a react-and-wind method using 61-multifilamentary Bi-2223/Ag tapes. To obtain large critical currents, three tapes were bundled and wound into a pancake coil with a polyimide tape for insulation. To reinforce the coil, a stainless steel tape was also co-wound. The inner, the outer diameters and the height of the magnet are 240mm, 306mm and 352mm, respectively. The magnet is cooled down to 13 K with a Gifford-MacMahon (GM) cryocooler. For a magnetic separation system to be practical, it is important to energize and de-energize the magnet rapidly. Excitation test up to 1.7T was repeated at a speed of 1.7T/min. The temperature of the magnet was increased with increasing number of cycles. For an interval of 3.5min between each energizing and de-energizing cycle, the magnet temperature was saturated at 36K after about 20 cycles, and stable magnet operation was accomplished at this temperature. This indicates that the Bi-2223 magnet is promising for magnetic separation. A visual demonstration of the magnetic separation operation will also be reported.

This work was implemented as part of the multicore superconductivity project from 1995 supported by the Japanese Science and Technology Agency (STA).

### II9.3

$P_{O_2}$ -T STABILITY OF THE 2223 SUPERCONDUCTOR IN THE SYSTEM (Bi,Pb)-Sr-Ca-Cu-O-Ag. Lawrence Cook, Winnie Wong-Ng, Ceramics Division, NIST, Gaithersburg, MD; Ralph Klein, Physical and Chemical Properties Division, NIST, Gaithersburg, MD.

The thermodynamic stability of the BSCCO 2223 phase is a function of composition, temperature and oxygen partial pressure; all must be controlled during processing to ensure optimum and reproducible results. Using phase equilibrium data it is ideally possible to control the amount of liquid, and to maximize superconductor phase purity, or, alternatively to cause the formation of accessory phases. We have investigated the effect of  $P_{O_2}$  over a range of four orders of magnitude and found that initial melting temperatures fall by 70°C as the oxygen pressure is decreased over this interval. Therefore the 2223 processing window available in  $P_{O_2}$ -T-X space is significantly expanded relative to that for isobaric conditions. The status of our data set on the location of the 2223 liquid two-phase region in  $P_{O_2}$ -T-X space will be discussed, including data from thermal analysis, powder x-ray diffraction and quantitative microchemical analysis. We will also discuss the status of ongoing calorimetric experiments relevant to the thermodynamic stability of the 2223 phase.

### II9.4

HOMOGENEITY AREA OF  $Bi_2Sr_2CaCu_2O_8$  - BASED SOLID SOLUTIONS WITH SUBSTITUTION OF ALKALINE EARTH ELEMENTS BY Nd AND La. Alexander G. Veresov, A.V. Knotko, D.V. Korolyov, A.V. Garshev, V.I. Putlayev, Dept of Chemistry, Moscow State Univ, Moscow, RUSSIA.

Creation of precipitates in bulk matrix to act as pinning centres is a perspective way to improve critical current density in high temperature superconductors. It is possible to generate such precipitates via partial decomposition of the supersaturated 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 consisted in determination of homogeneity area of  $Bi_2Sr_2Ca_{1-x}R_xCu_2O_{8-d}$  and  $Bi_2Sr_{2-x}CaR_xCu_2O_{8-d}$  ( $R = Nd, La$ ). Semi-quantitative XRD analysis was used to evaluate phase composition of specimens quenched from various temperatures. It was found that solubility limits of Nd and La in Bi-2212 for cases of alkaline earth element substitution by rare earth elements with smaller ionic radii (substitution Ca by Nd; Sr by Nd and La) were the same (values of x are about 0.8). These limits are determined by fraction of Cu ions in lattice site attributed to  $Cu^{2+}$ . In case of substitution of Ca by La stability area of 2212 was found to be much more narrower (values of x about 0.3) compared to other substitutions. One can deduce the fact from the difference of  $Ca^{2+}$  and  $La^{3+}$  ionic radii.

### II9.5

GROWTH OF  $Bi_2Sr_2CaCu_2O_x$  SUPERCONDUCTOR SINGLE CRYSTALS. Takeshi Iwamoto<sup>1</sup>, Satoshi Watauchi<sup>1,2</sup> and Isao Tanaka<sup>1,2</sup>. <sup>1</sup>Yamanashi Univ, Inst Inorganic Synthesis. <sup>2</sup>Japan Science and Technology Corporation, CREST.

Since  $Bi_2Sr_2CaCu_2O_x$  (Bi-2212) is an incongruent-melting compound as well as solid solution, Bi-2212 single crystals are grown mostly by the traveling solvent floating zone (TSFZ) and flux methods.

However, the crystals grown by those methods are very thin along the crystallographic c-axis due to highly anisotropic growth rate and also their composition is not homogeneous. In this study, Bi-2212 single crystals of high quality and large size were grown by the traveling solvent zone melting (TSZM) method using a silver crucible. The merits of the TSZM method are precise growth temperature control and stable transfer of the molten zone compared with the TSFZ method. Silver is used as crucible material so that even contaminated from the crucible does not affect the superconductivity in Bi-2212 crystals. The silver crucible was transferred along the vertical and horizontal directions using a vertical and a horizontal type furnace, respectively. The growth conditions such as the zone transfer rate, the growth temperature, and the growth atmosphere were varied, and then the optimum growth conditions were determined. The results of the crystal growth are described in details.

### II9.6

Pb-DOPED Bi-2212: HOMOGENEITY AREA, OXIDATION AND EFFECT ON  $j_c$ . A. Veresov, M. Pulkin, Moscow State Univ, Dept of Materials Sciences, Moscow, RUSSIA; A. Knotko, V. Putlayev, Moscow State Univ, Dept of Chemistry, Moscow, RUSSIA; E.K.H. Salje, Univ of Cambridge, Dept of Earth Sciences, Cambridge, UNITED KINGDOM.

The most promising way to improve  $j_c$  in superconductors is likely to be a creation of small precipitates in the material during decomposition of supersaturated solid solution. It was recognised recently that Pb-doping of 2212 could have beneficial effect on  $j_c$ .  $Bi_{(2.1-x)}Pb_xSr_2CaCu_2O_{(8-d)}$  ( $x=0.1...0.7$ ) was prepared at different temperatures and oxygen partial pressure ( $p_{O_2}=10^{-3} - 0.21$  atm) via a nitrate route. The effect of  $p_{O_2}$  and Pb-doping on Bi-2212 phase formation has been explored with XRD, SEM/EPMA and TEM/SAED. The limit of Pb solubility in Bi-2212 was found to increase with  $p_{O_2}$  decreasing and corresponded to  $x=0.3$  at  $p_{O_2} = 0.21$  atm (850°C) and  $x=0.6$  at  $p_{O_2} \sim 10^{-3}$  atm (750°C). TEM study of the as-synthesised solid solutions revealed significant nanoscale inhomogeneity attributed to Pb-segregation. For the samples with  $x > 0.3$  the inhomogeneity quite often looked like well-ordered wave with a period of about 10 nm. The samples of (Bi,Pb)-2212 processed under  $p_{O_2}$  conditions were oxidized at 600-750°C in air for 0.5-60 hrs in order to cause precipitation of secondary phases from the super-saturated solid solution. Thermogravimetric study (TG) of the oxidation gave a strong evidence of two stages composing the process. It was found that  $j_c$  was enhanced by factor of 2 after annealing at 650°C (i.e. during the first stage of oxidation).

### II9.7

A NEUTRON POWDER DIFFRACTION STUDY OF THE FERROMAGNETIC SUPERCONDUCTOR  $Gd_{1.3}Ce_{0.7}Sr_2Cu_2RuO_{10-d}$ . Christopher S. Knee, Mark T. Weller, University of Southampton, Dept of Chemistry, UNITED KINGDOM.

The crystal structure of the ferromagnetic superconductor  $^{160}Gd_{1.3}Ce_{0.7}Sr_2Cu_2RuO_{10-d}$  which orders magnetically at  $T_N \sim 180$  K and exhibits a  $T_c \sim 40$  K has been investigated by neutron powder diffraction. Superconductivity occurs in the  $CuO_2$  layers while the magnetic order is associated with the Ru moments. The tetragonal structure evolves from that of  $YBa_2Cu_3O_{7-d}$  by inserting a fluorite-type  $Gd_{1.3}Ce_{0.7}O_2$  layer instead of the Y ion and replacing the chain copper site with  $RuO_6$  octahedra. The fluorite layer shifts the alternate  $CuO_5$ - $RuO_6$ - $CuO_5$  perovskite blocks by a/2 resulting in body centred symmetry (S.G. I4/mmm) and an increased c-parameter of  $\approx 28.5$  Å. The basal oxygen of the  $RuO_6$  octahedra are displaced within the xy-plane to accommodate physically reasonable Ru-O in-plane bonds. These displacements are consistent with rotations of the octahedra by  $\approx 13.5^\circ$  perpendicular to the c-axis. No evidence of a supercell arising from extended ordering of the  $RuO_6$  orientation is observed. The temperature dependence of the structure was monitored with scans at 10 K, 140 K and 298 K. The basal Cu-O interactions display the expected contraction, however, the apical Cu-O bond exhibits negative thermal expansion and increases significantly upon cooling. The increase is offset by the contraction of the apical Ru-O bond, which leaves the Cu-Cu separation almost unchanged. The largest changes in the apical interactions occur between 10 K and 140 K with the oxygen moving  $\sim 0.02$  Å closer to the central Ru ion whilst the Cu-Cu thickness remains constant. This behaviour is the exact opposite of that displayed by the closely related  $GdSr_2RuCu_2O_8$  and suggests a structural transition

associated with  $T_c$  rather than  $T_N$ . Additional magnetic scattering intensity consistent with either ferromagnetic or antiferromagnetic ordering of the Ru spins was not observed.

#### II9.8

TUNING OF STRUCTURE AND PROPERTIES THROUGH DIAMAGNETIC CATION SUBSTITUTIONS IN THE FERROMAGNETIC SUPERCONDUCTOR  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$ . Abbie C McLaughlin, Veronika Janowitz, Judith A. McAllister, J. Paul Attfield, Univ of Cambridge, Dept of Chemistry and IRC in Superconductivity, Cambridge, UNITED KINGDOM.

Superconductivity and ferromagnetism do not usually coexist in the same phase, however, their coexistence has recently been observed in the 1212 type layered cuprate  $\text{RuSr}_2\text{GdCu}_2\text{O}_8$ , which is a bulk superconductor with  $T_c = 46$  K. The magnetic transition at 136 K results from the ordering of moments in the ruthenate planes which persists through the onset of superconductivity. Solid solutions  $\text{Ru}_{1-x}\text{M}_x\text{Sr}_2\text{GdCu}_2\text{O}_8$  have been prepared with diamagnetic substituents  $M = \text{Sn}$  ( $x < 0.075$ ) or  $\text{Nb}$  ( $x < 0.20$ ). Powder diffraction and physical measurements show that substitution of Sn and Nb occurs only at the Ru sites, and a suppression of the ferromagnetism of the ruthenate layers is observed in both cases. However,  $T_c$  increases with Sn doping but decreases with Nb substitution, evidencing a tuning of the hole transfer to the cuprate layers by band overlap.

#### II9.9

IMPROVEMENT IN FABRICATION OF Hg-1223 SUPERCONDUCTOR BY Te DOPING. K.K. Yau, C.C. Lam, L.J. Shen, S.H. Li, City University of Hong Kong, Dept of Physics and Materials Science, Hong Kong, PR CHINA.

The sealed quartz tube method is one of the usual methods used for sintering mercury-based copper oxide superconductors. By this method, precursor pellets are inserted into the quartz tube together with certain mass ratio of the reagent sample pellets to control the mercury vapour pressure. A certain amount of excess HgO is required to ensure the fabricated sample with the desired composition. In our research, it was found that if a small amount of  $\text{TeO}_2$  was added and mixed with the pulverized precursor after first calcination, Hg-1223 superconductors can be fabricated without using the precursor pellet and no excess HgO is required. Thus, the fabrication process can be simplified. As mercury and its oxide are toxic, if no excess Hg is involved, it is safe and good for the laboratory-environment. In this research, some amounts of 0.1 and 0.18 wt% (weight percentage) of  $\text{TeO}_2$  were added into the precursor, it is shown that the critical temperature of the fabricated samples is comparable to that of the undoped Hg-1223 samples. For 0.1 wt% of  $\text{TeO}_2$ , the  $T_C$  of the as-synthesized sample is about 111 K whereas the annealed Te-doped Hg-1223 is about 133 K. For 0.18 wt% of  $\text{TeO}_2$ , the  $T_C$  of the as-synthesized and annealed samples are 114 K and 134 K, respectively. The phase structure of samples fabricated in this research has been analyzed using the X-ray diffraction technique. It shows that the Hg-1223 phase is dominated.

#### II9.10

SYNTHESIS OF  $\text{Hg}(\text{RE})\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  SINGLE CRYSTALS USING BAZRO<sub>3</sub> CRUCIBLE BY  $\text{BaF}_2$  ADDITION. Takayuki Sato, Jun-ichi Shimoyama, Shinya Ueda, Shigeru Horii, Kenji Otzsch and Kohji Kishio, Tokyo Univ, Dept of Superconductivity, Tokyo, JAPAN.

$\text{HgBa}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  (Hg1223) superconductor exhibits the highest  $T_c$  of 135K under ambient pressure among all superconducting materials, and its chemical instability and poor flux pinning performance were well-improved by Re substitution for Hg. Although several groups succeeded in preparation of Hg(Re)1223 single crystals, their basic physical properties have not been systematically studied. In the present study, synthesis of  $\text{Hg}_{0.75}\text{Re}_{0.25}\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  (HgRe1223) single crystals was attempted by flux method using high quality  $\text{BaZrO}_3$  crucibles in order to evaluate the anisotropic properties as well as the magnetization behaviors. After the precursor was prepared from the starting materials of  $\text{ReO}_3$ ,  $\text{BaCO}_3$ ,  $\text{CaCO}_3$  and  $\text{CuO}$  powders, HgO and  $\text{BaF}_2$  reagents were added and mixed to be a nominal composition of  $(\text{Hg}_{0.75}\text{Re}_{0.25})\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_y\text{F}_z$  ( $z = 0 - 0.2$ ). The mixed powders were put into  $\text{BaZrO}_3$  crucible which was inactive to the flux liquid in contrast to  $\text{Al}_2\text{O}_3$  crucible, then sealed in an evacuated quartz ampoule. Typical temperature pattern of crystal growth was as follows, heating up to 950°C, kept for 1h, cooled down to 800°C at a rate of 10°C/h and cooled in the furnace. By  $\text{BaF}_2$  addition, partial melting point of the mixture was lowered and melt-solidification could be performed. Analyses and characterization of the grown crystals are now in progress.

#### II9.11

SYNTHESIS AND CHARACTERIZATION OF LAYERED OXYSULFIDES  $(\text{Cu}_2\text{S}_2)(\text{Sr}_{n-1}\text{M}_n\text{O}_{3n-1})$ . Hiraku Ogino, Kenji

Otzsch, Jun-ichi Shimoyama, Kohji Kishio, Univ of Tokyo, Dept of Superconductivity, Tokyo, JAPAN.

Layered oxysulfides  $(\text{Cu}_2\text{S}_2)(\text{Sr}_{n-1}\text{M}_n\text{O}_{3n-1})$  ( $n = 1-3$ ), which are newly discovered in 1997, are made of a stacking of perovskite like  $\text{MO}$  layer and anti-fluorite  $\text{Cu}_2\text{S}_2$  layer, and this perovskite-based layered structure is similar to that of high- $T_c$  superconductors and low-dimensional magnetic materials. More than ten compounds have been discovered by changing transition metal  $M$ , number of  $\text{MO}_2$  sheets  $n$ . In our previous study, new layered oxysulfides,  $(\text{Cu}_2\text{S}_2)(\text{Sr}_3\text{Sc}_2\text{O}_5)$ ,  $(\text{Cu}_2\text{S}_2)(\text{Sr}_2\text{NiO}_2)$ , and  $(\text{Cu}_2\text{S}_2)(\text{Sr}_2\text{CuO}_2)$ , were obtained by improvement of synthesis conditions [1]. Among them,  $(\text{Cu}_2\text{S}_2)(\text{Sr}_2\text{CuO}_2)$  has  $\text{CuO}_2$  planes and, therefore, appearance of superconductivity can be expected if appropriate amount of carrier can be doped. In the present study, we have discovered a new layered oxysulfide  $(\text{Cu}_2\text{S}_2)(\text{Sr}_2\text{MgO}_2)$ . Samples were prepared by solid-state reaction starting from  $\text{SrS}$ ,  $\text{Cu}_2\text{O}$ , and  $\text{MgO}$ . The stoichiometric composition powders were mixed in an inert gas atmosphere, pelletized and sealed in evacuated quartz tube. Sintering conditions was optimized to be at 450°C for 12h. The resulting sample was characterized by XRD, HR-TEM, and SQUID magnetometer. From Rietveld refinement,  $(\text{Cu}_2\text{S}_2)(\text{Sr}_2\text{MgO}_2)$  was found to have a stacking of tetragonal  $\text{MgO}_2$  plane and  $\text{Cu}_2\text{S}_2$  layer, which is an identical structure with  $(\text{Cu}_2\text{S}_2)(\text{Sr}_2\text{CuO}_2)$ . Since  $(\text{Cu}_2\text{S}_2)(\text{Sr}_2\text{MgO}_2)$  contains monovalent Cu and divalent Mg and does not contain any magnetic element, this material can be a comparative material for understanding paramagnetic behavior of  $(\text{Cu}_2\text{S}_2)(\text{Sr}_2\text{CuO}_2)$ . [1] K.D. Otzsch et al. *J. Low. Temp. Phys.*, 117, 729 (1999).

#### II9.12

CORRELATION OF STRUCTURAL DISORDER WITH ELECTRICAL TRANSPORT IN EPITAXIAL  $\text{Sr}_2\text{RuO}_4$  FILMS. M.A. Zurbuchen, Y. Jia, S. Knapp, A.H. Carim, and D.G. Schlom, Penn State Univ, Dept of Materials Science and Engineering, University Park, PA; L.-N. Zou and Y. Liu, Penn State Univ, Dept of Physics, University Park, PA; W. Tian and X.Q. Pan, Univ of Michigan, Dept of Materials Science and Engineering, Ann Arbor, MI; G.W. Brown and M.E. Hawley, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM.

$\text{Sr}_2\text{RuO}_4$  is the focus of great interest because it is likely a  $p$ -wave superconductor and is the only non-copper-containing layered perovskite superconductor. So far, superconductivity has been achieved only in single-crystalline samples. A number of experiments, however, would become possible if  $\text{Sr}_2\text{RuO}_4$  could be made in superconducting thin film form. (No superconducting  $\text{Sr}_2\text{RuO}_4$  thin films have been reported to date.) In characterizing our high-purity  $\text{Sr}_2\text{RuO}_4$  films, we have noticed a correlation between structural disorder, as revealed by transmission electron microscopy and x-ray diffraction, and electrical transport. Two types of planar defects have been observed: out-of-phase boundaries (OPBs) and stacking faults. The long coherence lengths and anisotropic order parameter of  $\text{Sr}_2\text{RuO}_4$  make it extremely sensitive to such defects. Strategies to overcome these defects in epitaxial thin films will be discussed.

#### II9.13

$\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$  CERAMICS: LOW TEMPERATURE SYNTHESIS, OXYGEN MOBILITY AND PERSPECTIVES OF CATALYTIC APPLICATIONS. Galina N. Mazo, Oleg A. Shlyakhtin, Stanislav N. Savvin, Moscow State Univ., Dept. of Chemistry, Moscow, RUSSIA.

Unique crystallochemical features of HTSC cuprates resulted in the development of the series of "nonsuperconducting" applications of these materials. Remarkable and easily variable oxygen nonstoichiometry of substituted  $\text{La}_2\text{CuO}_{4-\delta}$  is a good background for its applications in redox catalytic processes. Development of catalytic materials for particular reactions demands better understanding of synthesis-structure-properties relationships and kinetic features of oxygen diffusion in lanthanum cuprate ceramics. Special processing procedure based on freeze-drying synthesis has been developed in order to reduce synthesis temperature of  $\text{LaSrCuO}_{4-\delta}$  to 600°C and the size of powder crystallites - to 100 nm. The rate of phase formation is strongly influenced by the composition of the salt precursor while the most crucial factors are heating rate and  $\text{PO}_2$  during decomposition. Further sintering of finely grained  $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$  powders allowed to obtain dense ceramic samples at  $T = 1050 - 1100^\circ\text{C}$ . Studies of oxygen mobility performed by dynamic-thermal O18-isotope exchange method demonstrated that substitution of La for Sr in  $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$  ( $x = 0.15$  to 1) results in the decrease of oxygen release onset temperature since 460 to 380°C. Such a dependence correlates quite well with temperature dependencies of diffusion coefficients calculated for various  $x$  via molecular dynamics simulation. Calculated values of diffusion coefficients (for example,  $D = 4 \times 10^{-9}$  at  $T = 800$  K for  $\text{La}_2\text{CuO}_4$ ) fit well experimental data obtained by other authors. All ceramic samples demonstrated substantial catalytic activity in the isotope

exchange reaction between oxygen molecules  $^{32}\text{O}_2$  and  $^{36}\text{O}_2$ . Samples with  $x=0.15$  demonstrated also good catalytic activity in the reaction of methane oxidation.

#### II9.14

CATION CONTROL OF  $\text{A}_2\text{CuO}_4$  STRUCTURES AND PROPERTIES. Judith A McAllister, J. Paul Attfield, Univ of Cambridge, Dept of Chemistry and IRC in Superconductivity, UNITED KINGDOM; Kosmas Prassides, Univ of Sussex, Dept of Chemistry, UNITED KINGDOM.

The superconducting behaviour of high-Tc superconductors is known to depend on the A site doping level and the A cation size. In addition, the variance in the A cation size distribution has recently been found to have an effect on superconducting properties and the  $\text{A}_2\text{CuO}_4$  superstructure type. The control of structure and superconducting property by the size variance has been established through studies of three series of 15% doped  $\text{A}_2\text{CuO}_4$  materials using magnetic and transport measurements,  $\mu\text{SR}$  spectroscopy and variable temperature neutron powder diffraction. These results can be used to construct phase diagrams for all  $\text{A}_2\text{CuO}_4$  compositions using the doping level, average A cation size, and A cation size variance as axes.

#### II9.15

INTER-CHAIN INTERACTION OF METALLIC 1D CHAIN IN  $\text{PrBa}_2\text{Cu}_4\text{O}_8$ . Shigeru Horii, Univ of Tokyo, Dept of Superconductivity, Tokyo, JAPAN; Hidenori Takagi, Univ of Tokyo, Dept of New Materials Science, Tokyo, JAPAN; Hiroshi Ikuta, Uichiro Mizutani, Nagoya Univ, Nagoya, JAPAN; Yuh Yamada, Shimane Univ, Matsue, JAPAN; Izumi Hirabayashi, ISTECSRL, Nagoya, JAPAN; Jun-ichi Shimoyama, Koji Kishio, Univ of Tokyo, Dept of Superconductivity, Tokyo, JAPAN.

$\text{PrBa}_2\text{Cu}_4\text{O}_8$  (Pr124) does not show superconductivity because of "Pr-effect" of  $\text{CuO}_2$  planes in YBCO system. However, resistivity along only the b-axis direction showed a highly conductive behavior, of which the origin is attributed one-dimensional (1D) double chain sites from anisotropic resistivity between a- and b-axes. This result indicates that the 1D double chain in Pr124 is carrier-doped and one of the most reasonable candidates of studying non-Fermi liquid state in 1D system. On the other hand, a coherent behavior was observed at low temperatures for the a-axis resistivity perpendicular to the chain. In this work, in order to elucidate origin of the coherent a-axis transport, we measured a-axis magnetoresistance (MR) in magnetic field along the all crystallographic axes using high quality Pr124 single crystals. We found that temperature dependence of the a-axis MR could be understood in terms of a quasi-1D band picture and the coherent a-axis resistivity below 150K resulted from existence of finite inter-chain interaction along the a-axis direction at low temperatures. In other words, electronic structure of the chain is 2D (or more higher). This conclusion is consistent with quadratic temperature dependence of the a- and b-axis resistivities below 100K which suggests the Fermi liquid state.

#### II9.16

MECHANICAL PROPERTIES OF RE-Ba-Cu-O BULK WITH RESIN IMPREGNATION. Masaru Tomita, Masato Murakami, Superconductivity Research Laboratory, ISTECSRL, Tokyo, JAPAN.

Large single-grain bulk RE-Ba-Cu-O (RE: rare earth elements) superconductors can trap large fields exceeding several teslas and thus can function as very strong quasi-permanent magnets. The stress produced sometimes causes cracking. At present bulk superconductors which is capable of the maximum trapped field and maintaining its performance for a long period of time without being affected by internal or external force such as electromagnetic force or thermal stress or by corrosive environments, and also to a process for producing bulk superconductor. We have recently found that epoxy resin can penetrate into bulk superconductors under appropriate conditions. We measured the thermal expansion of the epoxy resin and bulk superconductors by thermal mechanical analysis alleviating the stress generation due to thermal stress. And furthermore, we measured the local tensile strength of bulk superconductors with resin impregnation by three-point bending test. As a result, it has been confirmed a dramatic improvement of durability and field trapping capability with resin impregnation.

This work was supported by the New Energy and Industrial Technology Development Organization (NEDO) as Collaborative Research and Development of Fundamental Technologies for Superconductivity Applications under the New Sunshine Program administered by the Agency of Industrial Science and Technology (AIST) of the Ministry of International Trade and Industry (MITI) of Japan.

#### II9.17

TEXTURE FORMATION AND SUPERCONDUCTING PROPERTIES OF  $\text{YBa}_2\text{Cu}_3\text{O}_x$  THIN FILMS PREPARED BY A

SOLUTION PROCESS ON  $\text{LaAlO}_3$  SINGLE CRYSTALS. Y.A. Jee, B. Ma, M. Li, B.L. Fisher and U. Balachandran, Argonne National Laboratory, Energy Technology Division, Argonne, IL.

Using trifluoroacetate (TFA) precursors, we grow  $\text{YBa}_2\text{Cu}_3\text{O}_x$  (YBCO) thin films on  $\text{LaAlO}_3$  (LAO) single crystals. The precursor coatings were prepared by first dipping the LAO single crystals in the solution, and then heat treating them at low and high temperature to develop a superconducting phase and c-axis-oriented texture. In this study, processing parameters during the treatment were optimized. Oxygen partial pressure and heat treatment temperature, in particular, were closely related to film properties. To characterize film texture, we used pole figure,  $\phi$ -scan, and SEM analysis. Superconducting properties were also examined by measuring inductive  $T_c$  and transport  $J_c$ . The possibility of fabricating our YBCO films on coated Ni-based substrate will be discussed.

#### II9.18

HIGH-TEMPERATURE SUPERCONDUCTING TAPES DEPOSITED BY THE NON-VACUUM, LOW-COST COMBUSTION CHEMICAL VAPOR DEPOSITION TECHNIQUE. Marvis K. White, Steve L. Krebs, Yibin Xue, Adam C. King, Ian H. Campbell, Todd A. Polley, Dave S. Mattox, Shara S. Shoup, MicroCoating Technologies, Inc., Chamblee, GA.

The enormous technological potential of high-temperature superconductors (HTS) was realized immediately following their discovery in 1986, yet these materials largely remain laboratory curiosities as scientists struggle to scale from coupons of high-performance material to long lengths of practical conductor. To date, all successful architectures for producing long lengths of HTS include a metal substrate to act as the backbone of the flexible tape, single or multiple buffer layers (usually ceramic oxides), and a superconductor (traditionally YBCO). Although both vacuum and non-vacuum processes are being investigated for commercial production, low-throughput vacuum techniques were the first to succeed in producing the buffer and superconducting layers necessary for superconducting tape with high critical currents. However, vacuum processes are not only expensive but impractical when addressing the needs for rapid production of kilometer lengths of wire. The innovative Combustion Chemical Vapor Deposition (CCVD) technology has shown significant promise in depositing the multi-layer structures necessary for successful HTS tape while overcoming many of the shortcomings of traditional vacuum techniques. The key advantage of the CCVD technology is its ability to deposit high quality thin films in the open atmosphere using inexpensive precursor chemicals in solution. This obviates the need for costly furnaces, vacuum equipment, and traditional reaction chambers. As a result, capital requirements and operating costs are reduced significantly when compared to competing vacuum-based technologies. Also, the ability to deposit thin films in the open atmosphere enables continuous, production-line manufacturing. Consequently, throughput potential is far greater than with conventional thin-film technologies, most of which are generally restricted to batch processing. The current status of long lengths of high-temperature superconductors made using CCVD will be discussed.

#### II9.19

TECHNOLOGICAL ( $B, T$ ) PHASE DIAGRAM FROM MAGNETO-TRANSPORT DATA. Marcel Ausloos, SUPRAS, University of Liège, Liège, BELGIUM; Rudi Cloots, Institute of Chemistry, University of Liège, Liège, BELGIUM; Jan Mucha, Institute for Low Temperature and Structure Research, Polish Academy of Sciences, Wrocław, POLAND; Marek Pękała, Department of Chemistry, University of Warsaw, Warsaw, POLAND.

In our on-going investigations about the mixed state properties of HTcS we have been intrigued by how to obtain the various lines used to dissect the ( $B, T$ ) plane in a systematic way. We have found that e.g. the Nernst voltage is an interesting property to be studied in order to probe the temperature dependence of phase transition field lines (PTFL), like the upper critical field  $B_{c2}$  line, the melting ( $T_m$ ) line, the irreversibility ( $T_i$ ) line, the glass ( $T_g$ ) transition line, the electrical resistivity percolation ( $T_p$ ) line and several structural PTFL separating regions in which the vortices form a triangular ( $T_t$ ) or square ( $T_s$ ) lattice or are in a fluid state. This is done by looking at the singularities, i.e. break in slopes, in quantities like the electrical resistivity  $\rho(T)$ , the thermoelectric power  $S(T)$ , or the Nernst effect  $N(T)$  at fixed  $B$  values. The number of observed PTFL is exactly that theoretically predicted. The agreement between the theoretical predictions for  $T_c(B)$  and the actual data, measured as  $B_c(T)$  is discussed. The case of Bi-based HTcS ceramics is used for illustration.

#### II9.20

PROTON BEAM TEXTURING OF SUPERCONDUCTING YBCO

CERAMICS. Makhmud Kalanov, Elvira M. Ibragimova, Institute of Nuclear Physics, Tashkent, UZBEKISTAN.

It is known, that under irradiation of ceramics the energy is lost mainly on the total grain surface including intergranular contacts, causing modification of texture and hence properties. Superconducting (SC) transition, current-voltage characteristics, X-ray diffraction (XRD) and scanning electron microscopy (SEM) of YBaCuO ceramics have been studied after exposure to 18 MeV proton fluences of  $10E13 - 10E15 / \text{cm}^2$  at 300 K. It was found, that in the interval of  $10E13 - 10E14 / \text{cm}^2$  the irradiation induced oxygenation of weak intergrain contacts as determined by XRD, grain alignment as observed both in XRD and SEM pictures, anisotropy and broadening of SC-transition as measured along and across the proton beam, and residual magnetization. The proton induced anisotropic texture is responsible for the critical current increase at 77 K and the resistivity decrease at 90-200 K. In a higher dose interval of  $10E14 - 10E15 / \text{cm}^2$  the SC-transition parameters degrade and the resistivity increases, depending on initial texture, weak intergrain links become deoxygenated and no texture occurs. The SC-structure in bulk of grains does not change. Thus, the existing or the irradiation induced texturing seems to be the way of stabilising ceramics under irradiation. The magnetization (pinning) and the anisotropy of SC-transition can be due to localization of charges at the proton induced defects (mostly in oxygen sublattices). Then, the higher ionization efficiency of particles, the more charged defects and the higher pinning force.

#### II9.21

ON THE PHASE STRUCTURE OF  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ . Irisali Khidirov, Ulmas Gafurov, Institute of Nuclear Physics, Tashkent, UZBEKISTAN.

It is investigated both superconducting and non-superconducting non-stoichiometric ceramics  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ . The measurements were carried out by the neutron diffraction method. The samples were irradiated up to a dose of 3,65(1010 R) in evacuated and scaled ampoules. It has been studied at the powder samples  $\text{YBa}_2\text{Cu}_3\text{O}_{6,98}$  and  $\text{YBa}_2\text{Cu}_3\text{O}_{6,77}$ . The first one had the temperature of the transition to superconducting state of  $T = 89$  K with usual orthorhombic structure. The other sample had semiconductive nature of temperature dependence of electrical resistance in the interval of 77 - 300 K. This sample is characterized by essential oxygen nonstoichiometry on O(1) and O(4) positions and oxygen in O(5) position being absent completely.

The treatment of neutron diffraction pattern of  $\text{YBa}_2\text{Cu}_3\text{O}_{6,77}$  by means of profile Rietveld's method showed that practically there are no changes in the structure. However, the large scatter of experimental points is fixed at the neutron pattern of irradiated sample being near stoichiometry composition ( $\text{YBa}_2\text{Cu}_3\text{O}_{6,98}$ ) and it is observed the changes in proportion of diffraction reflexes intensities and in doublet reflexes form which are visually well-marked at the Wolf-Bragg's angles of  $2(40^\circ)$ . Large statistic distortions arise in the structure when exposed after irradiation. The other changes of diffraction pattern can be caused by modifications in atom arrangement over the various positions or in atom concentrations. The treatment of diffraction patterns Rietveld's method showed a good agreement between experimental and calculated reflex intensities. If Meissner effect has been observed in the starting sample, after irradiation this effect was not found.

The results for powdery sample  $\text{YBa}_2\text{Cu}_3\text{O}_{6,98}$  is analogous to that obtained by annealing at temperature about 450 - 570 K persistently evacuated vacuum; the difference is that the large statistic distortions of the lattice arise in irradiated sample. The different consequences of effect of heat treatment and irradiation at the  $\text{YBa}_2\text{Cu}_3\text{O}_{6,98}$  structure are caused by the various mechanisms of these factors. The atom diffusion plays the main role in structure changes in the case of heat treatment. Irradiation of the samples being non-stoichiometric on oxygen, doesn't lead to noticeable modification of their structure. It is shown that power non-stoichiometric samples is more radiation-steady then stoichiometric one.

### SESSION II10: POSTER SESSION 123-TYPE SUPERCONDUCTORS - THIN FILM AND BULK FORMS

Chairs: Kamel Salama and Kaname Matsumoto  
Thursday Evening, November 30, 2000  
8:00 PM

Exhibition Hall D (Hynes)

#### II10.1

PREPARATION OF HIGHLY TEXTURED IBAD-YSZ BUFFER LAYERS ON LARGE METALLIC TAPES. Sibylle Sievers<sup>1</sup>, Jürgen Dzick<sup>1,2</sup>, Jörg Hoffmann<sup>1</sup>, Alexander Usoskin<sup>2</sup>, Herbert C. Freyhardt<sup>1,2</sup>. <sup>1</sup> Institut für Materialphysik, Universität Göttingen,

GERMANY. <sup>2</sup> Zentrum für Funktionswerkstoffe GmbH, Göttingen, GERMANY.

Biaxially textured yttria-stabilized zirconia (YSZ) buffer layers were deposited on long polycrystalline metallic tapes (up to 1 m length) by an ion-beam-assisted deposition process (IBAD) to serve as templates for high-current-carrying  $\text{Y}_1\text{Ba}_2\text{Cu}_3\text{O}_{7-x}$  (YBCO) films. The installation for large-area deposition of YSZ films is equipped with two 11 cm Kaufman ion sources. The usage of large diameter ion beams provides a number of characteristic features to the deposition process. Neither the deposition rate nor the current density of the assisting ion beam are spatially homogenous. Furthermore, the intrinsic divergence of the IBAD source gets more influential on large-area substrates. Therefore, the spatial distribution of the orientation and quality of the in-plane texture reflects the actual performance of the ion sources. The coating of large substrates requires in addition substrate movements to produce YSZ films of homogeneous texture quality. The movements lead to an averaging over various deposition conditions. Fast substrate motion does not allow, to accurately control the substrate temperature. Investigations on the temperature dependence of the IBAD process and its resulting in-plane alignment reveal a pronounced temperature variation. The best results are obtained for room temperature depositions. To counteract the significant rise of the temperature of small-heat-capacity substrates due to a high energy input during the deposition process, a high tape velocity can be applied that effectively prevents excessive heating of the substrate. Therefore, it is possible to deposit YSZ films on metal tapes with in-plane textures as good as  $15^\circ$  FWHM at  $1 \mu\text{m}$  film thickness. The high quality of such tapes is also confirmed by depositing YBCO films onto them. On a tape (10 mm  $\times$  500 mm) with an average in-plane texture of  $18.3^\circ$  FWHM the YBCO exhibits within 10% an average critical current density  $J_c$  at 77 K and 0 T of  $0.6 \text{ MA}/\text{cm}^2$ .

#### II10.2

STRUCTURAL AND ELECTRICAL PROPERTIES OF  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  THIN FILMS ON BIAXIALLY TEXTURED BUFFERED Ni-BASED ALLOY SUBSTRATES. M. Li, B. Ma, Y.A. Jee, B.L. Fisher and U. Balachandran, Argonne National Laboratory, Argonne, IL.

Oxide high-Tc superconducting wires and tapes with high critical current density  $J_c$  are essential in power applications. Recently,  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (YBCO) thin films grown on Ni-based alloy tapes have attracted intense interest because of their promise for these applications. In these studies, buffer layers were necessary to obtain biaxially aligned YBCO thin films with high  $J_c$ . In our studies, yttria-stabilized zirconia,  $\text{CeO}_2$  and MgO buffer layers were deposited on an Ni-based alloy substrate by inclined substrate deposition, ion-beam assisted deposition, and pulsed laser deposition (PLD) to examine the influences of these buffer layers. YBCO thin films were deposited on these buffered metallic substrates by PLD under optimized conditions. The orientation and in-plane textures of YBCO and the buffer layers were characterized by X-ray diffraction  $q/2q$  scan,  $f$ -scan, and pole figure analysis. The superconductive transition features were examined by measuring inductive  $T_c$  and transport  $J_c$ . Details of this study will be presented.

This work is supported by the U.S. Department of Energy(DOE), Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, under Contract W-31-109-Eng-38.

#### II10.3

RECRYSTALLIZATION TREATMENT OF Ni ALLOY SUBSTRATES FOR YBCO COATED CONDUCTORS. Rainer Nast, Wilfried Goldacker, Bernhard Obst, and Wolfgang Schauer, Forschungszentrum Karlsruhe, Institut fuer Technische Physik, Karlsruhe, GERMANY.

For mechanically stabilized Ni alloy substrate tapes the commonly used high recrystallization temperatures and secondary recrystallization has to be avoided. Being a nucleation-growth process, the kinetics of the cubic recrystallization is strongly temperature dependent and the activation energy correlates with the stacking fault energy (SFE) of the specific alloy. In combination with investigations of the cubic phase formation kinetics, the texture quality of Ni (99,99 wt.%), NiCr<sub>10</sub> and NiMn<sub>10</sub> tapes in dependence of pre-heating, recrystallization temperature and time was investigated.

Pre-annealing of the starting material before rolling affects the deformation state and so the recrystallization behavior. Large grains with worse cubic orientation are observed. Increasing recrystallization temperature and time sharpens the texture due to grain growth, limited by occurring secondary recrystallization exceeding a sample specific temperature. In binary alloyed Ni systems each alloying element lowers the SFE. This favors the formation of recrystallization twins with the orientation  $\{122\}\langle 212\rangle$ , which obstructs the biaxially textured coating. In Mn alloyed tapes this effect is strongly pronounced. The tapes show a good cubic texture, however also a

high volume fraction of twins due to the reduction of the SFE. To solve this problem we investigated ternary Ni based alloys with respect to possibly increased SFE and report first results.

#### II10.4

DISSIPATION AND ANISOTROPY IN ULTRATHIN  $\text{YBa}_2\text{Cu}_3\text{O}_7/\text{PrBa}_2\text{Cu}_3\text{O}_7$  SUPERLATTICES. J. Villegas, E. M. Gonzalez, J.L. Vicent, Dpto Fisica Materiales, Facultad CC. Fisicas, Universidad Complutense, Madrid, SPAIN; Z. Sefrioui, J. Santamaria, Depto. Fisica Aplicada III, Facultad CC. Fisicas, Universidad Complutense, Madrid, SPAIN; M. Varela, Dpto. Fisica, Universidad Carlos III, Leganes, SPAIN.

Ultrathin superlattices of 123 superconducting cuprates have been fabricated by high Oxygen pressure sputtering technique. The superlattices show high quality with negligible step disorder, roughness and interdiffusion (1). In the superlattices used in this work the insulating (PBCO) layer thickness has been kept constant at 5 unit cells (u.c.) and the superconducting (YBCO) layer thicknesses have been changed from 8 u.c. up to 1 u.c. The critical temperature changes from 44 K (1 u.c. YBCO) to 80 K (8 u.c. PBCO). A computer controlled rotatable sample holder is used to study the dissipation anisotropy with applied magnetic fields up to 9 T and temperatures above 0.7 T/T<sub>c</sub>. At constant temperature, the angular dependence resistivity scales down with the applied magnetic field values, in all the temperature range. This behavior allows us to study the anisotropy and dimensionality behavior of the mixed state in ultrathin superlattices. A dimensional crossover is found in the anisotropy with the 1 u.c. YBCO superlattices following a pure 2D behavior, exactly the same than in BSCCO films. A detail study and a comparison with the anisotropy 2D data taken from BSCCO films will be presented. (1) M. Varela et al. Phys. Rev. Lett. 83, 3936 (1999).

#### II10.5

THICKNESS DEPENDENCE OF CRITICAL CURRENT DENSITY OF YBCO ON ROLLING ASSISTED BIAXIALLY TEXTURED SUBSTRATES. B.W. Kang, A. Goyal, D.F. Lee, M. Paranthaman, D.M. Kroeger, P.M. Martin, and D.T. Verebelyi, Oak Ridge National Laboratory, Oak Ridge, TN.

High-current-carrying capability is one of the most important factors in the development of high temperature superconducting (HTS) tapes for electric power applications. One approach to fabricating high-current superconducting tapes involves the epitaxial growth of oxide buffer layers and YBCO films on biaxially textured metal substrates. It has been found that high critical current density ( $J_c$ ) over 1 MA/cm<sup>2</sup> can be achieved only up to a certain thickness of YBCO. We have investigated the thickness dependence of  $J_c$  of YBCO films grown by pulsed laser ablation on the Rolling-Assisted Biaxially Textured Substrates (RABiTS). The thickness of YBCO films varied from 0.2  $\mu\text{m}$  to 3  $\mu\text{m}$ .  $J_c$  of as high as 0.5 MA/cm<sup>2</sup> has been achieved in 3  $\mu\text{m}$  thick YBCO film. These results were compared to those of YBCO films on single crystal substrates. Further characterizations of the samples such as X-ray  $\theta$ -2 $\theta$ ,  $\omega$  and  $\phi$  scan, and cross section SEM analysis were performed. Possible cause of the degradation of  $J_c$  on thick films will be discussed.

#### II10.6

DEVELOPMENT OF 90° OFF-AXIS RF-MAGNETRON SPUTTERING FOR THE FABRICATION OF YBCO/PBCO-BASED SNS JOSEPHSON JUNCTIONS ON YTTRIA-STABILIZED ZIRCONIA SUBSTRATES. B.J. Gibbons, D.W. Reagor and Q.X. Jia, Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM.

The development of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  (YBCO)-based SNS Josephson junction technology has always hinged upon the use of very expensive single crystal substrates such as  $\text{LaAlO}_3$ ,  $\text{SrTiO}_3$ ,  $\text{MgO}$ , and many others. There has not been as much focus on the use of the comparatively inexpensive yttria-stabilized zirconia (YSZ) as a substrate for these devices. One of the drawbacks to using YSZ is the formation of multiple in-plane orientations of the YBCO thin film which can detrimentally affect the superconducting properties. Along with this, the YBCO (or PBCO) and the YSZ react to form an epitaxial interlayer of  $\text{BaZrO}_3$ . To get around these effects, a  $\text{CeO}_2$  buffer layer can be used between the YBCO/PBCO and the YSZ. However, in this case  $\text{BaCeO}_3$  can be formed as precipitates on the resulting film. In this work, we will discuss the growth mechanisms of the YBCO and PBCO films on the  $\text{CeO}_2$ /YSZ substrate and the minimization of  $\text{BaCeO}_3$  formation. Using a PBCO/ $\text{CeO}_2$  buffer layer, we have achieved YBCO films with  $T_c$  of 90 K and 0.3 K widths. In addition, the use of ozone in the fabrication process will be discussed. Finally, data will be presented for devices fabricated on YSZ.

#### II10.7

FABRICATION OF LARGE GRAIN RE-Ba-Cu-O (RE = Gd, Dy) SUPERCONDUCTORS AND THEIR TRAPPED MAGNETIC

FIELDS. Shinya Nariki, Naomichi Sakai, Masato Murakami, Superconductivity Research Laboratory, ISTEK, Tokyo, JAPAN.

Large single domain RE-Ba-Cu-O (RE: rare earth element) superconductors are promising candidates for practical applications such as levitation and quasi permanent magnets with high trapped magnetic fields. In the present work, we fabricated large single domain Gd-Ba-Cu-O and Dy-Ba-Cu-O bulk superconductors and measured their superconducting and field-trapping properties. Large c-axis oriented Gd-Ba-Cu-O bulk superconductors with Ag addition were melt-textured under controlled oxygen partial pressure of 1.0%. Nd123 was used as a seed crystal for the growth of large grain bulk. Single domain Gd-Ba-Cu-O samples 32 mm and 48 mm in diameters were successfully fabricated with no macro-sized cracks. The size of 211 particles dispersed in the bulk was reduced to 0.7-0.8  $\mu\text{m}$  with the employment of fine Gd211 starting powders. As a result, large critical current density of 70000 A/cm<sup>2</sup> was achieved throughout the sample in self-field at 77 K. The maximum value of trapped magnetic field reached 1.5 - 2.0 T at 77 K. These values far exceed those of Y-Ba-Cu-O with similar sizes. We also fabricated a single domain Dy-Ba-Cu-O bulk with the top-seeded melt-growth process in air. The trapped magnetic field of the sample 32 mm in diameter exhibited a high value of 1.4 T at 77 K.

This work was supported by the New Energy and Industrial Technology Development Organization (NEDO) as Collaborative Research and Development of Fundamental Technologies for Superconductivity Applications.

#### II10.8

Nd/Ba SUBSTITUTION AND OXYGEN DEFICIENCY EFFECTS ON THE PINNING PROPERTIES OF  $\text{NdBa}_2\text{Cu}_3\text{O}_{7-\delta}$  SINGLE CRYSTALS. S. Shibata, Y. Feng, A.K. Pradhan, T. Machi, K. Nakao and N. Koshizuka, Superconductivity Research Laboratory, International Superconductivity Technology Center, Tokyo, JAPAN.

$\text{NdBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (Nd123) crystal is known to have a peak effect in magnetization hysteresis curves at 77K. The cause of this peak effect is supposed to be due to the presence of local oxygen deficiencies or Nd/Ba substitution in the Nd123 matrix. In order to investigate the detail, it is necessary to clear the roles of the two pinning centers. Nd123 single crystals were prepared under partial O<sub>2</sub> pressures (PO<sub>2</sub>) of 0.04~1%. All of the as-grown single crystals were annealed at 325°C, for 14 days in O<sub>2</sub> atmosphere to fully oxidize the crystals. After that these crystals were heated in air for 2.5 hours at 365, 380 and 400°C, respectively and quenched for giving rise to oxygen vacancies in these crystals. As for the fully oxygenated crystals, the oxygen vacancy concentration is thought to be very low, and we found that T<sub>c</sub>(on set) increased slightly with lowering PO<sub>2</sub> and reached ~96K for PO<sub>2</sub>=0.04%. On the other hand, the normalized peak field  $h_p$ (=H<sub>p</sub>/H<sub>irr</sub> where H<sub>p</sub> is the magnetic field at which pinning force density  $F_p$ (=J<sub>c</sub>xB) is maximum, and H<sub>irr</sub> is the irreversibility field) has the maximum around PO<sub>2</sub>=0.13%. By heat treatments at higher temperatures, T<sub>c</sub> of all sample decreased. The higher PO<sub>2</sub> and heating temperatures, the more decreasing values of T<sub>c</sub> and quenching. In the case of heating temperature of 380°C, DT<sub>c</sub>=1.3K for PO<sub>2</sub>=0.04% and DT<sub>c</sub>=6.3K for PO<sub>2</sub>=1%.  $h_p$  decreased with increasing PO<sub>2</sub> namely with increasing Nd/Ba substitution. By moderate heating,  $h_p$  and  $F_p$  values of the sample grown under PO<sub>2</sub>=0.04% increased, but this  $F_p$  value was only about one fifth of  $F_p$  of the fully oxygenated sample grown under 0.13%PO<sub>2</sub>. This fact indicates that is impossible to obtain large pinning force with only introducing oxygen vacancies. It seems to be important to control the amount of Nd/Ba substitution. This work was supported by NEDO.

#### II10.9

SILVER DISTRIBUTION IN ISOTHERMAL SOLIDIFICATION OF  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}\text{Ag}$  COMPOSITE. C. Cai and H. Fujimoto, Railway Technical Research Institute, Kokubunji-shi, Tokyo, JAPAN.

The melting solidification of  $\text{REBa}_2\text{Cu}_3\text{O}_y$  system with proper content of silver addition undergoes a monotectic-peritectic reaction. Due to lower melting temperature, the silver exists as second liquid phase before and after the peritectic solidification of RE123. Present work studies the trapping process of second heterogeneous inclusion, i.e. silver liquid phase, and compares with that of first heterogeneous inclusion, i.e. Y211 solid particle. The dependencies of trapped Ag particle size on undercooling degree and maximum processing temperature are studied. Similar to high-temperature stable Y211, the silver trapping mechanism conforms to Uhlmann-Chalmers-Jackson theory, and the silver can not act as heterogeneous nucleation of RE123. However, unlike Y211, the ejecting and free trapping phenomena frequently occur for silver.

#### II10.10

ELECTRIC CONDUCTION OF  $\text{Nd}_{1-x}\text{Sm}_x\text{BaSrCu}_3\text{O}_{7-y}$  SOLID SOLUTIONS. Alexander A. Savitsky, Nikolay N. Turko, Bel State

Univ, Dept of Phys Chem, Minsk, BELARUS; Andrey I. Klyndyuk, Gennady S. Petrov, Dept of Phys and Col Chem, Minsk, BELARUS.

We carried out systematic study of DC electric conduction (by four-probe method) of polycrystalline samples of solid solutions  $\text{Nd}_{1-x}\text{Sm}_x\text{BaSrCu}_3\text{O}_{7-y}$  ( $x = 0.0-1.0$ ) (I) as well as similar solid solutions (II), having alkaline-earth elements deficit in comparison with 123 phase, at 293-1073 K at different partial pressures of oxygen (oxygen, air, argon). The synthesized samples of monophase solid solutions (I) had orthorhombic structure, their unit cell parameters and oxygen nonstoichiometry index ( $7-y$ ) decreased at substitution of barium by strontium. It is interesting to note, that for deficient (II), having  $\text{NdBaSr}_{0.6}\text{Cu}_3\text{O}_{7-y}$  and  $\text{Nd}_{0.75}\text{Sm}_{0.25}\text{BaSr}_{0.6}\text{Cu}_3\text{O}_{7-y}$  composition, the above structure was also found and their unit cell parameters were closed to those for undeficient (I). Dependence electric conduction on composition  $x$  of (I) had unmonotonous character, maximum value of electric conduction (about 100 S/cm at room temperature) was found for  $\text{SmBaSrCu}_3\text{O}_{7-y}$ ; conduction decreased at partial pressure of oxygen reduction. At about 700-750 K significant decrease of electric conduction for (I) took place; this fact was due to the structural phase transition of samples, which was accompanied by evolution of oxygen. For (I) electric conduction had metallic-type character in oxygen and in air. In argon for (I) samples, having constant oxygen content, conduction had semiconducting character and its values decreased at reduction of oxygen content in the samples. The values of apparent activation energy of electric conduction of (I) in argon, calculated from arrhenius plots, were about 2-16 kJ/mol, they practically did not depend on cationic composition, but significantly increased at oxygen nonstoichiometry index ( $7-y$ ) decrease. Note, that decrease of strontium content in samples (II) leads to the change of electric conduction (value, character) in comparison with (I). So, for  $\text{NdBaSr}_{0.6}\text{Cu}_3\text{O}_{7-y}$  electric conduction in oxygen and in air at low temperatures had semiconducting character, conduction value was larger than electric conduction for  $\text{NdBaSrCu}_3\text{O}_{7-y}$ .

#### II10.11

Abstract Withdrawn.

#### II10.12

SIMULATION AND EXPERIMENTAL DETERMINATION OF THE SPUTTERING AND TRANSPORT PROCESS DURING DEPOSITION OF MULTI-COMPONENT, OXIDE MATERIALS.

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Sputter deposition of complex oxide films - e.g. ceramic superconducting or ferroelectric films - require a reactive gas or gas mixture (e.g. oxygen and argon) at rather high pressures of typically 10-100 Pa. In this pressure regime the sputtering process at the stoichiometric target, the material transport from target to the substrate and the resulting stoichiometric distribution at the substrate depend strongly on the exact pressure, gas mixture, cathode-substrate distances and sputtering rate. In this contribution sputter process and material transport are simulated and experimentally analysed for  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  and  $(\text{Ba,Sr})\text{TiO}_3$  deposition via magnetron sputtering technique. The simulation is based on a Monte-Carlo algorithm. The transport of sputtered atoms in the cathode-substrate space are exposed to well known laws of sputtering processes and physics of atom-nuclear collisions. The simulation of large-area deposition demonstrates, that a reduction of the gas pressure from  $>100$  Pa for high-pressure diode sputtering to  $\sim 40$  Pa (high-pressure magnetron) and, finally, 1-4 Pa (magnetron) is necessary in order to achieve a homogeneous and stoichiometric film growth at a reasonable deposition rate. Experimentally the pressure reduction has been realised by a modification of the magnetron, the plasma stability is monitored by Langmuir probes, composition and deposition rate of the resulting ceramic layers are determined. The experimental data show a good agreement with the computer simulation for sputter yield and material transport of multi-component superconducting and ferroelectric oxides.

#### II10.13

VARIABLE SUBGRAIN BOUNDARY DISLOCATION NETWORKS IN SINGLE DOMAIN MELT TEXTURED  $\text{YBa}_2\text{Cu}_3\text{O}_7$ . IMPLICATIONS ON CRITICAL CURRENTS. F. Sandiumenge, Institut de Ciencia de Materials de Barcelona, CSIC, Campus de la UAB, SPAIN; J. Rabier, Laboratoire de Metallurgie Physique, Universite de Poitiers, FRANCE; X. Obradors, Institut de Ciencia de Materials de Barcelona, CSIC, Campus de la UAB, SPAIN.

Single domain melt textured  $\text{YBa}_2\text{Cu}_3\text{O}_7$  (123) materials are plagued by subgrain boundaries. Transmission electron microscopy observations reveal that the microstructure of such boundaries, involving rotation angles,  $\theta \sim 1^\circ$ , possesses many features reported for macroscopic growth induced grain boundaries. Periodic interfacial

steps and facetting, occurring on length scales ranging from a few to some tens of nanometers, are frequently found to cause distortions in the otherwise homogeneous underlying dislocation network. However, unlike macroscopic growth induced ones, they are likely to built up under complex stress patterns and, as a consequence, their dislocation networks display a variety of configurations. Dislocation densities are calculated for the general case using the generalized Frank's formula. According to this geometric scheme, for a fixed  $\theta$ , the dislocation core overlap model predicts a substantial depression of  $J_c$  when the boundary geometry deviates from a symmetrical tilt. Our results strongly suggest that subgrain boundaries may contribute significantly to the gap existing between maximum  $J_c$  values achieved to date in single domain bulk samples and thin films.

#### II10.14

LARGE HTS MONOLITHS WITH COMPLEX SHAPES PREPARED BY WELDING AND MULTI-SEEDING TECHNIQUES. Heribert Walter, Zentrum fuer Funktionswerkstoffe gGmbH, Goettingen, GERMANY; Christian Jooss, Boris Brिंगmann, Marie-Pierre Delamare, Institut fuer Materialphysik, Universitaet Goettingen, GERMANY; Andreas Leenders, Herbert C. Freyhardt, Zentrum fuer Funktionswerkstoffe Goettingen GmbH, Institut fuer Materialphysik, Universitaet Goettingen, Goettingen, GERMANY.

Many technical applications, i.e. magnetic bearings for flywheels and rotors for superconducting motors, require large and high-quality bulk HTS. Single-domain YBCO monoliths with diameters up to 70 mm are fabricated by the Top Seeded Melt Growth (TSMG) method. However, larger components of complex shapes can be processed by using more than one seed crystal (multi-seeding) or by appropriately joining YBCO monoliths. Joining of two samples leads to the formation of artificial grain boundaries. It requires a high connectivity between two adjacent domains in order to reach a high intergranular current density and sufficient mechanical strength. This is possible by bonding two single domains with  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-x}$  25 mol%  $\text{Er}_2\text{O}_3$  as joining agent. Magneto-optical measurements show local values of the intergranular critical current densities of up to  $10^9$  A/m<sup>2</sup>, which has to be compared to the  $1.3 \cdot 10^9$  A/m<sup>2</sup> in the adjacent bulk monodomain tiles.

Furthermore, the multi-seeding technique can be employed to produce large superconducting tiles of complex shapes. For example, a superconducting ring (dimensions:  $\Phi_a = 110$  mm,  $\Phi_i = 75$  mm,  $h = 28$  mm) was prepared by twelve-fold seeding, with a radial orientation of the c-axis. Similar rings will be used as magnetic bearings for a liquid hydrogen tank. Rings were melt textured with different spacings between the seeds, which also determined the angular orientation between the domains, to obtain precipitate-free grain boundaries and to improve the trapped field, respectively. The microstructure of the grain boundaries are characterized by optical and electron microscopy. The influence of artificial and natural grain boundaries on the superconducting properties were determined by transport critical current density, trapped field and magneto-optical measurements.

#### II10.15

CHARACTERIZATION OF SUPERCONDUCTING  $\text{YBa}_2(\text{Cu}_{3-x}\text{Au}_x)\text{O}_7$  and  $\text{YSr}_2(\text{Cu}_{3-x}\text{Au}_x)\text{O}_7$  USING HIGH PRESSURE SYNTHESIS. Brian R. Hickey, Luiz Marcos Dezaneti, D. Kent Ross, Irene A. Rusakova, James K. Meen and Don Elthon, Dept of Chemistry and Texas Center for Superconductivity at the University of Houston, TX.

In this study, we are investigating the structure-property relationships of superconducting YBCO and YSCO as a function of Au-doping in the charge reservoir block layer. We have synthesized under high pressure (30 kbar) and high temperature (1050°C) Au-doped samples of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  and  $\text{YSr}_2\text{Cu}_3\text{O}_7$ . We characterized the compounds using powder x-ray diffraction, electron microprobe analysis, and transmission electron microscopy (TEM), while the  $T_C$  was measured magnetically and resistively. Previous studies on Au incorporation into YBCO also required Ca-substitution for Y, which is a doping of the active block layer, in order to achieve superconductivity. Our results indicate that Au dopes into the compounds as a solid solution. For  $x = 0.6$ , YBCO has a  $T_C = 43$  K. This is consistent with previous studies on Au-doped YBCO. However, while the parent compound of pure YSCO has a  $T_C = 60-64$  K, it has been previously determined to be an oxygen-overdoped compound. We observed for  $x = 0.6$  on YSCO that  $T_C = 80$  K, indicating that the compound is closer to the optimal doping point. TEM results indicate that Au-doped YBCO has a primitive tetragonal structure with lattice parameters  $a = 3.94$  and  $c = 12.01$  Å.

#### II10.16

TRAPPED MAGNETIC FIELD OF A MINI - BULKMAGNET USING  $\text{YBaCuO}$  SUPERCONDUCTORS. Hiroyuki Fujimoto, and Hiroki Kamijo, Railway Technical Research Institute, Kokubunji-shi, Tokyo, JAPAN.

It is well known that melt-processed YBaCuO (Y123) or Rare Earth (RE)123 superconductors and NdBaCuO (Nd123) or (LRE)123 superconductors have a high  $J_c$  at 77 K and high magnetic field. Solidification processes for producing (L)RE123 superconductors are effective for obtaining high  $J_c$ , leading to high field application as a superconducting quasi-permanent bulk magnet with the liquid nitrogen refrigeration. One of the promising applications is a superconducting magnet for the magnetically levitated (Maglev) train. We fabricated a mini-superconducting bulk magnet of 200 mm x 100 mm, consisting of 18 bulks which are a square 33 mm on a side and 10 mm in thickness, and magnetized the mini-magnet by field cooling. The mini-magnet showed the trapped magnetic field of larger than 0.1 T on the surface of the outer vessel of the magnet. The present preliminary study discusses trapped magnetic field properties of the mini-bulkmagnet using YBaCuO superconductors at 77K.

#### II10.17

INTERACTION THERMODYNAMICS OF OXYGEN IONS AND VACANCIES IN THE  $Nd_{1-y}Ba_{2-y}Cu_3O_z$  SOLID SOLUTION.  
Eugene A. Trofimenko<sup>1</sup>, Nikolay N. Oleynikov<sup>2</sup>, Eugene A. Goodilin<sup>2</sup>, Yury D. Tretyakov<sup>2</sup>. <sup>1</sup>Faculty of Matls Sci, Moscow State Univ. <sup>2</sup>Dept of Chemistry, Moscow State Univ, Moscow, RUSSIA.

This work is devoted to analysis of oxygen absorption by Nd123 based solid solution from a thermodynamic point of view. Thermodynamic evaluation of the system  $Nd_{1-y}Ba_{2-y}Cu_3O_z - O_2$  ( $x=0-0.6$ ) was carried out using reference data obtained by Lindemer et al. The thermodynamic features were discussed in terms of solution of oxygen ions and vacancies in the  $CuO_x$  layer. Oxygen absorption was considered as a sequence of processes such as (1) oxygenation of a compound with minimal oxygen content  $z$  up to its maximal  $z$  and (2) mixing of terminal compounds e.g. oxygen ions and vacancies in Nd123ss with low and high oxygen content. The last one leads to a non-stoichiometric Nd123ss phase with intermediate oxygen content. Calculated activities of terminal phases via oxygen content allowed us to account an excess free energy of mixing and to consider it in the frame of regular solution model. It was found that a mean free energy of mixing is negative and decreases linearly with the substitution degree  $x$ , indicating either growth of Cu-O interaction potential (at lower Cu-O distance) or formation of different type environment of an oxygen site. It was also found that the pure Nd123 phase exhibit only the regular-like dependence of its free energy of mixing on composition. For substituted solid solutions the behaviour of free energy of mixing was found to be different for low- and high- oxygen content intervals. This has led to partition of  $\Delta G^E_{mix}$  into three contributions (free energies of: (1) a specific compound formation in the middle of the oxygen nonstoichiometry interval and (2,3) mixing it with terminal compounds). This allowed us to treat the changes of mixing energy. These changes are discussed with respect to structural changes of investigated solid solutions. An evidence of lowering energy of oxygen sites formation with substitution was derived.

#### II10.18

ON THE PHYSICO-CHEMICAL PROPERTIES OF  $Y_{2-x}Me_x$  BaCuO<sub>5</sub> (Me - Bi, Cd, Ca) SOLID SOLUTIONS.  
Andrey I. Klyndyuk, Ekaterina S. Bugrina, Gennady S. Petrov, Leonid A. Bashkurov, Bel State Technol Univ, Dept of Phys and Col Chem, Minsk, BELARUS.

It is known, that so-called "green phase" ( $211, Y_2BaCuO_5$ ) may be considered as precursor for synthesis of bulk samples of  $YBa_2Cu_3O_{7-y}$  (123) superconductor, having high current density ( $j_c$ ), as substrate in preparation of films of 123-type superconductors etc. But physicochemical data for other such phases and their solid solutions are sparse, though complex study of these solid solutions may be useful both for manufacturing of superconducting devices and for better understanding of influence of cationic composition on the properties of similar non-superconducting as well as respective superconducting phases. The aim of this paper is investigation of influence of yttrium substitution by bismuth, cadmium or calcium on crystal structure, thermal expansion (293-1173 K, air) and electrical conductivity (dc and ac, 293-1173 K, air) of  $Y_{2-x}Me_xBaCuO_5$  (Me - Bi,  $x = 0-1$  (I); Cd (II),  $x = 0-0.5$ ; Ca (III),  $x = 0-0.25$ ) solid solutions. Polycrystalline samples of (I-III) were prepared by ceramic method in air at 1173-1273 K. X-ray phase analysis showed, that samples were monophasic and had structure similar to  $Y_2BaCuO_5$ , though for  $x > 0.35$  in (I) the structure was more simple and similar to  $Nd_2BaCuO_5$ -structure ("brown phase") and these samples had brown colour. The unit cell parameters of (I-III) were close to the parameters of unsubstituted phase. The linear thermal expansion coefficient value for (I) slightly decreased, but for (II) it slightly increased with decreasing of yttrium content. It was found, that electrical conductivity of (I-III) at high temperatures was higher, but at low temperatures for all samples with  $x=0.01$  it was less than that of unsubstituted "green phase". The values of apparent activation energy of electrical conductivity, calculated from arrhenius plots, for (II) decreased linearly with increase of cadmium content.

### SESSION III1: SUPERCONDUCTORS OF THE 1212- AND 1223-TYPE CONTAINING Tl, Hg, OR Ru

Chairs: Raghu N. Bhattacharya and Dezhi Wang

Friday Morning, December 1, 2000

Room 306 (Hynes)

#### 8:30 AM III1.1

SYNTHESIS AND CHARACTERIZATION OF THALLIUM-BASED 1212 FILMS. J.Y. Lao, D.Z. Wang, S.X. Yang, Y. Tu, H.L. Wu, J.G. Wen, Z.F. Ren, Boston College, Department of Physics, Chestnut Hill, MA; J.H. Wang, SUNY at Buffalo, Department of Chemistry, Buffalo, NY; D.K. Christen, Oak Ridge National Laboratory, Division of Solid State, Oak Ridge, TN; R.N. Bhattacharya, R.D. Blaugher, National Renewable Energy Laboratory, Golden, CO.

Epitaxial (Tl,Bi)Sr<sub>2</sub>Cr<sub>0.15</sub>Ca<sub>0.85</sub>Cu<sub>2</sub>O<sub>y</sub> [(Tl,Bi)-1212] films have been successfully grown on both LaAlO<sub>3</sub> and CeO<sub>2</sub>/YSZ substrates by post-deposition annealing in either static air or flowing argon gas. These superconducting Tl-based, 1212-structured films on LaAlO<sub>3</sub> substrates have T<sub>c</sub> values in the range of 94 - 100 K, and J<sub>c</sub> values over 1.5 mega A/cm<sup>2</sup> at 77K. The films are predominately 1212 phase, with excellent epitaxy as determined by XRD theta - 2 theta, omega and phi scans. The films on CeO<sub>2</sub>/YSZ substrates have T<sub>c</sub> values in the range of 94 - 96 K, J<sub>c</sub>(77K) values close to 0.6 mega A/cm<sup>2</sup> and excellent epitaxy on the CeO<sub>2</sub> buffer layers.

#### 8:45 AM III1.2

SUPERCONDUCTING THALLIUM OXIDE FILMS. Raghu N. Bhattacharya, Richard Blaugher, National Renewable Energy Laboratory, Golden, CO; Yao-Te Wang, Allen Herman, CU Boulder, CO; Huailing Wu, Jun Chen, Zhifeng Ren, Boston College, MA.

Electrodeposition and spray pyrolysis offer a promising and economic approach for fabricating high-temperature superconducting (HTS) wire and tape for the Tl-oxide superconductors. The Tl-oxides, 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 single layer compound with Pb, Bi, and Sr substitution. Recent advances in the development of Tl-oxide superconductors on Ag-Pd alloy and rolled-textured Ni substrates will be discussed. At present we are making two layers electrodeposited films to increase the film thickness and also the uniformity of the films. The morphology for the electrodeposited materials is a very important step of the electrogrowth because it influences directly the structure of the annealed film and, therefore, its properties. In the electrodeposition process, the adatoms or adions incorporate in the substrate. With time, as the film thickness increases, the deposition continues either by the buildup on previously deposited material (old nucleation centers) or the formation and growth of new ones. These two processes are in competition and can be influenced by different factors. The two-layer technique, where a new Ag layer was deposited in-between, helped to fill up the voids by creating new nucleation centers, which improved the film morphology. The two-layer, 2.6 micron thick (width = 3.2 mm) Tl-oxide superconductor film by the electrodeposition process showed transport critical current of 28.24 A at 77 K (normalized transport critical current = 88.25 A for 1 cm wide samples). At 77 K and no magnetic field, the critical current density value of a two-layer, 0.8 micron thick film is  $1.1 \times 10^6$  A/cm<sup>2</sup>.

#### 9:00 AM III1.3

STRUCTURAL STUDIES OF DIFFERENTLY DOPED Tl<sub>2</sub>Ba<sub>2</sub>CuO<sub>y</sub> FILMS. Jianguo Wen, Dezhi Wang, Shaoxian Yang, H.I. Ha, M.J. Naughton, Z.F. Ren, Department of Physics, Boston College, Chestnut Hill, MA.

Tl<sub>2</sub>Ba<sub>2</sub>CuO<sub>y</sub> (Tl-2201) films are used for many basic studies such as determination of pairing symmetry, pseudogap, upper critical field, etc., because of the structural simplicity of only one CuO<sub>2</sub> plane in each unit cell in comparison with Bi-2212 and YBCO and of the continuous control of doping level. Previous TEM study on symmetry of Tl-2201 of optimal-doped films showed they are tetragonal, which is important for studying pairing symmetry. Recently, extremely over-doped Tl-2201 films with low T<sub>c</sub> have been obtained by an off-axis configuration deposition. Here, we report the structural studies of Tl-2201 films with different doping levels prepared by on- and off-axis configurations using transmission electron microscopy and convergent-beam electron diffraction. A modulated structure in the bc plane was observed in over-doped Tl-2201 films. The modulated structure becomes weak from over-doped to optimal doped regimes and finally disappears in optimal doped films. Convergent-beam electron diffraction study on the symmetry indicates that over-doped films are orthorhombic while optimal-doped films are tetragonal. A twin-like structure was also observed in the over-doped films, resulting from a direction change of modulation. The symmetry breaking is found due to the modulated structure, which depends on oxygen content in films. The influence of deposition configuration on

modulated structure and symmetry of films is also studied. It was found that off-axis deposition reduced the symmetry of films, which may result from an inhomogeneous distribution of cations in films.

#### 9:15 AM I11.4

SYNTHESIS OF HEAVILY OVERDOPED  $Tl_2Ba_2CuO_6$  THIN FILMS WITH VERY LOW  $T_c$ . D.Z. Wang, H.I. Ha, S.X. Yang, J.G. Wen, J.I. Oh, J. Moser, M.J. Naughton and Z.F. Ren, Boston College, Dept of Physics, Chestnut Hill, MA.

Heavily overdoped epitaxial superconducting  $Tl_2Ba_2CuO_6$  d (Tl-2201) thin films with single transition and very low zero resistance  $T_c$  ( $< 5K$ ) have been successfully synthesized. The films were made by off-axis RF sputtering deposition and post-deposition heat-treatment of slow-cooling. The investigations showed that the single transition was contributed to off-axis sputtering deposition, and the low  $T_c$  to post-deposition heat-treatment of slow-cooling. TEM investigations revealed that the films are pure single Tl-2201 phase with good crystallinity. Such samples are entirely appropriate and being measured for detailed investigations of  $Hc_2$  using conventional laboratory superconducting magnets

#### 10:00 AM I11.5

SUPERCONDUCTIVITY AND MAGNETIC PROPERTIES OF HIGH-PRESSURE OXYGEN SYNTHESIZED  $Ru_{1-x}Sr_2GdCu_{2x}O_{8-z}$  COMPOUNDS. P.W. Klamut, B. Dabrowski, J. Mais, M. Maxwell, Department of Physics, Northern Illinois University, DeKalb, IL.

We report on the new superconducting materials with the formula  $Ru_{1-x}Sr_2GdCu_{2x}O_{8-z}$  that have been successfully synthesized under high-pressure oxygen conditions. By changing the ratio between Ru and Cu in the compound we were able to increase the temperature of the superconducting transition up to 70K for  $x=0.3$ . The temperature of the magnetic transition of Ru sublattice decreases from 132K for  $x=0$  to 100K for  $x=0.5$ . The magnetic characteristics of these compounds are discussed and compared to the existing data on  $RuSr_2GdCu_2O_8$  magnetic superconductor ( $T_c^{on}=50K$ ,  $T_N=132K$ ). Work supported by the ARPA/ONR, and by the State of Illinois under HECA.

#### 10:15 AM I11.6

Ca/Sr & Ho/Gd SUBSTITUTIONS IN  $RuSr_2GdCu_2O_8$ . R. Ruiz-Bustos, J.M. Gallardo, E. Moran, R.Saez-Puche and M.A. Alario-Franco. Laboratorio Complutense de Altas Presiones, Facultad de Ciencias Químicas, Universidad Complutense, Madrid, SPAIN.

One of the most intriguing materials derived from  $YBaCuO_7$  (Ybco) is, undoubtedly, the ferromagnetic superconductor  $RuSr_2GdCu_2O_8$ , in which the charge reservoir layer is composed of [Ru-O6] octahedra, the remaining of the structure being essentially identical to Ybco. It appears that the Ru atoms, which seem to be in the V oxidation state, order ferromagnetically below a  $T_{curie} \sim 130-140$  K while the material becomes superconducting with a critical temperature ( $T_c \sim 15-40$  K) that depends of the sample microstructure. At room pressure, Sm, Eu and Gd seem to be the only RE elements that accept to enter into the structure of  $RuSr_2GdCu_2O_8$ . In order to better understand this, most unusual behaviour, we have performed some substitutions in the title compound. At room pressure, we have replaced up to 10% calcium for strontium and observed that the unit cell contracts  $V_{Sr} = 169.93 > V_{Ca} = 169.33 \text{ \AA}^3$ . On the other hand, the calcium doped sample shows a positive magnetic susceptibility at 5K. Working at 70 Kbar and 1100°C we have replaced gadolinium by holmium and we have obtained a new material,  $RuSr_2HoCu_2O_8$ , which also shows a smaller unit cell volume ( $V=168.27 \text{ \AA}^3$ ). Magnetic measurements are in progress. As the charge reservoir Ru-O layer has a C4 symmetry axis, both substituted materials are tetragonal, while Ybco is orthorhombic. The unit cell is a perovskite supercell with parameters:  $a = 3.8218(5) \text{ \AA}$ ,  $c = 11.512(2) \text{ \AA}$  and  $V = 168.1 \text{ \AA}^3$ .

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#### 10:30 AM I11.7

LAYERED OXIDES WITH FLUORITE BLOCKING BLOCKS.

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Structural and physical properties of two novel layered-oxide systems, Ru-based superconductors and layered oxysulfides, will be reported. The  $RuSr_2RECu_2O_{8-\delta}$  ( $Ru1212(RE)$ ,  $RE = Eu, Gd$ ) phase has a Y123-related structure, where Cu-O chains are replaced by highly conductive  $SrRuO_3$  perovskite-blocks. The structurally related  $RuSr_2(RE,Ce)_2Cu_2O_{10-\delta}$  ( $Ru1222(RE)$ ,  $RE = Sm, Eu, Gd$ ) phase is also known to have this blocking layer, as well as  $(RE,Ce)_2O_2$  fluorite-block between  $CuO_2$  planes. While  $SrRuO_3$  blocking-layer shows little oxygen nonstoichiometry, this fluorite layer can accommodate much larger oxygen deficiency ( $\delta \sim 0.15$ ). As a result, unlike  $Ru1212$  phase,  $T_c$  of  $Ru1222$  phase depends on the oxygen content. Moreover, detailed analysis suggested a structural change of fluorite block to rocksalt-like structure-type along reduction. On the other hand, newly found layered oxysulfides,  $(Cu_2S_2)(Sr_2CuO_2)$ ,  $(Cu_2S_2)(Sr_2NiO_2)$  and  $(Cu_2S_2)(Sr_3Sc_2O_5)$ , belong to a series of compounds,  $(Cu_2S_2)(Sr_nMnO_{3n-1})$  ( $1 < n < 3$ ). They have perovskite-based  $MO_2$  plane(s) sandwiched by  $Cu_2S_2$  anti-fluorite layer, which is a potential candidate for a new blocking layer because of their structural similarity to HTSC. Formation of these phases is considered to be dominated by the size of the M ions and elasticity of the  $Cu_2S_2$  layers. These versatile (anti-) fluorite building blocks are useful for material designing of new cuprate superconductors.

#### 10:45 AM I11.8

SYNTHESES AND CHARACTERIZATION OF SUPERCONDUCTING  $RuSr_2GdCu_2O_9$ . D.Z. Wang, H.I. Ha, S.X. Yang, J.G. Wen, J.I. Oh, J. Moser, M.J. Naughton, Z.F. Ren, Boston College, Department of Physics, Chestnut Hill, MA; M. DeMarco, State University of New York, Department of Physics, Buffalo, NY; D.T. Verebelyi, M. Paranthaman, T. Aytug, D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN; Michael Haka, Steve Toorongian, SUNY-Buffalo, Nuclear Medicine Department, Buffalo, NY.

A simple procedure to synthesize superconducting  $RuSr_2GdCu_2O_9$  has been successfully developed. The stoichiometric mixture of  $RuO_2$ ,  $2SrCO_3$ ,  $GdO_{1/2}$ ,  $2CuO$  was annealed in air for 40 hours with one intermediate grinding, then pelletized and annealed directly at a proper temperature in flowing oxygen for an appropriate time without either pre-annealing in flowing nitrogen or intermediate grinding. The superconductivity is measured via both ac-susceptibility and four-probe transport method. The composition and the structure of the synthesized  $RuSr_2GdCu_2O_9$  were studied with XRD, TEM and SEM. The dependence of zero resistance  $T_c$  on both annealing temperature and annealing time is thoroughly investigated. Both the magnetic and the superconducting behavior in magnetic fields from 0 to 9 Tesla were also investigated. In addition, superconducting samples have also been prepared using elemental Ru101 or Ru99 which is very much desired for determination of internal local magnetic field by 99Ru Mossbauer spectroscopy, and the result is being expected.

#### 11:00 AM I11.9

$YBa_2Cu_3O_7/La_{0.67}Ca_{0.33}MnO_3$  SUPERLATTICES SHOWING SIMULTANEOUSLY SUPERCONDUCTING AND FERRO- MAGNETIC ORDERING PHENOMENA. Hanns-Ulrich Habermeier, Georg Cristiani Max Planck Institut für Festkörperforschung, Stuttgart, GERMANY.

The physical properties of the perovskite-type oxide  $RuSr_2GdCu_2O_8$  have been recently discussed in the view of a simultaneous occurrence of ordering mechanisms leading to superconductivity and ferromagnetism in different parts of the unit cel. In order to explore some peculiarities of these compounds such as presence or absence of the Meissner state we have prepared superlattices of oxides that are known to be either ferromagnetic [ $La_{0.67}Ca_{0.33}MnO_3$ ] or superconducting [ $YBa_2Cu_3O_7$ ]. Superlattices of different periodicity serve as model systems for the understanding of the peculiarities of the  $RuSr_2GdCu_2O_8$  system and are used to compare their properties with those of our single phase epitaxially grown  $RuSr_2GdCu_2O_8$  thin films. The YBCO/LCMO superlattices have been grown by pulsed laser deposition with individual layer thickness ranging from 4 to 200 unit cells for the  $YBa_2Cu_3O_7$  and 10 to 500 unit cells for the  $La_{0.67}Ca_{0.33}MnO_3$ . The films are characterized by X-ray diffraction analysis, Raman spectroscopy, susceptibility and transport measurements. Whereas simple heterostructures [single layer  $La_{0.67}Ca_{0.33}MnO_3$  and single layer  $YBa_2Cu_3O_7$  50 nm thickness each] reproduce the intrinsic properties of the constituent material rather well [ Curie temperature 250K superconducting transition at  $T = 70K$  ] there are some novel effects emerging due to the coupling between the layers observed in the superlattices. Superlattices with individual thickness of the constituent materials of 4 nm e.g. show a reduced Curie temperature of 120K and a superconducting transition temperature of 60K. Lowering the temperature a reentrant normal

state occurs at  $T = 25\text{K}$ . The experimental findings are discussed within the frame of a phenomenological model based on ferromagnetic interlayer coupling and superconducting proximity effect.

**11:15 AM III1.10**

MICRON-THICK Sr-BASED (Hg,Pb)-1223 FILMS WITH  $J_c(77\text{K})$  OF ABOVE  $10^6\text{ A/cm}^2$ . S.O. Klimonsky, S.V. Samoylenkov, O. Yu. Gorbenko, N.P. Kiryakov, D.A. Emelianov, A.V. Liashenko, A.R. Kaul, Chemistry Department, Moscow State University, Moscow, RUSSIA; D.G. Andrianov, Institute GIREDMET, Moscow, RUSSIA.

Sr-based  $\text{Hg}_{1-x}\text{Pb}_x\text{Sr}_{2-y}\text{Ba}_y\text{Ca}_2\text{Cu}_3\text{O}_z$  compound ( $x \sim 0.2-0.3$ ,  $y \sim 0.5$ ) is characterized by relatively high chemical stability and high critical currents in magnetic fields of 1-2T [1]. For the first time we have synthesized (001) oriented films of that composition. Hg-free precursor films with the thickness up to 1 micron were deposited by MOCVD and laser ablation on (001)LaAlO<sub>3</sub> substrates. Then, the annealing of the precursor films was carried out in sealed quartz ampoules together with the synthesized (Hg,Pb)-1223 ceramic pellets or pressed precursor of the same cation composition. If the (Hg,Pb)-1223 ceramics was used, the mixture of Hg Cu<sub>2</sub>O (molar ratio 1:1) was also added to achieve the desired high vapor pressure of mercury. The (Hg,Pb)-1223 ceramic pellets was synthesized as described in [1,2]. The temperature of film annealing varied from 830 to 850°C, annealing time was in the 3-12h range. No post-annealing in oxygen was used. It was observed, that the phase composition of the films prepared by laser ablation depended crucially on the deposition temperature of the precursor films. For the laser-ablated films, the quantity of CaHgO<sub>2</sub> decreased considerably as the deposition temperature of the precursor film increased from 150 to 600°C. The MOCVD films prepared at 600°C and annealed for 12h contained above 85% of (Hg,Pb)-1223 phase and very small amount of non-superconducting phases according to XRD.  $T_c$  of 118K and  $J_c(77\text{K}, 0.01\text{T})$  of  $2 \cdot 10^6\text{ A/cm}^2$  were determined by ac magnetic susceptibility measurements for these films. These results are among the best reported so far for micron-thick Sr-free Hg-1223 films [3,4], that makes the material interesting for applications in high magnetic fields.

[1] S. Lee et al., Appl. Phys. Lett., 73 (1998) 3586.

[2] S. Lee et al., Physica C 290 (1997) 275.

[3] S.H. Yun et al., Appl. Phys. Lett. 68 (1996) 862.

[4] Y. Moriwaki et al., Physica C 303 (1998) 65.