SYMPOSIUM II

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

November 27 – December 1, 2000

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SESSION III: RECENT PROGRESS IN COATED CONDUCTOR SCIENCE AND TECHNOLOGY
Chihiro Nakajima, Sumitomo Electric Industries
Monday Morning, November 27, 2000
Room 306 (Hynes)

8:30 AM *III.1 RECENT PROGRESS IN R&D OF COATED CONDUCTORS IN JAPAN
Yah Shiobara, 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 Jc. Additionally, Jc and low cost processing are also important factors for the tape applications. In Japan, the development has been proceeding mainly in 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-Al alloy. SE (Surface 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 SE tape showed over 3010 A/cm². On the other hand, “Buffer Layer Alignment” group has made effort to make a long tape using 1SD (Inclined Substrate Deposition) and IBAD (Ion Beam Assisted Deposition) techniques, which can both 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 0.2 km using 1SD process. For aiming thicker superconducting layer with low cost, the third group has been trying MOB (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 Jc. However, high growth temperature and reaction between liquid metal and 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 substrate technique which is YBa2Cu3O7-δ (LPE)/YBa2Cu3O7-δ (MOB) / Applied Superconductivity Technology Center (ISTEC).

9:00 AM *III.2 YBCO COATED CONDUCTOR DEVELOPMENT AT JGC SUPERCONDUCTOR

Intermetalics is developing YBCO Coated Conductor using flexible metal tapes for electrical power applications. These tapes consist of bi-axially-textured buffer layers deposited by Ion Beam Assisted Deposition (IBAD). IBAD has been used to deposit buffer layers of yttrium-stabilized-zirconium (YSZ) on flexible nickel alloy substrates. Using optimized IBAD conditions, bi-axially-textured YSZ has been obtained and the implications of the implications 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/cm² 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 MA/cm² at 75 K and 600 MA/cm² 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法官BASED COATING BY CONTINUOUS COATING processes
D.M. Kroeger, F.A. List, D.E. Lee and Ed Specht, Oak Ridge National Laboratory, Oak Ridge, TN.

Our effort to define and address problems associated with the manufacturing process, described by the RABITS approach in this session, 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 series rather than in parallel, to permit uncoupled development and study of individual processes. Roll-to-roll systems for annealing of rolled metal tapes, deposition of buffer layers by electron beam evaporation and rf sputtering, electron beam co-evaporation of Y-Ba2Cu3-Ox and precursor deposition 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 spray diffusion 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 Jc. Local variations in Jc are thought to be associated with compositional variations occurring during 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.


10:15 AM *III.4 ENHANCING THE CURRENT TRANSPORT IN EX SITU PROCESSED YBCO COATED CONDUCTORS

Processes featuring BaF2 as one of the reactants to form YBCO during ex situ annealing are attractive for coated conductor technology because of inherent precursors, the lack 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 was formed by annealing at 700–800°C, controlled by a decomposition reaction of BaF2 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, 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-Jc, YBCO coatings with thicknesses greater than 1 µm 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. Bokyn, Q.X. Jin, P.N. Arendt, T.G. Holesinger, H. Hung, 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 wire with critical currents (Ic) values (75 K, self field) of 100 A and above. On shorter lengths, Ic values of over 200 A have been reached. In principal, arbitrarily high Ic levels are achievable by simply increasing the thickness of the YBCO layer, which we have demonstrated using 5 µm thick YBCO on single crystal substrates with an Ic equivalent of 700 A/cm with. Similarly, using IBAD-on-metal substrates, we found that as the superconductor thickness was increased to about 1.5 µm the product of critical current density (Jc) and thickness increased to a maximum value of about 220 A/cm. Above this thickness, however, additional YBCO produced no increase in Ic. We investigated this phenomenon by ion-milling the YBCO, remeasuring Ic, and repeating, thereby allowing us to determine the thickness of film up to which the entire film thickness. We discovered two problems. First, many samples with thick YBCO had a 0.25 µm 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 µm. 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 µm. By modifying the superconductor deposition process, we have been able to overcome this problem and make dense coatings up to 5 µm thick. The performance has improved accordingly, with a best result for an IBAD-on-metal substrate of 1.4 MA/cm² at a thickness of 3.7 µm. The equivalent Ic is over 500 A/cm².
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 that required for thermodynamic stability is required. The use of activated oxygen is being investigated. Low fluxes of isonic 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 mm/sec so far. Critical currents up to a MA/cm² at 77 K and JT are found so far.

**11:45 AM 11.I.5**

**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 [011] tilt misorientations lying in the range 3-7°, we find domains of temperature and field well above 4K in which both the intergranular (Jb) and the intragranular current densities (Jc) 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% [011] tilt bicrystals tested at 4K, it is Tc = 0.98 for pure YBCO, Tc = 0.56 for a grown Ca-doped YBCO and Tc/Tc = 0.62 for oxygen-overdoped Ca-doped YBCO, there is a marked improvement in both intergranular properties on doping. Jb/Tc (5%)[011] is 0.51, 0.34 and 2.3 A/cm² respectively. There is a broad separation between Jb and Jc for the pure YBCO and the m-grown Ca-doped sample, but this separation almost disappears for the lowest Tc, oxygen overdoped sample. A broad study of the low angle properties of both monolithic and multilayer samples of varying misorientation and misorientation angles 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 II.2: RECENT PROGRESS ON BI-BASED SYSTEMS - SYNTHESIS, PROPERTIES, AND PERFORMANCE**

Chairs: Victor A. Misochin and Martin W. Rupich

**1:30 PM**

**11.2.1**

**PHASE FORMATION IN Bi-BASED HIGH Tc SUPERCONDUCTORS**


The Bi-Pb [2212] tapes with the highest critical current density values contain around 25% of Bi, 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 percolating pathways within the formation of the Bi-Pb[2223] inside the Ag sheath involves the presence of small quantities of liquid phase (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 note melted crucible-free close to 1170°C in air, and the kinetics of the reactions during protracted 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] rods formed in high density samples (~56%) 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**

**11.2.2**

**MULTIPLE SATURATED LIQUIDS IN PORTIONS OF THE BSCCO SYSTEM**

V. J. Syve, J. K. Moon and D. Eiblen, Department of Chemistry and Texas Center for Superconductivity, University of Houston, Houston, TX.

This study synthesizes our complete work on the BiO₁₋ₓSrₓCaₓCu₂Oᵧ (BSCCO) quaternary system that includes 3 known superconductors belonging to the homologous series Bi/SrCuxCa₁₋ₓCu₂O₆₋ₓₙ (n=3). The BSCCO quaternary is quite complex, containing secondary, ternary and quaternary phases of multicrystalline solid solutions or variable alkali earth ratios. The majority of the quaternary phase volume is occupied by primary phase volumes (PVPs) of A-O (CaO) with disordered SrO and CuO. Separating these two phases is a thin refined phase for the alkali earths. The dominance of these volumes in the quaternary results in the confinement of PVPs of Bismuth bearing phases to a small portion of the ternary. Moreover, Bi-AeO's have PVP's between Bi₂O₃ and Bi₆O₁₇ PVPs. Thus, liquidus relations of all ternary and quaternary phases exist in a small region of interest (ROI). Within this ROI, phases relations vary rapidly with temperature and composition. Our study has determined 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 in 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 BiO₁₋ₓSrₓCaₓCu₂Oᵧ ternary projecting towards the analogous point on the BiO₁₋ₓSrₓCu₆O₁₇ face. The region is bounded by PVP’s of Bi₂O₃, CaO and CuO and Bi-LMs (Low temperature Melting bismuth based Compounds).

2:15 PM

**11.2.3**

**SINGLE CRYSTALS OF Bi-2223 PHASE FORMATION, GROWING MECHANISM AND SUPERCONDUCTING PROPERTIES**

Kenji Naka, Ayako Yamasato, and Setsuo Taguchi, Japan Superconductor Research Laboratory, ISTEC, Koto-ku, Tokyo, JAPAN.

Bi-based 2223 phase with the superconducting transition temperature of 100 K has been considered as the most attractive compound for high temperature applications and commercial applications. However, fundamental studies of this phase are 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 such as 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 unleded 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 reaction techniques, where formation of 2223 phase is 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-melting in different atmospheres in the KCl melt and superconducting properties were studied by XRD, SEM/EDX, 18P/AES, susceptibility and resistivity measurements, thermal and wet chemical methods.

2:30 PM

**11.2.4**

**PHASE RELATIONS AROUND THE Bi-2221 AND Bi-2221**

James K. Moon, Beh Nguyen, Evan J. Syve, Karoline Miller, and D.E. Eiblen, Department of Chemistry and Texas Center for Superconductivity, University of Houston, Houston, TX.

Neares-Bi2201 crystalline Bi-2221 melts incompatibly at all values of p(O2). Liquids in the primary phase volume of Bi-2212 have more Bi than Bi-2221. Our best estimate of the composition of the most Bi-depleted liquid in equilibrium with Bi2212 crystallizes a Bi2212 solid solution (Sr-Ca-SrO-CuO 2.2:2.2:2.2:2.2) at 880°C in oxygen. The liquidus-solidus interval is only 10°C and the second phase is crystalline in a quaternary solid solution (Sr-Ca-CaO-O) from 3 to 13% (Bi). These phases change composition only slightly (low-T Bi2212 2.2:2.2:1.0:0.2:1.0) until liquid is exhausted. Liquids in this system contain CaO and the p(O2) value varies in the Bi2212-SrO-CaO-CuO-O systems, thus truncating some of the phase relations. Furthermore, these lines between Bi2212 and Bi2201 extend into melts and coexisting liquids can only sensibly be interpreted in the quaternary system; they do not show a simple trend toward Bi. A pseudobinary section from Bi2201 to CuO in pure oxygen also demonstrates a compositional change in composition of the solid solution with increasing the liquid composition. Increase in Ca in the bulk composition results in an increase in both Bi and Cu in the Bi2212 solid solution as the...
liquidus phase changes from \( \text{Sr}_2\text{Cu}_3\text{O}_4 \) to \( \text{Cu}_2\text{O} \). Tying together the phase relations of \( \text{Bi-2201} \) and \( \text{Bi-2221} \) (and \( \text{Bi-2231} \)) requires a major problem in phase relations of \( \text{Bi} \) 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 #I12.5**

**RECENT PROGRESS IN THE DEVELOPMENT OF Bi-2212 SUPERCONDUCTORS**

Kazumasa Togano, National Research Institute for Metals, Tsukuba, Ibaraki, JAPAN.

Among the many high temperature superconductors, \( \text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{x} \) (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 been working on Bi-2212/Ag, superconductors, they have not yet been deployed by industry in commercial form. One of the obstacles for commercialization is the high cost of conductor (\( \text{K}\text{A}\text{m} \)). In order to reduce the conductor cost, it is essential to increase the engineering \( J_c \) and to reduce the amount of expensive Ag as much as possible. This paper reports our recent progress 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 \( \text{c-axis} \) grain alignment for the whole oxide layer is the key to achieve larger transport \( J_c \). In addition to this \( \text{c-axis} \) alignment, we have succeeded to achieve in-plane (\( \text{a}\text{b}\text{c}\text{axis} \)) alignment carrying the current process in temperature gradient. The tape showed a transport \( J_c \) value of well over 500kA/m2 under 4.2K and 10T. The paper also proposes a new approach to the improvement of \( J_c \) in the conductor, while keeping the advantage of Ag. The technique is the pre-treatment of \( \text{Ag}/\text{Ni} \) clad tape as a cost-effective substrate instead of expensive Ag. Bi-2212/Ag 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 #I12.6**

**COMPETITIVENESS OF PROCESSING GOALS FOR ATTAINING IDEAL MICROSTRUCTURE IN MELT PROCESSED Bi-2212/Ag CONDUCTORS**


Single-phase, 100% dense, strongly textured microstructure with nanometer-size inhomogeneities is the ultimate processing goal for most HTS materials. In melt processed Bi-2212/Ag conductors, the reported microstructures do not yet meet the above criteria. In this work, we focus on the optimization of melt-processing for Bi-2212/Ag conductors. We show that almost all processing goals are to a certain extent achievable: Porosity-Phase Purity. The soak time in the melt state necessary to attain a certain level of porosity depends on crucible geometry being larger for mechanically stirred shapes (round wires, multilayered tapes). Longer soak times lead to corner-second-phase particles and difficulties in their consumption during solidification. Texture-Phase Purity. Highly textured Bi-2212 layers tend to have such solidification that becomes oblong for mass transfer in the system, because the diffusion along the \( \text{c}\text{-axis} \) is much slower than in the \( \text{a}\text{-b} \) planes. Texture-Porosity. The smaller surface energy of BSCCO-melt/Ag pair as compared to the surface energy of Ag with other phases (including \( \text{O}_x \)) 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 #I12.7**

**RECENT DEVELOPMENTS OF Bi-2212 WIRE AND CABLE**


Recent Progress in the development of high performance Bi-Sr-Ca-Cu-O multicomponent round wire and cable is presented. The critical current and bend strain properties of a 6 ft cable design as function of applied magnetic field up to 8 Tesla will be presented. Cable designs and progress including silver, silver alloy, and nickel wires will be discussed. The cables are fabricated with six active strands each 0.8 mm diameter. The conductor utilized in the cable was a double stack design [7691] comprising of 637 filaments. The critical current (\( I_c \)) of the cable was measured as liquid helium in applied magnetic fields up to 8T. The softfield \( I_c \) of the strand is over 500 Amps. The \( J_c \) is over 400,000 A/cm².

4:45 PM #I12.8


We succeeded in reducing the AC transport self-field losses at 77K and 50Hz in (Bi-Pb)-2223 tape-form conductor fabricated using a four-roll 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 on the performance 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 II: COATED CONDUCTOR SUBSTRATES AND BUFFER LAYERS**

Chair: Ron Feenstra and Yah Shishura

**Tuesday Morning, November 28, 2000**

**Room 306 (Hyves)**

8:30 AM #I13.1

**BIAXIAL TEXTURE CONTROL OF YBCO FILMS ON METALLIC SUBSTRATE BY SURFACE-OXIDATION EPITAXY METHOD.**

Kazumasa Morimoto, Kyoichi Iijima, Masao Oyama, Keiga Kojima, National Research Institute for Metals, Tsukuba, Ibaraki, JAPAN; Tensioni Watanabe, Furukawa Electric, Nikko, JAPAN; Toshihiko Miwa, SeikoBecem Kin, Isumi Hishiyama, SRL/ISTEC, Nagoya, JAPAN.

Surface-oxidation epitaxy (SOE) technique has been investigated for controlling biaxially textured YBCO films on metallic substrates. Highly biaxially textured NiO layers are grown on cubic textured Ni tapes by SOE. High \( J_c \) YBCO films with about 0.5 MA/cm² (IT, 77K) have been obtained on the SOE grown NiO/NiO tapes. Grain boundary thermal grooves in Ni tapes, however, produce grooves in NiO. The NiO grooves become a cause of large single grain boundaries and Ni contamination in YBCO films, resulting in low \( J_c \) values. To reduce the number of large single grain boundaries in YBCO films, oxide capping layer for flattening NiO surface is very effective. MgO, YSZ, CeO₂, etc. are suitable for the capping layer material. These materials are epitaxially grown on NiO/NiO 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 5-10 nm caps the grooves of NiO surface and suppresses the Ni contamination completely. We have successfully fabricated the biaxially textured 50m-long NiO/NiO tape by SOE. The in-plane texture (FWHM) of NiO layer was about 1-2° degrees throughout the length. An effort is underway to deposit the cap layer on the moving NiO tape by physical vapor deposition system. In this paper, the microstructure of YBCO films deposited NiO/NiO tapes with cap layer and their superconducting properties will be reported.

9:00 AM #I13.2

**MECHANISMS OF ION-BEA AMIASSISTED TEXTURING OF YSZ LAYERS.**

Jürgen Dzieck, Sylwia Sievers, Jörg Hoffmann, Lina-Oliver Kutscher, Herbert C. Freyhardt, Zentrum für Funktionswerkstoffe GmbH, Göttingen, GERMANY; Institut für Materialphysik, Universität Göttingen, GERMANY.

Biaxially textured yttriasanitized zirconia (YSZ) buffer layers deposited by an ion-beam-assisted deposition process (IBAD) serve as templates for high-current density Y-Ba-Cu-O 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 ion beam current density 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 (001)-oriented grains nucleate. This preferential orientation is stress induced due to the ion bombardment. In a second step, (001)-oriented grains nucleate epitaxially on (001) grains which are formed 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 (101)-oriented grains with their (111) axis parallel to the assisting beam. The relevant underlying mechanisms have been identified. Ion-beam-induced homeopaxy 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 microrcrystalline 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 energy temperatures and of the divergence of the assisting ion beam will be discussed. Work in part supported by Alcotel and Siemens, within the frame of projects of the BMFT and European Union, respectively.

9:30 AM IB3.3
FORMATION OF BIAXIALLY TEXTURED MgO BUFFER LAYERS ON TEXTURED MGO SUBSTRATES BY IBAD
DEPOSITION. R. Hübner, Ch. Beyer, C.-G. Oertel, W. Skroczki
Technische Universität Dresden, Institut für Kristalllographie und Festkörperphysik, GERMANY; B. Holzapfel, L. Schultz, Institut für 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° with respect to the substrate normal binarily textured MgO films were grown. Under certain conditions a cubic texture is observed in the nucleation state below 10 nm. During further growth this nucleation texture changes to a texture with the (210) direction parallel to the ion beam. This behaviour can be explained with an anisotropic growth of MgO found with spatter epitaxy on single crystals. Furthermore MgO films were grown homeopaxyally 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 IB3.4
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 part, we examined spatter (two-dimensional growth) to determine whether the texture evolution observed in crystals also operated for islands. Although all very small islands are destroyed when hit by high energy ions, the islands only destroyed larger islands are oriented for coalescing. 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 evolution. The results of these simulations are used to facilitate the growth of models for texture development during the early stages of ion beam assisted deposition.

10:30 AM IB3.5
CONDUCTIVE/OXIDE BUFFER LAYERS FOR HIGH CRITICAL CURREN Density YBCO COATED CONDUCTORS.
Thita-Aying J. F. Wu, Univ. of Kansas, Dept. of Physics

Astronomical Sciences Division, Lawrence KS; C. C. Canzani, D. Verhees, D. R. Christen, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN; B. W. Kang, A. Goyal, E.D. Spector, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; M. Paranthaman, Chemical and Materials Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN.

The ultimate development of the YBa2Cu3O7−x (YBCO)-based coated conductors for power applications will require electrical stabilization of the HTS coating. Epitaxial buffer layers of electrically conductive LaNiO3 (LNO) and SrRuO3 (SRO) have been deposited on binarily textured nickel substrates (RABITS) by rf-sputter deposition technique. In the present study, electrical contact between the thin HTS layer and the thick metal, Ni, substrate improves the electrical stability in the event of a transient to the dissipative regime. The new conductive buffer layer structure comprises the interlayer sequence of SRO/LNO/Ni. Due to its significance as a base layer, we have investigated the optimal deposition conditions for the epitaxial growth of LNO on Ni. High quality LNO layers were achieved in oxidizing conditions at growth temperatures between 450−600°C. The surface morphology of the YBCO-plate-like 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 layer process. Epitaxial dense YBCO films by annealing SRO/LNO/Ni multilayer structure yielded self-field Jc values as high as 1.4×10⁶ A/cm² 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 IB3.6

Compared with more conventional oxide buffer such as Yttria-stabilized Zirconia and Ceria, nitride buffers offer the advantages of high electrical conductivity, potential for in-situ epitaxial growth of YBCO directly on the nitride surface, and reduced oxygen transport during the growth of YBCO. The mechanism of YBCO growth on nitride buffer is more complex than on oxide buffer, but has been studied extensively. In this talk, we describe results on a new transition metal nitride based buffer. Unlike nitride buffer previously studied, such as TiN, V3N or VC, we have developed a new compound, that shows great promise in the subsequent oxide growth, including epitaxial YBCO. Buffers of thicknesses ~200 nm were grown on Ni RABITS by reactive magnetron sputtering at ~600-650°C. X-ray diffraction (XRD) analyses of the nitride (111) reflection showed cube-on-cube epitaxy, despite large lattice mismatch. Typical full width half maximum (FWHM) values were 10⁶, while that of the textured Ni substrate was ~8°. A thin (~<100 nm thick) layer of YSZ was coated as an intermediate layer on the nitride buffer coated Ni, followed by ~300 nm thick YBCO layer. The XRD phi scan showed a YBCO (220) reflection with a FWHM of <13°, and XRD rocking curve FWHM of ~6° on (~300) line, indicating relatively good YBCO plate and out-of-plane epitaxy. Simple Photon, however, very low cost method was used for YSZ deposition. The reason for amenity and robustness of result of going-on superconductivity measurement is discussed.

11:00 AM IB3.7

The fabrication of 2nd generation high temperature superconducting materials is currently driven by the need for high temperature superconducting tape products, chemical solution deposition routes to sol-gel, the nucleation and growth characteristics of the YBCO, film in sol-gel routes, the nucleation and growth behavior is defined by the difference in free energy between the amorphous and crystalline state. For a further fundamental understanding of the nucleation mechanism, it is essential to determine the free energy barrier to nucleation in these films independent of 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. This non-Arrhenius method reveals the free energy of nucleation rather than the enthalpy, entropic contributions, such as the differences between in short-range order, can be obtained. We have extended this method, which was first introduced for solidification by Garofolini and Zini, to include surface and interface energy contributions 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
bimially 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 IE.3.8
ISSUES RELATED TO THE FABRICATION OF LONG LENGTHS OF SOLUTION DEPOSITED YBCO CONDUCTOR IN THE DBCO TO YBCO TRANSITION

A low-cost, non-vacuum, solution precursor route has been developed to fabricate epitaxial buffer layers and superconductors on bimially textured-Ni [100] substrates. Most lengths of thick buffered-Ni tapes were grown on solution-based superconducting films and crack-free YBCO films with a Jc of over 140 K/A/cm² at 77 K were grown on all solution buffers using Pulsed Laser Deposition. In addition, YBCO films with a Jc of over 1 MA/cm² at 77 K and self-field were also growth 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 Energy, and Office of Power Technologies-Superconductivity Program. The research was performed at the Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the USDOE under contract DE-AC05-00OR22725.

11:30 AM IE.3.9

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 fields, which is the key to developing high Jc superconductors. In this study we investigated the role of defects (dislocations, grain boundaries, second-phase artificial pinning centers) in YBCO islands growth on low quality substrates. We have prepared YBCO films on thermally oxidized silicon substrates at 100 K with variable substrate orientations. The thickness and the orientation of YBCO films have been measured by X-ray diffraction and surface morphology. The samples were then exposed to a high-flux ion beam (2 MeV Ar²⁺) which results in a high density of defects, and the sample surfaces were characterized by X-ray diffraction and surface morphology. The results show that the ion-irradiated YBCO films have a higher density of defects and a higher density of second-phase artificial pinning centers than the un-irradiated films. The ion-irradiated YBCO films also have a higher critical current density than the un-irradiated films.

Supported by the U.S. Department of Energy under contract DE-AC05-00OR22725 to the Oak Ridge National Laboratory, managed by UT-Battelle, LLC.

SESSION IE : CHEMICAL METHODS FOR DEPOSITING COATED conductors
Chair: Venkat Selynam and Rene Fleiquer
Tuesday Afternoon, November 28, 2000
Room 306 (Hydes)

1:30 PM IE.4.1
APPLICATION OF SOLUTION DEPOSITION TECHNIQUES TO COATED CONDUCTORS: Kaushik Sahana and Srinivasan 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 solution deposition techniques which are vacuum-free. 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 metallic vapor deposition buffer and YBCO layers using simple solution based techniques which can be readily scaled. Processing of oriented buffer layers of barium zirconate and strontium titanate using simple metal vapor deposition techniques have been achieved [1]. These processes, which use precursor solutions instead of soluble, are generally higher oriented buffer layers even when processed in a partial vacuum atmosphere. This route, under optimized process conditions, yields Y123 films with a Tc of 110 K at 10 T on single crystal substrates. These results give promise to the applicability of these scalable solution deposition techniques to coated conductor fabrication.

The first generation HTS conductor, based on the Bi-2212 oxide-powder-in-tube technology, is currently moving from the laboratory into industrial applications. As a result, R&D efforts are increasingly focused towards development of Second Generation HTS conductors with improved performance which will broaden the commercial market for HTS applications. One approach to the development of Second Generation HTS conductor is based on the deposition of YBCO films on buffered, deformation-textured metal substrates with the architecture YBCO/CaZrO$_2$/Y$_2$O$_3$/RE$_2$O$_3$/Ni. One particularly attractive path to the manufacture of such 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 deposition techniques with critical current densities on oxygen buffered, metal substrates approaching 2 MA/cm$^2$ (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-2212 oxide-powder-in-tube conductor, then present improvements in the performance of the solution deposited YBCO films and progress on the development of react-to-vasel processing of the YBCO conductors.

Development of LPE Process for Fabrication of Coated Conductors

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 main processing for coated conductors, 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 the silver. Therefore, the buffer layer to prevent the reaction should be prepared between the metal tape substrate and the LPE layer. Several kinds of oxide materials were tried for the buffer layer. Consequently, MgO was selected for the buffer layer, because of its 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 substracted by the buffer materials was grown from the saturated solvent. This layer is lower Te than YBCO because of its substitution. Therefore, the LPE layer without substation was coated further on the coated substrate LPE layer to obtain high superconducting properties such as $T_c$ above 100 K. Actually, several 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 organization will also be shown.

This work is supported by the New Energy and Industrial Technology Development Organization (NEDO) in Collaborative Research and Development of Fundamental Technologies for Superconductivity Applications.
4:45 PM 114.10
SYNTHESIS OF SINGLE CRYSTAL THIN FILMS 
BY THERMAL EPITAXY

Kwang Sung Yun, Jeong Hwan Song, Byeong Doch Choi, Yuji Matsumoto, Tatsushiko Tech
Institute of Technology, Toyohiro Chikyow, COMET-NIIH, Toyko, JAPAN
Masaaki Kaneko, Hokokuni Kogaku, Tokyo Institute of Technology, Yokohama, JAPAN

we report a novel method of depositing Ni1-xBnzCuOy thin films satisfying both requirements of single crystal quality as well as atomic scale superconductivity in the fabrication of high Tc superconducting devices. this new approach involves the solid thin film growth on a substrate used gas-liquid phase epitaxy within a vacuum chamber or superconducting. This method combines film growth from a liquid phase and pulsed laser deposition. A liquid phase film (non-stoichiometric BaCuO mixture) is intentionally formed on an ultrathin Ni1-xBnzCuOy seed layer prior to the growth of the rest of the film by PLE. The laser ablated Ni, Ba, Cu, and their oxides dissolve into a liquid BaCuO layer on the film/substrate surface and penetrate through 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 PLE technique. Since all three phases, gas, liquid, and solid, are involved in this form of film growth, we have termed it as "Phase Epitaxy." This microstructural characterization by cross-sectional transmission electron microscopy (TEM) performed on several PLE films reveals high epitaxial quality of these films without any grain boundaries over the widest observable area available in the TEM microscope. The Ni1-xBnzCuOy PLE films fabricated on SrTiO3 substrates show the same structure as bulk single crystals over a large area.

SESSION H5: CHARACTERIZATION OF COATED CONDUCTORS I - PHASE CHEMISTRY AND MICROSTRUCTURE

Chair: Roger Weedenweber and Kazumasa Togano
Wednesday Morning, November 29, 2000
Room 3016 (Hynes)

8:30 AM 115.1
MICROSTRUCTURAL DEVELOPMENT IN YBa2Cu3O7. COATED CONDUCTORS BASED ON ION-BEAM-ASSISTED DEPOSITION (IBAD) OF YSZ BUFFER LAYERS. Terry Heiligenstein, Steve Foloy, Paul Arent, Quanxi Jin, Harriet Kung, J. Smith, James Gross, Paul Dowlon, Ray DePaula and E.J. Peterson, Los Alamos National Laboratory, Los Alamos, NM.

A detailed summary of the relevant microstructural features of Y-Ba2Cu3O7 coated conductors fabricated with the IBAD YSZ process on nickel alloy substrates with or without additional buffer layers is presented. Each process step is described and the bulk deposited template used to deposit the superconductor creates an interface which affects 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 was shown how these changes have minimized defect generation and optimized the performance of these coated conductors.

9:00 AM 115.2
YBa2Cu3O7-GROWTH KINETICS AND MICROSTRUCTURES IN BaF2 PROCESSES. M. Sheng, Division of Materials and Chemical Sciences, Brookhaven National Laboratory, Upton, NY.

The so-called BaF2 process is one of the major candidates for commercially viable processes in the fabrication of YBa2Cu3O7 coated conductors for high-current applications. For this reason, significant research and development effort is being expended on this process. The key factors for successful application of this process for conductor production is the control of the c-axis texture of YBa2Cu3O7 crystals on single-crystalline or textured substrates. Here, we describe the kinetics of the nucleation and growth of the YBCO layers on SrTiO3, MgO, and Si substrates. Since two methods of preparing the precursor films, sol gel and chemical 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-A02-76CH00016.

10:15 AM 115.4
PHASE EQUILIBRIA OF THE Ba3Sr3Cu4O10 AND Ba3Sr3YbCu3O10 SYSTEMS. Winnie Wong Ng, Lawrence Cook, Brian Toby, M&SE Laboratory, NIST, Gaithersburg, MD; John Suh, Jeremy Dillingham and Rachel Gouta, University of Maryland, College Park, MD; James Hudson, BP-Amoco Research Center, Nopperville, IL; Terry Heiligenstein and Dean Peterson, LANL, Los Alamos, NM.

In response to the need for improved Ba3Sr3Cu4O10 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% O2 lowering) were developed. A special experimental procedure was used for preparing BaO and for the handling and best-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 lanthanum oxide such as Yb with Ba3Sr3Cu4O10 (Nd-123), both film-成長ing and melting properties of the resulting high Tc phases can be tailored and optimized. The solid solution range of Ba3Sr3Yb1–xCu3Oy under 0.1% O2; 2% O2 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 115.5
CHARACTERIZATION OF COATED CONDUCTOR PHASE

Spectroscopic and synchrotron x-ray methods are being used to optimize 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 microscopy provide microstructure analysis about the YBCO phase 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. Attempted to determine and improve the phase composition and oxygen stoichiometry in the BSCCO type and in the LSCO type. Phase characterization is aimed at determining 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 with substrate features 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 in Argonne Laboratory is supported 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 IE5.6
texture analysis of coated conductors by μ-Raman and synchrotron X-ray diffraction


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 α-crystals in in-plane large angle grain boundaries may benefit from an additional complementary analysis. The non-destructive method of μ-Raman spectroscopy enables an easy analysis of the film homogeneity by determining the distribution of α and c-oriented grains within the tape with a 1 μm 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 α and c-crystals and in-plane textures of substrates, buffers and superconducting layer without sample rotation. Thus, combining film growth technique to in-situ analysis of epitaxial growth of coated conductors prepared by ex-situ processes (solution methods, sol gel, MOD, BaF2 evaporation, ...). Real-time evolution of the microstructure is shown in this paper from these two techniques for stainless steel tapes buffered with HTAD-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 IE5.7
Structural Transport Correlations in Photodoped YBCO Crystals and Films

T. A. Tyson, L. Deng and C. Cui, NIST, NJ.

Photodoped 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 and charge density within the crystal. This research was supported by NSF Grant DMR-9735682.

SESSION IE6: Characterization of Coated Conductors - Microwave, Junction, and Grain Boundary

Chair: Terry G. Horsinger and Juegen Dieck

13:00 PM IE6.1
Impact of Microscopic Defects on Mechanical and Microwave Properties of YBCO Thin Films

Roger Wöllwendler, Jan Einfeld, Peter Lühl, 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 of YBCO films in terms of the HTS model and the two-fluid model, respectively. YBCO films with different size and densities of Y-O-X precipitates are grown on NbAlO and sapphire by variation of the energy of the ions during sputter deposition. The temperature dependence of the microwave surface resistance Rs 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 IQF curve which shifts to higher temperature and decreases in size with increasing density of defects. Temperature dependence and reduction of Rs with increasing density of defects are explained in terms of the two-fluid model with thermally excited quasi-particles characterized 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 Matthiessen 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 IE6.2
Effect of YBCO Film Characteristics on RF Properties of MSL Resonators with these Films

Kazumi Yamanaka, Atsuko Asukawa, Tetsu Nakashima, Fujitsu Laboratories Ltd., Asuigu, Kanagawa, Japan.

Oxide-superconducting films such as YBCO have received much attention due to their application potentials in high-speed electronics, including resonators and filters. In these devices, the unloaded-Q (Q) 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 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 for the frequency of about 3.5 GHz and the measurement results were compared with the experimental results. It was found that the films with Q value of 1E4 - 5E4 at 70 K exhibited strong C-axis orientation, Y211 segregation and porous structures. On the other hand, the films with higher values (5E4 - 1E5) 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 disoriented Y123 grains. It was also found that in similar type of films the influence of the weak-links decreases with increasing the film thickness. Moreover, it was observed that high-Q films possessed fewer weak-links compared with low-Q films irrespective of the film thickness, suggesting that the existing pores and Y123 disorientation in low-Q films result weak-links. Ake, twin boundaries are observed in some weak-links in high-Q films.
In our previous experiments we have developed a highly-doped single-domain YBCO film for component development. The material exhibits excellent physical properties including extremely sharp Tc's, well-controlled crystal orientation, and low surface resistance. However, we believe that Tc can be further reduced if the surface of the single-domain film is modified. In addition, some material defects may arise from the strain on the YBCO film. We have developed a method to reduce the strain on the YBCO film 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 IE.5 SUPERCORRECTING DENSITY IN ULTRATHIN YBCO FILMS. Lyuba Delmoulova, Igor Grekhov, Ian Lainitchik, Ivan Veselovsky, Ioffe Physical Technical Inst, Dept of Solid State Electronics, St. Petersburg, RUSSIA.

The study of ultrathin (<100nm) 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 80nm thickness were deposited on both a SrTiO3 substrate [YBCO/STO] and a YBa2Cu3O7-Nb2O5 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.Masuda et al. [Phys Rev B52 (1995)] have shown that the transport properties of ultrathin YBaCuO films can be regarded as a 2D-fluctuation dominated transition involving vortex-pairing pairs. Both types of films manifested a power law for current-voltage characteristics, and the temperature was shown to decrease linearly with a subsequent drop to unit critical temperature, in agreement with the model. The model parameters were found from the temperature dependence of the transition temperature, which is consistent with the measured resistive transition. The superfluid density n = 2.0 × 1021 cm-2 was found from the Tc value for YBCO/STO to be 2% of the total carrier density in the film and showed an anomalously slow rise just below Tc, an order of magnitude lower than that in single crystals. In contrast, the superfluid density at TK for YBaCuO deposited on the buffer was 2% of the total density in the film and increased just below TK, only by a factor of three slower than in single crystals. The possible reason for the anomalous increase of n, in ultrathin YBaCuO films is discussed.

The work was supported by OMS Grants 3/19604.

3:15 PM IE.6 MICROINTERMODULATION DISTORTION IN BI-CRYSTAL YBCO OR STO BOUNDARY FILMS. H. Xie1,2, D.E. Oates1,2, G. Dresden2,2, and M. Dresden2. 1MIT, Dept of Physics, Lincoln Lab, Cambridge, MA. 2MIT, Cambridge, MA. 3AFRL, Hanscom AFB, MA.

Measurements of the microwave intermodulation distortion (IMD) in a YBCO/SrTiO3 bi-crystal were performed. The IMD measurement is a sensitive probe to study the origin of the non-linear properties of high and low conducting thin films. The two-tone IMD at 4.4 GHz of thin YBaCuO films and films with engineered YBCO/srTiO3 boundaries show the presence of intermodulation distortion of angles from 2 to 3 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 high-angle grain boundary were studied. Different experimental results of high and low mismatch angles are discussed. The correlation between the microwave impedance of the YBCO grain boundaries and the intermodulation distortion were investigated. Modelling 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 IE.7 SUPERCONDUCTING AND MICROSTRUCTURAL PROPERTIES OF 100[100] TILT YBCO THICK FILM GRAIN BOUNDARIES ON SrTiO3. S. Hwang, R. Silcock, and N. Korstanje. National Institute of Standards and Technology, Gaithersburg, MD.

A study is presented of superconducting and microstructural properties of [100] tilt YBCO thick film grain boundaries on SrTiO3 bicrystal substrates and textured Ni substrates, prepared using BaF2 ex-situ photo reaction process. The microstrctural angle of the grain boundaries ranged from 0 to 24.0 degree. In addition, columnar defects were introduced by heavy ion-irradiation along the common 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 photolithography or laser patterning. An electric configuration, with up to 5 voltages pairs located along the bottom grain boundaries, was used to measure the resistance and the voltage-current [V-I] characteristics of the grain boundary in broad hold times. The microstructure of the grain boundary patterned bridge was examined using advanced TEM. We shall discuss the relationship 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-AC02-88CH1088.

3:45 PM IE.8 COMPARISONS OF MICROSTRUCTURE IN PURE AND Ca-DOPED YBa2Cu3O7-δ EPITAXIAL THIN-FILM BICRYSTAL GRAIN BOUNDARIES. Andrew H. Song, G.A. Daniels, J.L. Reeves, D.C. Larbalestier and S.E. Bilbroch. MFEand Applied Superconductivity Center, University of Wisconsin-Madison, WI.

Ca substitution for Y has been shown to show the superconducting properties of epitaxial thin film bicrystals of YBa2Cu3O7-δ for the case of both low-angle and high-angle [001] tilt. For this work, there was deduced deposition was used to grow ~ 250 nm thick YBa2Cu3O7-δ and of YBa2Cu3O7-δ epitaxial films on SrTiO3 substrates. The superconductive properties of the bicrystal improved in the Ca-doped samples relative to the undoped sample in each case. TEM samples that show the magnitude of the change in the upper critical field 11 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 changes in superconducting properties.

Furthermore, the microstructural differences in the single crystal part of the film have already been identified. For example, the density of non 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 IE.9 THE ORIGIN OF REDUCED CRITICAL CURRENTS AT GRAIN BOUNDARIES IN YBCO. M. Kim et al. 1G. Ducham 1-3, N.D. Browning, 2K. Schiller, 3S. Pancek, 3J.S. Penny, 3Oak Ridge National Lab, Solid State Division, Oak Ridge, TN. 2University of Illinois at Chicago, Dept of Physics, Chicago, IL. 3Vanderbilt 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 SrTiO3 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 spectroscopy 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.


Current-limiting defects in coated conductors were investigated using focused ion beam cross-sectioning, scanning electron microscopy, backscattered electron Kikuchi pattern analysis, and magnetic-optical imaging. These combined techniques reveal current barriers in the YBCO superconductor. These barriers are associated with a depletion of YBaCuO film that influence the connectivity of the YBCO, even in coated conductors with high critical current density. These current barriers may be produced by different methods (PLD and BaF2). 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 observed images.
in the YBCO layer are copied from the nickel substrate below. Current barriers appear in the magneto-optic image above all nickel barriers with microcrack formation indicating the influence of the anneal temperature and degree of damage 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 17: CURRENT FLOW AND FLUX PINNING IN YBCO

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


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 multiphase seeding melt-textured growth, (ii) interfaces which are formed by bonding of multilayers with and without chemical additions, (iii) thin film bicrystals on Ni and SrTiO3 substrates, (iv) Ca-doped YBaCuO thin film grain boundaries, and (v) networks of grain boundaries in RABITs and IBAD coated conductors. We investigated 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.


We have isolated small systems of grain boundaries (GBs) on high-Jc coated conductor tapes using magneto-optical (MO) imaging to examine 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 Jc of 0.8-2.7AMa/cm2 (11.77kK) about the value of 2.3 MA/cm2 (11.77kK) 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 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 Jc values.

9:15 AM #11.3 PENETRATION BEHAVIOR OF CURRENT INDUCED MAGNETIC FIELDS IN YBCO THIN FILMS. M. Kuhn, B. Schey, W. Biekel, B. Strüderk, Universität Augsburg, Institut fuer Physik, Augsburg, GERMANY; A. Heinrich, K. Nimsen, H. Knder, Technische Universität München, Physik Department E10, Garching, GERMANY.

Flux penetration of external and current induced magnetic fields can be studied by magneto-optical apparatus it is possible to investigate YBCO thin films as large as 20 x 20 cm². In the present work we studied the penetration behavior of current induced fields in patterned HTS thin films of 8 x 0.7 cm² dimensions at 77K. At an applied alternating current the image intensification 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 Jc > 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 stronger. The investigations were carried out up to currents of Jc = 3 finally resulting in a quench of the YBCO path. The aim was 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 BMFB.

9:30 AM #11.4 MAGNETIC IMAGING OF SUPERCONDUCTING TAPES TO DETERMINE CURRENT FLOW. G.W. Brown, 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 Tc 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 superconducting film 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 area 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 SrTiO3 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 (Ic), based on prior experience. In all samples, for 1 < Ic, current was confined along the edges of the bridge, consistent with the Critical State Model (CSM). For high Ic samples, the supercurrent was increased, current began to flow down the center of the tape. As the supply current approached Ic, we observed a roughly constant J across the width of the sample, also consistent with the CSM. In a sample with low Ic, 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 microscopes.

10:15 AM #11.5 IMPROVED INVERSION TECHNIQUES FOR OBTAINING TRANSPORT CURRENT PATHWAYS FROM MAGNETIC FIELD DATA. F.M. Mueller, D.J. Brown, G.W. Brown and M. Mueller, 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 accurately 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 #11.6 TAILORING MICROSTRUCTURES UNDER STRONG NON-EQUILIBRIUM CONDITIONS: A FEASIBLE PATH TOWARDS HIGH Jc IN MELT TEXTURED YBa2Cu3O7. F. Sandtmann, Institut de Ciencia de Materiales de Barcelona, CSIC, Campus de la UAB, SPAIN; J. Plana, Institut de Ciencia de Materiales de Barcelona, Campus de la UAB, SPAIN; J. Plana, Instituto de Soluciones de Física y Química, Universitat de Poitiers, FRANCE; T. Puig, Instituto de Ciencia de Materiales de Barcelona, CSIC, Campus de la UAB, SPAIN; J. Rabier, Laboratoire de Métallurgie Physique, Université de Poitiers, FRANCE; J. Oberlé, Instituto de Ciencia de Materiales de Barcelona, CSIC, Campus de la UAB, 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 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 I17.10
AC LOSS IN SUPERCONDUCTIVE CABLE: COMPARISON OF YBCO THIN FILMS AND COMPOSITE BSCCO/Au TAPES
H.R. Kerchner and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.
YBCO films grown on both 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 the current flow causes power loss [W.T. Norris, J. Phys. D 3 488 (1970)]. In the low-current regime, loss in a film strip much larger than that calculated by Norris is associated with the peak current to improve superconducting current properties. Behavior of crystal 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 agree with the calculated results depends primarily upon the quality of filmlet-to-filmlet coupling. The possible role of a barrier to vortex entry into a filmlet will be discussed.

SESSION IIB: PROPERTIES AND PERFORMANCE OF BULK 123-TYPE SUPERCONDUCTORS
Thursday, November 30, 2000
Room 305 (Hyres)

1:30 PM I18.1
MICROSTRUCTURE CONTROL OF RE-123 SUPERCONDUCTOR ON INTERACTION OF PINNING CENTERS AND GROWING CRYSTAL SURFACE. Masahiko Awanou, Yoshinobu Fujishiro, Na1, Indust, Res, Int of Nigoya, Nigoya, Japan; Forsh Dogan, Washington Univ, Dept of Materials Science and Engineering, Seattle, WA.
Crystall growth condition of 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 Jc.

1:45 PM I18.2
ENHANCEMENT IN TRANSPORT PROPERTIES OF MELT TEXTURED YBCO BY DOPING. Wei Lo2, Yu X. Zhu2,1, Tong B. Ting2, and Kuemn Salama2. 1Texas Center for Superconductivity and Department of Mechanical Engineering, University of Houston, Houston, TX; 2Department 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 (Jc), trapped fields and levitation force. The enhancement in the transport properties is derived from the fact that 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 Nd2−xBa2−xCuO4−y (NdBCO) solid solution, where substitution of Nd3+ for Ba2+ results in the formation of mm scale low transition temperature (Tc) regions and raises the Jc in melt textured NdBCO. Making use of the properties of the charge reservoir layer as well as the Cu2O2 plane conductance in YBCO, this study shows that apart from Bi2O3 doping, transport properties of YBCO are very sensitive to CuO2 doping on both Cu2O1 and Cu2O2 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 dopant-enhanced melt textured YBCO are
2:00 PM 1B.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 melt processing technique and processing method. Thus 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, 0.3wt%, 1.5wt%, 9vol%, 19vol% and 29vol%) were fabricated by a power melting process method. It is found that the maximum processing temperature is reduced very much by adopting the submicron 211 particles. The magnetostriction curves and magnetic relaxation were measured by a SQUID magnetometer at various temperatures. The results indicate that the irreversibility line and Jc increase with the increase of 211 addition, while Tc 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 submicron 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 Jc(H) curves is observed in the sample with 19vol% 211 addition. These results indicate that even in the present power melting process method, the addition of 211 particles can improve the flux pinning feature and mechanical property of YBCO. Further investigation is underway to optimize the processing conditions to obtain high performance YBCO bulk material.

2:15 PM 1B.4
MAGNETIC PROPERTIES OF MELT-TEXTURED YBCO SINGLE-DOMAINS. Ph. Vanderdenend, M. Ausloos and R. Cloots, SUPRAS, University of Liege, BELGIUM.

This communication aims at reporting the superconducting properties measured on several melt-processed DyBa2Cu3O7−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)B2O3 material with Tc = 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 properties 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 the flux lines has been taken into account in order to extract the critical current density from the magnetic properties.

2:30 PM 1B.5
THEORETICAL MODELING OF STRUCTURAL AND DEFECT PROPERTIES OF RE1−xLnxBa2Cu3Oy, WHERE RE = Y, Nd, Eu and Ln = Eu, Nd, La, 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 YBa2Cu3Oy (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 compounds and used them to estimate various cohesive and structural properties, including heats of formation, elastic properties and the response of structural parameters, such as Cu-O plane buckling, to hydrostatic and uniaxial stress. The energy and structure of various point defects, such as vacancies, interstitials and complexes, was calculated. Trends with the type of RE-123 compound (RE = Y, Nd, Eu) and with non-orthochromism and doping will be discussed, and the implications for flux pinning and processing will be considered. This research was supported under the auspices of the U.S. Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under Contract No. DE-AC02-88CH10886.

3:15 PM 1B.6

4:00 PM 1B.8
LOW TEMPERATURE THERMAL CONDUCTIVITY OF CUPRATE SUPERCONDUCTORS. S. Nakamura, K. Behnia, ESPC, Paris, FRANCE; N. Husey, Dept. of Physics, Loughborough University, Loughborough, UNITED KINGDOM; F. Bulleb-Albenque, SPEC-CEA Saclay, Gif-sur-Yvette, FRANCE; T. Takegai, Dept. of Applied Physics, University of Tokyo, Tokyo, JAPAN; L. Farro, Ecole Polytechnique Federale de Lausanne, Lausanne, SWITZERLAND; C. Unemori, T. Takagi, Dept of Advanced Technical Sciences, School of Frontier Sciences, University of Tokyo, Tokyo, JAPAN; S. Adachi, S. Tajima, Superconducting Research Laboratory, International Superconducting Research Center, Tokyo, JAPAN.

Thermal conductivity (κ) measurements offer rich information on the internal 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 melting of HTS in a wide range (100 K - 1000 mK) in order to verify the validity of some of the existing theories. First, the effect of point defects introduced by electron irradiation in the in-plane a on a sample was investigated on several optimally doped Bi2212. It has been shown that the number density decreases with increased number of point defects at all temperature range, while the linear term in a becomes negative when the temperature shows little or no change. These observations are in quantitative agreement with the theoretical predictions made on the nature of quasiparticles in HTS. Second, we have performed the very first measurements on a along the crystal axis a and b of Y124 crystals, the only known compound that is naturally stoichiometric and undoped. Surprisingly, we find no linear thermal conductivity term in this compound in any thermal 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 1B.10
LOCAL STRUCTURE ANALYSIS OF THE NdBa2Cu3Oy PHASE FOR PROCESSING ADVANCED SUPERCONDUCTING MATERIALS. E.A. Goddard, D. Petrykina, M. Mikurina, A. N. Oleynik, Yu.D. Tret’yanov, Department of Chemistry, Moscow State University, Moscow, RUSSIA; V. V. Petrykin, M. Kakiyana, 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 NdHgBa2Cu3Oy 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 74-1110K in air using reactive, carbon-free and highly homogeneous spray-dried submicron precursors. It was found that the most-substituted solid solution (x=0.01) with the best superconductivity transition temperature Tc~54K and an "ideal" structure forms in air at 980-1000K only while Tc decreases and orthorhombicity parameters drop down significantly when preparing below or above this range. When choosing appropriate oxygenation temperature in air or pure oxygen, the Nd213 (x=0) phase can undergo solid state decomposition into a solid solution (x=0) and Bi2CuO5 with a drastic suppression of Tc whereas low-Tc NdHgBa2Cu3Oy samples decompose drastically faster compared to initially high-Tc single phase samples. The most essential suppression of Tc in a fully oxygenated state was found to occur after aging for 16.450 hrs near 700K. Structural features of the low-Tc neodymiosudetic NdHgBa2Cu3Oy phase (x<0.05, a=6.6, b=6.897, c=6.89 A, b=3.902, c=3.98 A, c=11.707, 11.719 A) were investigated for the first time by XRD, EXAFS structure refinement techniques and Raman spectroscopy. It was found that the lattice parameters are the low superconductivity transition temperature (55-60K) of such a phase are related to structural disorder in the Ba and Nd sites (anisotropic defects) resulting in oxygen disordering which cannot be avoided by a standard oxygenization procedure. Longer annealing at optimal high temperatures is an important factor for cation order that reduces oxygen disorder and enhances superconducting properties.

4:30 PM 1B.11
STRUCTURAL PARAMETERS EVOLUTION IN HTS CERAMICS. B.N. Kolesnik 1,2, V.A. Saran 2, J.P. Zarnowiecki 2.
1Department of Crystal Metrology VNIIIS, Moscow, RUSSIA; 2Material Science Department, IC&RS at Denver, 7311 RN, 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 CuKα radiation, the single and double reflections have been collected in small-angle (15-25 degrees in two-theta scale), middle (45-60) and high angle (100-145) regions. In each case of these measurements the variation in net position, Bragg reflection and halfwidth connected with microstress level has been determined. Data have been collected at ambient temperature and constant orientation of specimen. Earlier these phenomena we introduced as the results either accidental measurements during several years or in turn three-spectrometers in during six weeks only. In addition these experiments we have been carried continuously in 900-1500 hours corresponding to life service following x-ray tube. For data on all of these 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-acclimations process on the n, b, c lattice parameters and orthorhombicity (b-a) degree, grain separations on the become poor and enriched parts. The atomic positions, their occupancies as the result of socrption and description 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 transions 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 microtropic mass transfer and defect interactions with various lengths scales.

SEASON III: POSTER SESSION
SUPERCONDUCTING MATERIALS - CHARACTERIZATION, APPLICATIONS, AND PRODUCTION METHODS

Chairs: Pier W. Knauss and U. (Balu) Balachandran
Thursday Evening, November 30, 2000
8:00 PM
Exhibition Hall D (Hynes)

1D.1
MICROTTEXTURE MAPPING OF ELECTRON BACKSCATTER DIFFRACTION IN (Bi,Pb)2Sr2CaCu2O8+0.3 SUPERCONDUCTOR Tapes. Ming Li, Thien Thanh Tran, Department of Materials Engineering, National University of Singapore, SINGAPORE.

It is believed that the grain boundaries act as weak-link to limit critical current density in bulk high-Tc 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 (Bi,Pb)2Sr2CaCu2O8+x [Bi2223] 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 Bi2223 phase. However, the microtexture distribution of (Bi,Pb)2Sr2CaCu2O8+x crystals with three-dimensional orientations in (Bi,Pb)2Sr2Ca2Cu3O10+x superconductor tapes has not been experimentally determined yet. In present work, Electron Backscatter Diffraction technique was employed to map the crystallographic orientation distribution, the misorientation of grain boundaries and also map the misorientation distribution in Bi2223 superconductor tapes. The results indicate that the c-axes of most Bi2223 grains were perpendicular to the rolling plane, while the 45% of a/b axes and 45% of [110] of Bi2223 grains were aligned along the rolling direction of the Bi2223 superconductor tapes after heavy mechanical deformation. From backscatter electron image and the orientation distribution, it can be observed that the low angle grain boundary are the main boundaries in Bi2223 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 area while the boundaries with 45° misorientation gathered in the middle zone of the measurement area along the rolling direction. To understand the relationship between the misorientation distribution, the boundary misorientation distribution and the fabrication parameters could be used to optimize the fabrication techniques, thus significantly increasing the critical current density.

1D.2
DEVELOPMENT OF LAB-SCALE MAGNETIC SEPARATION SYSTEM USING A CRYOCOOLER-COOLLED Bi-2223 SUPERCONDUCTING MAGNET. Takeshi Ohara, Hiroaki
Kumikawa, Hitoshi Kitaguchi, Hiroshi Wada, Kazunari Togo, National Research Institute for Metals, Takushu, Ikari, JAPAN; Hitoshi Makai, Masayoshi Ohmatsu, Sunmi Electric Industries, Ltd., Osaka, JAPAN; Hidetoshi Okada, Iwate Industrial Promotion Center, Morioka, Iwate, JAPAN.

We have developed a micro-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 water. We report here the present stage of the Bi-2223 magnet development and experimental separation efficiency of weakly magnetized and very fine a-biometric particles from water. We used a-biometric particles from the pelletizer of neutron scavengers, and their average grain size and magnetic susceptibility are 1.0 mm and 1/500, respectively. The Bi-2223 magnet consists of 42 double pancake coils and has a 200 mm inside diameter bore. Each pancake coil was fabricated by a react-then-wind method using pulsed laser deposited 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 tube was also co-wound. The inner, the outer diameters and the height of the magnet are 246 mm, 320 mm and 352 mm, respectively. The magnet is cooled down to 13 K with a Gifford-McMahon (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 1.7 T was repeated at a speed of 1.7 T/min. The temperature of the magnet was increased with increasing number of cycles. For an interval of 3 min between each energizing and de-energizing cycle, the magnet temperature was raised to 34 K 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 be reported.

This work was implemented as part of the multicomponent superconductor project from 1999 supported by the Japanese Science and Technology Agency (STA).

1.9 Po2T STABILITY OF THE 2223 SUPERCONDUCTOR IN THE SYSTEM (Bi,Pb)-Sr-Ca-Cu-O: Ag Lawrence Ack, Winnie Wong, Gifford-McMahon Division, National Institute of Standards and Technology, Gaithersburg, MD; Ralph Klein, Physical and Chemical Properties Division, NIST, Gaithersburg, MD.

The thermodynamic stability of the BiSCCO 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 minimize superconductor phase purity, or, alternatively to cause the formation of necessary phases. We have investigated the effect of Po2 over a range of four orders of magnitude and found that initial melting temperatures fall by 70°C as the oxygen pressure is decreased by 0.3% during this interval. Therefore the 2223 processing window available in Po2-T-X space is significantly expanded relative to that for isochronal conditions. The status of our data set on the location of the 2223 liquid two-phase region in Po2-T-X space will be discussed, including data from thermal analysis, powder x-ray diffraction and quench microscopy analysis. We will also discuss the status of ongoing cryogenic experiments relevant to the thermodynamic stability of the 2223 phase.

1.10 Homogeneity Area of Bi2Sr2Ca2Cu2O8+4 Based Solid Solutions with Substitution of Alkaline Earth Elements By N.D. and L.A. Alexander and G. Verem, A.V. Korobov, V.V. Gavrilov, G.T. Prusakov, 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 by partial decomposition of the superconducting solid solution derived from the superconductive 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 Bi2Sr2Ca2Cu2O8+4 and Bi2Sr2Ca2Cu2O8 (R = Nd, La). Semi-quantitative method 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 Cu2+ in case of substitution of Ca by La. Stability area of 2212 was found to be much more narrower (values of x above 0.3) compared to the other substitutions. One can deduce the fact from the difference of Ca2+ and La3+ ionic radii.

1.11 Growth of Bi2Sr2Cu2O6+δ Superconductor Single Crystal. Takashi Yamanishi 1, Yotatsu Watanabe 1,2, and Isao Tanaka 1,2. 1Yamanashi Univ, Inst Inorganic Synthesis. 2Japan Science and Technology Corporation, CREST.

Bi2Sr2Cu2O6+δ (Bi-2212) is an aggregate-making 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 these methods are very thin along the crystallographic c-axis due to intrinsic crystal growth, and 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 mass transfer in the TSZM method is a combination of 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. The silver crucible was transferred along the vertical and horizontal directions using a vertical and a horizontal type furnace, respectively. The growing 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 is described in detail.

1.12 Peptized Bi-2212: Homogeneity Area, Oxidation and Effect on Jc. A. Veresov, M. Pulkin, Moscow State Univ, Dept. of Materials Sciences, Moscow, Russia; A. Knoglo, V. Putilov, Moscow State Univ, Dept. of Chemistry, Moscow, Russia; E.K.H. Suh, Univ of Cambridge, Dept. of Earth Sciences, Cambridge, United Kingdom.

The most promising way to improve Jc in superconductors is likely to be a creation of small precipitates in the material during decomposition of superconducting solid solution. It was recognized recently that Pb-doping of 2212 could have beneficial effect on Jc, Bi-2212-δPb1-xSrxCu2Oδ (0 ≤ x ≤ 0.7) prepared at different temperatures and oxygen partial pressure (pO2 = 10−2−3, 0.21 atm) via a nitrate route. The effect of Po2 and Pb-doping on Bi-2212 phase formation has been explored with XRD, SEM/EPMA and TEM/SAED. The limit of Po2 solubility in Bi-2212 was found to increase from Po2 decreasing decreasing temperature corresponded to x = 0.21 atm (850°C) and x = 0.6 x Po2 = 10−3 atm (750°C). TEM study of the as-synthesized solid solutions revealed significant nanoscale inhomoogeneity attributed to Pb-segregation. For the samples with x > 0.8 the inhomogeneity quite often looked like wellordered wave with a period of about 10 nm. The samples of (Bi,Pb)-2212 processed under Po2 conditions were oxidized at 600−750°C in air for 0.5−60 hrs in order to cause precipitation of secondary phases from the superconducting solid solution. Thermogravimetric study (TG) of the oxidation gave a strong evidence of two stages composing the reaction. It was found that Jc was enhanced by factor of 2 after annealing at 650°C (i.e. during the first stage of oxidation).

1.13 A Neutron Powder Diffraction Study of the Ferromagnetic Superconductor Gd3Al2Sn2Cu3O10+x. Christopher S. Knee, Yves T. W. Russel, Sheffield University, Department of Chemistry, Southampton, Dept of Chemistry, United Kingdom.

The crystal structure of the ferrimagnetic superconductor Bi(3)Cu-O(2)Cu(2)O(2)Rb(2)O(4) (a = 13.4 Å, perpendicular to the c-axis) has been determined by neutron powder diffraction. Superconductivity occurs in the CuO layers while the magnetic order is associated with the Ru moments. The crystal structure evolves from that of Bi(3)Cu-O(2)Cu(2)O(2), by inserting a fluoro-type Gd3Al2Sn2Cu3O10+x layer instead of the Y layer and replacing the copper site with RuO2 octahedra. The fluoro layer shifts the alternate Cu-O-RuO2-CuO2 perovskite blocks by ½ resulting in both mirror symmetry (S.G. Hi) and an increased c-parameter of 12.5 Å. The basal oxygen of the RuO2 octahedra are displaced within the xy-plane to accommodate physically reasonable Ru-O-Inplane bonds. These displacements are consistent with rotations of the octahedra by 13.5° perpendicular to the c-axis. No evidence of a supercell arising from extended ordering of the RuO2 orientation is observed. The temperature dependence of the structure was measured with scans at 10 K, 140 K and 298 K. The bond Cu-O interactions display the expected transverse nature, however, the spical Cu-O bond exhibits negative thermal expansion and increases significantly upon cooling. The increase is offset by the contraction of the spical Ru-O bond, which leaves the Cu-O separation almost unchanged. The largest changes in the spical Cu-O length between 10 K and 140 K with the oxygen moving ~ 0.2 Å closer to the central Ru ion whilst the Cu-O thickness remains constant. This behaviour is the exact opposite of that displayed by the closely related Gd3Ru2O7 and suggests a structural transition.
10. IMPROVEMENT IN FABRICATION OF Hg-1223 SUPERCONDUCTOR BY Tc DOPING

K.K. You 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 samples to control the molar ratio. In our previous work, 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 oxides 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 TeO2 were added into the precursor, it is shown that the critical temperature of the fabricated samples is comparable to that of the undoped Hg-123 samples. For 0.1 wt% of TeO2, the Tc of the as-synthesized sample is about 113 K whereas the undoped Tc-doped Hg-123 is about 133 K. For 0.18 wt% of TeO2, the Tc 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.

11. SYNTHESIS OF Hg(RE)Ba2Cu3Oy SINGLE CRYSTALS USING B4C O2 CRUCIBLE BY B4C ADDITION


Hg2Ba2Cu3Oy (Hg-123) superconductor exhibits the highest Tc of 135K under ambient pressure among all superconducting materials, and its chemical instability and poor flux pinning performance were well-improved by Te substitution for Hg. Although several groups succeeded in preparation of Hg(RE)123 single crystals, their basic physical properties have not been systematically studied. In the present synthesis, single crystals were obtained by a flux method. The critical temperature of flux was investigated and the maximum critical temperature of (Hg,Re)Ba2Cu3Oy crystals was obtained to be 110 K. The ratio of powders of Ba and CuO was found to be 2:3. The critical temperature for the final samples was measured to be 110 K. The large value of the critical temperature is the result of a well-defined structure.

12. SYNTHESIS AND CHARACTERIZATION OF LAYERED OXYSULFIDES (Cu2S2)/(Sr2−xMox−1)

Hiraku Ogin, Kenji Otsuchi, Jun-ichi Shimo inami, Kohji Kishio, Univ. of Tokyo, Dept. of Superconductivity, Tokyo, JAPAN.

Layered oxysulfides (Cu2S2)/(Sr2−xMox−1) (x = 1.3), which are newly discovered in 1997, made a streaking of perovskite-like JF0 layer and anti-fluorite Cu2S2 layer, and this perovskite-like layered structure is similar to that of high-Tc superconductors and low-dimensional magnetic materials. More than ten compounds have been discovered by changing transition metal M. number of MeO shows us that our previous studies on Cu2S2/(Sr2−xMox−1) (Cu2S2)/(Sr2−xMox−1) and (Cu2S2)/(Sr2−xMox−1) were obtained by improvement of synthesis conditions [1]. Among them, the Cu2S2/(Sr2−xMox−1) has CuO 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 (Cu2S2)/(Sr2−xMox−1). Samples were prepared by solid-state reaction starting from SrS, Cu2S2, and MeO. The stoichiometric composition powders were mixed in an inert atmosphere, pelletized and sealed in evacuated quartz tube. Sintering conditions was optimized to be 450°C for 12h. The resulting sample was characterized by XRD, HR-TEM, and SQUID magnetometer. From Rietveld refinement, (Cu2S2)/(Sr2−xMox−1) was found to have a stacking of tetragonal MgO2 plane and Cu2S2 layer, which is an identical structure with (Cu2S2)/(Sr2−xMox−1). Since (Cu2S2)/(Sr2−xMox−1) contains monovacant Cu and divacant Mg and does not contain any magnetic element, this material can be a comparative material for understanding paramagnetic behavior of (Cu2S2)/(Sr2−xMox−1). [1] K.D. Otsuchi et al. J. Low. Temp. Phys., 117, 729 (1999).

13. Lanthanum Sr2−xOx−4 CERAMICS: LOW TEMPERATURE SYNTHESIS, OXYGEN MOBILITY AND PERSPECTIVES OF CATALYTIC APPLICATIONS

Galina N. Masl. Oleg A. Shilykhin, Shtanskil, Savin.

Unique crystallochemical features of HTSC cuprates resulted in the development of the series of "nonconventional" applications of these materials. Remarkable and easily variable oxygen nonstoichiometry of substituted La2−xOx−4 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 LaSr2−xOx−4 to 600°C and the rate of powder crystallites to ~100 nm. The rate of powder formation is strongly influenced by the concentration of the salt precursor while the most crucial factors are heating rate and Po2 during sintering. Further sintering of finely ground LaSr2−xOx−4 powders allowed to obtain dense ceramic samples at T = 1050 - 1100°C. Studies of oxygen mobility performed by dynamic-thermal O2-isotope exchange method demonstrated substitution of La for Sr in La2−xSrO2−4 (x = 0.15 to 1) reduces the decrease of oxygen release. Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM.

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exchange reaction between oxygen molecules $\text{O}_2$ and $\text{O}_3$. Samples with $x=0.15$ demonstrated good catalytic activity in the reaction of methane oxidation.

**IE.14 CONTINENTAL DISTRIBUTION OF $\text{A}_2\text{Cu}_4\text{O}_9$ STRUCTURES AND PROPERTIES**
Judith A. McAllister, J. Paul Atfield, Univ. of Cambridge, Dept. of Chemistry and IRC in Superconductivity, UNITED KINGDOM; Klaus Prassides, Univ. of Sussex, Dept. of Chemistry, UNITED KINGDOM.

The superconducting behaviour of high-$T_c$ superconductors is known to depend on the $\alpha$ site doping level and the $\alpha$ site size. In addition, the variation in the $\alpha$-site size distribution has recently been found to have an effect on superconducting properties and the $\text{A}_2\text{Cu}_4\text{O}_9$ superstructure type. The control of structure and superconducting properties by the size variance has been established through the synthesis of various $\text{A}_2\text{Cu}_4\text{O}_9$ materials using magnetic and transport measurements, $\mu$SR spectroscopy and variable temperature neutron powder diffraction. These results can be used to construct phase diagrams for all $\text{A}_2\text{Cu}_4\text{O}_9$ compositions using the doping level, average $\alpha$ site size, and $\alpha$ site size variance as axes.

**IE.15 INTER-CHAIN INTERACTION OF METALLIC 1D CHAIN IN $\text{Pr}_2\text{Ba}_2\text{Cu}_3\text{O}_y$**
Shigenori Horii, Univ. of Tokyo, Dept. of Superconductivity, Tokyo, JAPAN; Hidenori Takagi, Univ. of Tokyo, Dept. of New Materials Science, Tokyo, JAPAN; Hiroshi Ikuta, Uchiro Masayoshi, Nagoya Univ., Nagoya, JAPAN; Yuki Yamada, Shimane Univ., Matsue, JAPAN; Isamu Hirokagoshi, ISTEC, Nagoya, JAPAN; Jumpei Nakamura, Keiji Kusuhara, Univ. of Tokyo, Dept. of Superconductivity, Tokyo, JAPAN.

$\text{Pr}_2\text{Ba}_2\text{Cu}_3\text{O}_y$ (Pr124) does not show superconductivity because of the "Pr effect" of $\text{Pr}_2\text{O}_3$ planes in YBCO systems. However, resistivity along only the $\alpha$-axis direction showed a highly conductive behavior, of which the origin is attributed to one-dimensional (1D) double chain sites from nontoxic resistivity between $\alpha$ and $\beta$-axes. This result indicates that the 1D double chain in Pr124 is carrier-doped and one of the most reasonable candidates for studying non-Fermi liquid state in 1D system. On the other hand, a coherent behavior was observed at low temperatures for the $\alpha$-axis resistivity perpendicular to the chain. In this work, in order to elucidate the origin of the coherent $\alpha$-axis transport, we measured $\alpha$-axis magnetoresistance (MR) in magnetic field along the all crystallographic axes using high quality Pr124 single crystals. We found that temperature dependence of the $\alpha$-axis resistivity below 150K resulted from existence of finite inter-chain interaction along the $\alpha$-axis direction at low temperatures. In other words, electronic structure of the chain is 2D (or more higher). This conclusion is consistent with quenched temperature dependence of the $\alpha$ and $\beta$-axis resistivities below 100K which suggests the Fermi liquid state.

**IE.16 MECHANICAL PROPERTIES OF RE-RE$_2$O$_3$ SUPERCONDUCTOR BULK WITH RESIN IMPREGNATION**
Masahiro Tamura, Masato Murakami, Superconductivity Research Laboratory, ISTEC, Tokyo, JAPAN.

Large single-grain bulk RE-RE$_2$O$_3$ (RE: rare earth elements) superconductors can trap large fields exceeding several teslas and thus can function as high-field superconducting magnets and magnets 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 forces 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 Industry and Science and Technology (AIST) of the Ministry of International Trade and Industry (MITI) of Japan.

**IE.17 TEXTURE FORMATION AND SUPERCONDUCTING PROPERTIES OF $\text{YBa}_2\text{Cu}_3\text{O}_7$ THIN FILMS PREPARED BY A**

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

Using trifluoroacetone (TFA) precursors, we grow $\text{YBa}_2\text{Cu}_3\text{O}_7$ (YBCO) thin films on $\text{LaAlO}_3$ (LAO) single crystals. The precursor complexes were prepared by first dissolving the LAO single crystal in the solution, and then heat treating at low and high temperature to develop a superconducting phase and $\alpha$-axis-oriented texture. In this study, processing parameters during the treatment were optimized. Oxygen partial pressure and laser treatment temperature in particular, were closely related to film properties. To characterize film texture, we used pole figure, $\phi$-scan, and X-ray analysis. Superconducting properties were also examined by measuring inductive $T_c$ and transport $J_c$. The possibility of fabricating our YBCO films on coated Niobased substrate will be discussed.

**IE.18 HIGH-TEMPERATURE SUPERCONDUCTING TAPES DEPOSITED BY THE NON-VACUUM LOW-COST COMBUSTION CHEMICAL VAPOR DEPOSITION TECHNIQUE.** Marvin R. White, Steve L. Krebs, Yihin Xue, Adam C. King, Ian H. Campbell, Todd A. Polley, Dave S. Martin, Shara S. Sopou, MicroGosting Technologies, Inc., Chambly, 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-up 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, superlattice or multiple buffer layers, 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 multilayer 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 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.

**IE.19 TECHNOLOGICAL (B, T) PHASE DIAGRAM FROM MAGNETOTHERMAL TRANSPORT DATA.** Marcel Anzud, SUPRAS, University of Liège, Liège, BELGIUM; Rudi Cloos, Institute of Chemistry, University of Liège, Liège, BELGIUM; Jan Macha, Institute for Low Temperature and Structure Research, Polish Academy of Sciences, Wroclaw, POLAND; Marek Palka, Department of Chemistry, University of Warsaw, Warsaw, POLAND.

In our ongoing investigations about the mixed state properties of HTSC 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 $B_c2$ at the melting $(T_m)$ line, the irreversibility $(T_i)$ line, the glass $(T_g)$ transition line, the electrical resistivity peak $(T_p)$ line and several structural PTFL separating regions in which the vortex 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 structural predictions for $T_p(B)$ and the actual data, measured as $B_p(T)$, are discussed. The case of Bi-based HTSCs is used for illustration.

**IE.20 PHOTON BEAM TEXTURING OF SUPERCONDUCTING YBCO**
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) ceramics, 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 10E14, 10E15/cm² at 300 K. It was found that in the interval of 10E14 - 10E15/cm² the irradiation induced oxidation of weak intergran 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 amorphous 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/cm² the SC-transition parameters degrade and the resistivity increases, depending on temperature about 460 K. The intergran 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 stabilizing ceramics under irradiation. The magnetization (pinning) and the anisotropy of GSCeramis 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.

**On the Phase Structure of YBa2Cu3O7-δ**

Ibragim Khidirov,
Umar Galimov,
Institute of Nuclear Physics, Tashkent,
UZBEKISTAN.

It is investigated both superconducting and non-superconducting non-stoichiometric ceramics YBa2Cu3O7-δ. The measurements were carried out by the neutron diffraction method. The samples were irradiated up to a dose of 3.15E10 R in evacuated and sealed ampoules. It has been studied at the powder samples YBa2Cu3O6.98 and YBa2Cu3O4.77. The first one had the temperature of the transition to superconducting state of Tc = 89 K with usual orthorhombic structure. The other sample had semiconductive nature of temperature dependence of electrical resistance in the range 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 non-superconducting powder pattern of YBa2Cu3O4.77 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 (YBa2Cu3O6.98) and it is observed the changes in proportion of diffraction reflexes intensities and in doublet reflexes form which are visually well-marked at the W.B. Berger's angles of 2(θ)°. Large statistic distortions arise in the structure with exposure temperature, and current-voltage characteristic intensities. If Meissner effect has been observed in the starting sample, after irradiation this effect was not found.

The results for powder sample YBa2Cu3O6.98 is analogous to that obtained by measurements at temperature of 450 - 570 K, it is characterized by essentially evacuated vacuum; the difference is that the large statistic distortions of lattice arise in the irradiated sample. The different consequences of effect of heat treatment and irradiation at the YBa2Cu3O6.98 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 powder non-stoichiometric samples is more radiation-stable then stoichiometric one.

**SESSION 110: POSTER SESSION**

**12-LAMP TYPE SUPERCONDUCTORS - THIN FILM AND BULK FORMS**

Chair: Koki Shima and Kousuke Matsumoto
Thursday evening, November 30, 2000
8:00 PM
Exhibition Hall D (Hyatt)

**110.1**

**Preparation of Highly Textured YBa2Cu3O7-δ Buffer Layers on Large Metallic Tapes**

Sylhie Sievers1, Jürgen Drix1,2, Iber Hoffmann1, Alexander Usachenkin1, Herbert C. Freyhardt1,2

1 Institut für Mikrostrukturphysik, Universität Göttingen, GERMANY. 2 Zentrum für Funktionswerkstoffe GmbH, Göttingen, GERMANY.

Bi-axially 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 YBa2Cu3O7-δ (YBCO) films. The deposition of large-area deposition of YSZ films is equipped with two 11 cm Kaufman ion sources. The usage of large diameter ion beam provides a number of characteristic features to the deposition process. Namely, the deposition rate now of the current density on the tape are spatially homogenous. Furthermore, the intrinsic divergence of the ion beam source gives more influence on large-area substrates. Therefore, the spatial distribution 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 merging 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 deposition. To counteract the significant rise of the temperature of smaller-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° FWHM at 1 μm film thickness. The high quality of such tapes is also confirmed by depositing YBCO films onto them. On a tape (10 mm wide and 500 mm) with an in-plane texture of 18.5° FWHM the YBCO exhibits with 10% averaging critical current density Jc at 77 K and 0 T of 0.6 MA/cm².

**110.2**

**Structural and Electrical Properties of YBa2Cu3O7-δ Thin Films on Biaxially Textured Buffered Ni-Based Alloy Substrates**

M. Li, B. M., Y.A. Joe, B.L. Fisher and U. Balachandran, Argonne National Laboratory, Argonne, IL.

Ni-based high-Tc superconducting wires and tapes with high critical current density Jc are essential in power applications. Recently, YBa2Cu3O7-δ (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 bi-axially aligned YBCO thin films with high Jc. In our studies, yttria-stabilized zirconia, CeO2 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 influence 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 3θ scan, X-ray, and pole figure analysis. The superconducting properties such as the critical current density of the YBCO and YBCO film/substrate interfaces were examined by measuring inductive Tc and transport Jc. Details of this study will be presented.

The US is supported by the US Department of Energy (DOE), Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, under Contract W-31-109Eng38.

**110.3**

**Recrystallization**

TREATMENT OF NI ALLOY SUBSTRATES FOR YBCO COATED CONDUCTORS

Rainer Nast, Willfried Goldacker, Bernhard Olt, and Wolfgang Schäfer, Forschungszentrum Karlsruhe, Institut für 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 self-field energy (SFE) of the specific alloy. In combination with investigations of the cube phase formation kinetics, the texture quality of Ni (99.99 wt. %), NiCrTi and NiMnTi 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 recrystallisation behavior. Large grains with worse cubic orientation are observed. Increasing recrystallisation 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 recrystallisation twins with the orientation (123)-2, which leads to a partially textured coating. In Mg 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.

110.4 DISSIPATION AND ANISOTROPY IN ULTRATHIN YBa$_2$Cu$_3$O$_{6.98}$Pb$_{0.02}$Cu$_{0.07}$ SUPERLATTICES J. Villagran, E. M. Gonzalez, J.L. Vicent, Depto Fisica Materiales, Facultad CC. Fisicas, Universidad Complutense, Madrid, SPAIN; Z. Sefrioui, J. Santamaria, Depto. Fisica Aplicada, Facultad CC. Fisicas, Universidad Complutense, Madrid, SPAIN; M. Varela, Depto. 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 thickness of the superconductor layer 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/Tc. 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 dimensionally 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 3D behavior, exactly the same trends as the PbCO films. A detailed study and a comparison with the anisotropy 3D data taken from BSCCO films will be presented.


110.5 THICKNESS DEPENDENCE OF CRITICAL CURRENT DENSITY OF YBCO ON ROLLING-ASSISTED BIAXIALLY TEXTURED SUBSTRATES. B. W. Kang, A. Goyal, D. F. Lee, M. Parmar, D.M. Kroeger, P.M. Mincer, and D.T. Varebelyi, 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 is the epitaxial growth of YBCO films on biaxially textured metal substrates. It has been found that high critical current density ($J_c$) over 1 MA/cm$^2$ 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 to 3 mm. $J_c$ of as high as 0.5 MA/cm$^2$ has been achieved in 3 mm thick YBCO film. These results were compared to those of YBCO films on single crystal substrates. Further characteristics such as Jc vs Tc curves, dJc/dT -26, w -18, and 1/2 scan, and cross section SEM analysis were performed. Possible cause of the degradation of $J_c$ on thick films will be discussed.

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

The development of YBa$_2$Cu$_3$O$_6$ (YBCO) based SNS Josephson junction technology has always hinged upon the use of very expensive single crystal substrates such as LaAlO$_3$, SrTiO$_3$, MgO, and many others. There has not been as much focus on the use of the comparatively low cost yttrium 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 Ba$_2$ZrO$_4$. To get around these effects, a CeO$_2$ buffer layer can be used between the YBCO/PBCO and the YSZ. However, in this case Ba$_2$CeO$_4$ 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 CeO$_2$/YSZ substrate and the minimization of Ba$_2$CeO$_4$ formation. Using a PBCO/CeO$_2$ buffer layer, we have achieved $J_c$ 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.

110.7 FABRICATION OF LARGE GRAIN RE-Ba$_2$CuO$_4$ (RE = Gd, Dy) SUPERCONDUCTORS AND THEIR TRAPPED MAGNETIC FIELDS. Shinya Nakai, Naohide Sakai, Masato Murakami, Superconductivity Research Laboratory, ISTEC, Tokyo, JAPAN.

Large single domain RE-Ba$_2$CuO$_4$ (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$_2$CuO$_4$ and Dy-Ba$_2$CuO$_4$ bulk superconductors and measured their superconducting and field-trapping properties. Large c-axis oriented Gd-Ba$_2$CuO$_4$ bulk superconductors with Ag addition were manufactured under controlled processing conditions. Dy-Ba$_2$CuO$_4$ single crystal Y, Nd$_{123}$ was used as a seed crystal for the growth of large grain bulk. Single domain Gd-Ba$_2$CuO$_4$ samples 32 mm and 48 mm in diameter were successfully fabricated with no macro-sized cracks. The size of 211 particles dispersed in the bulk was reduced to 0.5-1.8 µm with the employment of fine Gd211 starting powder. As a result, large critical current density of 70000 A/cm$^2$ 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 for exceed those of Y-Ba$_2$CuO$_4$ with similar sizes. We also fabricated a single domain Dy-Ba$_2$CuO$_4$ 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.

110.8 NITROGEN SUBSTITUTION AND OXYGEN DEFICIENCY EFFECTS ON THE PINNING PROPERTIES OF NdBa$_2$Cu$_3$O$_{6.95}$ SINGLE CRYSTALS. S. Shihara, Y. Feng, A.K. Pandhan, T. Misui, K. Nakao, and N. Koshiba, Superconductivity Research Laboratory, International Superconductivity Technology Center, Tokyo, JAPAN.

NdBa$_2$Cu$_3$O$_{6.95}$ (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 crystal were prepared under partial O2 pressures of 0.04-1%. All of the as-grown single crystals were annealed at 325°C, for 14 days in O2 atmosphere to fully oxidize the crystals. After that these crystals were heated in air for 2.5 hours at 365, 380, and 4100C, respectively and quenched for giving rise to oxygen vacancies in these crystals. As for the fully oxygenated crystals, the oxygen vacuum concentration is thought to be very low, and we found that $Jc$(on set) increased slightly with lowering PO2 and reached ~96k for PO2=0.04%. On the other hand, the normalized peak field $Fp$=Hp/Hirr where Hp is the magnetic field at which pinning force density $Fp$=max is maximum, and Hirr is the irreversibility field has the maximum around PO2=0.13%. By heat treatments at higher temperatures, Tc of all sample decreased. The higher PO2 and higher temperatures, the more $Fp$ decreased. In the case of heating temperature of 380°C, DTc=1.3K for PO2=0.04% and DTc=6.3K for PO2=1%, but lower PO2 increased $Fp$ namely with increasing Nd/Ba substitution. By further increasing $Fp$ within PO2=0.04% increased, but this $Fp$ value was only about one fifth of $Fp$ of the fully oxygenated sample grown under 0.13%PO2. This fact indicates that it is impossible to obtain large pinning force with only increasing oxygen vacancies. It is important to control the amount of Nd/Ba substitution. This work was supported by NEDO.

110.9 SILVER DISTRIBUTION IN ISOThERMAL SOLIDIFICATION OF YBa$_2$Cu$_3$O$_{6.95}$/Ag COMPOSITE. C. Cai and H. Fujimoto, Railway Technical Research Institute, Kokubunji, Tokyo, JAPAN.

The melting solidification of REBa$_2$Cu$_3$O$_x$ system with proper content of silver addition undergoes a monotectic-peritectic reaction. Due to lower melting temperature, the silver exists as 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 ejection and free trapping phenomena frequently occur for Ag.
II.10.12 SIMULATION AND EXPERIMENTAL DETERMINATION OF THE SPATIAL DISTRIBUTION AND TRANSPORT PROCESS DURING DEPOSITION OF MULTI-COMPONENT, OXIDE MATERIALS.
E. Hollmann, V. Gelbrin, S. Bunte, R. Weenderweber, Forschungszentrum Jülich, IS, GERMANY; V.A. Volovik, Electrotechnical Univ., St. Petersburg, RUSSIA.

Sputter deposition of complex oxide films - e.g. ceramic superconducting or ferroelectric films - require reactive gas or gas mixture (e.g. oxygen and argon) at high pressures of typically 10-100 Pa. In this pressure regime the sputtering process at the stoichiometric target, the material transport from target to substrate and the resulting stoichiometric distribution at the substrate depends on the gas mixture, the cathode-substrate distance and sputtering rate. In this contribution sputter process and material transport are simulated and experimentally analysed for YBa$_2$Cu$_3$O$_7$ and Bi$_2$Sr$_2$CaCu$_2$O$_8$. The deposition was carried out by a sputtering technique. The simulation is based on a Monte-Carlo algorithm. The transport of sputtered atoms in the cathode-substrate space is exposed to well-known laws of sputtering processes and physics of matter nuclear collision. The simulation of large-area deposition demonstrates, that a reduction of the gas pressure from 100 Pa to high-pressure diode sputtering to 

II.10.13 VARIABLE SUBGRAN BOUNDARY DISLOCATION NETWORKS IN SINGLE DOMAIN MULTITEXTURED YBa$_2$Cu$_3$O$_7$. 1PM-LATTICE ON THE BASIS OF CURRENTS, SURFACE FIELDS, AND CATIONIC DISTRIBUTIONS.
J. Rubies, Instituto de Ciencia de Materiales de Barcelona, CSIC, Campus de la UAB, SPAIN; J. Rubies, Laboratoire de Metallurgie Physique, Universite de Poitiers, FRANCE; X. Broduer, Instituto de Ciencia de Materiales de Barcelona, CSIC, Campus de la UAB, SPAIN.

Single domain melt textured YBa$_2$Cu$_3$O$_7$ [123] material is plagued by subgrain boundaries. Transmission electron microscopy observations reveal the microstructure at the boundaries, involving rotation angles, $\theta$ $\phi$, possessing 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 build 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 generalised 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.
SESSION III: SUPERCONDUCTORS OF THE 1212 AND 1223-TYPE CONTAINING TI, Mg, OR RE

Friday Morning, December 1, 2000
Room 306 (Hyves)

8:30 AM III.1.1
SYNTHESIS AND CHARACTERIZATION OF THALLIUM-BASED 1212 FILMS.
J.Y. Lin, D.Z. Wang, S.X. Yang, Y. Tu, H.L. Wu, J.G. Wen, Z.F. Reil, Boston College, Department of Physics, Chestnut Hill, MA; and W. SUNY-Buffalo, SUNY-Buffalo, Buffalo, NY; D.R. Christen, Oak Ridge National Laboratory, Division of Solid State, Oak Ridge, TN; N.N. Bhattacharyya, R.D. Blaugher, National Renewable Energy Laboratory, Golden, CO.

Epitaxial (Ti,Bi)Sr2Ca2Cu3Ox, (Ti,Bi)-1212 films have been successfully grown on both LaA1O3 and CeO2/YSZ substrates by post-deposition annealing in either static air or flowing argon gases. The superconducting Tc has been 1224 structure which gives Tc values in the range of 94-100 K, and Jc values over 1.5 megA/cm² 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 CeO2/YSZ substrates have Tc values in the range of 94-96 K, Je (7K) values close to 0.6 megA/cm² and excellent epitaxy on the CeO2 buffer layers.

8:45 AM III.1.2
SUPERCONDUCTING THALLIUM OXIDE FILMS.

Electrodeposition and spray pyrolysis offer a promising and economic approach for fabricating high-temperature superconducting (HTS) wire and tape for the Thallium superconductor. The Thallomax process offers the potential for deposition at 77K and in magnetic fields of 3-5 T, which is supported by measurements of the irreversibility behavior of the Ti-1223 single layer compound with Pb, Bi, and Sb substitution. Recent advances in the development of Thallium superconductors on Ag-Pd alloy rolled and textured Ni substrates will be discussed. At present we are making two layered electrodeposited films to increase the film thickness and also the uniformity of the films. The morphology for the electrodeposited material is a very important step of the electrochemical reaction which influences directly the structure of the annealed film and, therefore, its properties. In the electrodeposition process, the anodars or anions incorporated in the substrate. With time, as the film thickness increases, the deposition continues either by the buildup on previously deposited material (cold 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] Ti-oxide superconductor film by the electro-deposition process showed a critical current of 28.3 A at 77K (normalized transport critical current = 88.5 A for 1 cm wide samples). At 77K and no magnetic field, the critical current density value of a two-layer, 0.8 micron thick film is 1 x 10⁸ A/cm².

9:00 AM III.3
STRUCTURAL STUDIES OF DIFFERENTLY DOPED TB2201.
Jiaoning Wen, Debi Wang, Xiaoxiang Yang, H.I. Hua, M.J. Nautong, ZF Ren, Department of Physics, Boston College, Chestnut Hill, MA.

TB2201 (Ti-2211) 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 CuO2 plane in each unit cell in comparison with Bi-2212 and YBCO and of the continuous control of doping degree. Previous TEM study on symmetry of Ti-2201 of optim-doped films showed they were tetragonal, which is important for studying pairing symmetry. Recently, extremely overdoped Tb-2201 films with low Tc have been obtained by an off-axis configuration deposition. Here, we report the structural studies of Tb-2201 films with different doping levels prepared by on- and off-axis configurations using transmission electron microscopy and convergent-beam electron diffusion. A modulated structure in the bc plane was observed in overdoped Tb-2201 films. The modulation structure becomes weak from overdoped to optimal doped regimes and finally disappears in optimal doped films. Convergent-beam electron diffraction study on the symmetry indicates that overdoped films are orthorombic-doped films are tetragonal. A twin-like structure was also observed in the overdoped 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 infiltration of deposition configuration on...
modulated structure and films of this type was studied. It was found that the d-wave superconductor was the symmetry of the films, which may result from an inhomogeneous distribution of carriers in films.


Heavily overdoped epitaxial superconducting Tl2Ba2CuOy d (Tl-2201) thin films with single transition and very low zero resistance Tc (<5 K) have been successfully synthesized. The films were made by off-axis RF sputtering deposition and post-deposition heat-treatment of low-casting. The investigations showed that the single transition was contributed to off-axis sputtering deposition, and the low Tc to post-deposition heat-treatment of low-casting. TEM investigations revealed that the films are pure single Tl-2201 phase with good crystalinity. Such samples are entirely superconductive and being measured for detailed investigations of He2 using conventional laboratory superconducting magnets.

10:00 AM III.5 SUPERCONDUCTIVITY AND MAGNETIC PROPERTIES OF HIGH-PRESSURE OXYGEN SYNTHESIZED Rb2-xSrxCd0.5CuO4-x, COMPOUNDS: P.W. Klajn, B. Balakrishna, J. Misaj, M. Rosz, and D. Good, Department of Physics, Northern Illinois University, DeKalb, IL.

We report on the new superconducting materials with the formula Rb2-xSrxCd0.5CuO4-x, that have been successfully synthesized under high-pressure oxygen conditions. By changing the ratio between Rb and Cu in the compound we were able to increase the temperature of the superconducting transition up to 70K for x=0.3. The magnetic characterization of these compounds is discussed and compared to the existing data on Rb2-xSrxCd0.5CuO4-x superconductor (Tc=0.2K for x=0). Work supported by the ARPA/ONR, and by the State of Illinois under HECA.

10:15 AM III.6 Ca/Sr & He/Gd SUBSTITUTIONS IN Ru2SrGdCu2Oy, R. Ruiz-Bustos, J. M. Gallardo, E. Moren, R. Saz-Puente and MA. Alvaro-Franco, Laboratorio Complutense de Almas Presiones, Facultad de Ciencias, Quimica, Universidad Complutense, Madrid, SPAIN.

One of the most intriguing materials derived from YBa2Cu3Oy (YBCO), is undoubtedly the ferromagnetic superconductor Ru2SrGdCu2Oy, in which the charge reservoir layer is composed of Ru2O3 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 Tc≈130-140K, while the material becomes superconducting with a critical temperature (Tc≈15-40K) that depends of the sample microstructure. At room pressure, Sr, Ru and Gd seem to be the only RE elements that accept to enter the structure of Ru2SrGdCu2Oy, in order to better understand this, some previous work we 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 Vc=169.99 to Vc=168.33. On the other end, the calcium doped sample shows a positive magnetic susceptibility at 3K. Working at 70K and 1100K we have replaced gadolinium by holmium and we have obtained a new material, Ru2SrHoCu2Oy, which also shows a smaller unit cell volume V=188.27 Å3. Magnetic measurements are in progress. As the charge reservoir Ru 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=3.824 Å, c=15.92 Å and V=188.4 Å3.

References:

10:30 AM III.1.7 LAYERED oxide WITH FLUORIDE BLOCKING BLOCKS.

Kenji D. Otsuchi, Teru Hinouchi, Hikaru Ogino, Jun-ichi Shimoyama, Koji Kimishio, Univ. of Tokyo, Dept. of Applied Chemistry, Tokyo, JAPAN.

Structural and physical properties of two novel layered-oxide systems, Ru-based superconductors and layered oxysulfides, will be reported. The Cu2Ru3O7−8Y [RE=Ho, Gd] phase has a 123-like related Y2BaCuO5 structure, where CuO chains are replaced by highly conductive SrRuO3 perovskite flakes. The structurally related RuSr2(RE,Er)2Oy+1 ([Ru1222(RE)] phase is also known to be this blocking layer, and as the rare-earth content increases, the blocking layer is reduced in thickness by more than a factor of two, and can be reduced to just a few layers. This is an important result, because the blocking layer thickness is closely related to the Tc of the superconducting layers. The blocking layer has a large contribution to the Tc, and can be used to control the Tc.

10:45 AM III.8 SYNTHESIS AND CHARACTERIZATION OF SUPERCONDUCTING Ru2SrGdCu2Oy, D. Z. Wang, H. H. Hu, S. X. Yang, J. L. Oh, J. M. Naughton, J. F. Ren, Boston College, Dept. of Physics, Chestnut Hill, MA; M. DeMicco, State University of New York, Department of Physics, Buffalo, NY; D. T. Verbeelen, M.礬thann, T. Attig, D. K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN; and D. J. Toorongian, SUNY-Buffalo, Nuclear Medicine Department, Buffalo, NY.

A simple procedure to synthesize superconducting Ru2SrGdCu2Oy has been successfully developed. The stoichiometric mixture of Ru2O2, SrCO3, and Gd2O3 was annealed in air for 40 hours with one intermediate grinding, then pelletized and annealed directly in a pure oxygen atmosphere for 10 ppm in nitrogen for 40 hours without any annealing in nitrogen. The superconducting properties are measured via both ac-susceptibility and four-probe transmission method. The composition and the structure of the synthesized Ru2SrGdCu2Oy were studied with XRD, TEM and SEM. The dependence of zero resistance Tc on both annealing temperature and annealing time is thoroughly investigated. Both the magnetic and the superconducting behavior in magnetic fields from 0 to 9 T 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 Mössbauer spectroscopy, and the result is being expected.

11:00 AM III.9 YBa2Cu3Oy+δ/La2-xCaxMn3Oy SUPERLATTICES SHOWING SIMULTANEOUSLY SUPERCONDUCTING AND FERRO-O MAGNETIC ORDERING PHENOMENA. Hans-Ulrich Hubermann, Georg Christli Max Planck Institut für Festkörperforschung, Stuttgart, GERMANY.

The physical properties of the perovskite-type oxide La2-xCaxMn3Oy 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 cell. In order to explore some peculiarities of these compounds such as presence or absence of the Meisseri state we have prepared superlattices of oxides that are known to be either ferromagnetic [La2-xCaxCu3Oy] or superconducting [YBa2Cu3Oy]. Superlattices of different periodicity serve as model systems for the understanding of the peculiarities of the YBa2Cu3Oy system and are used to compare their properties with those of our single phase epitaxially grown YBa2Cu3Oy thin films. The YBCO/La2-xCaxMn3Oy superlattices have been grown by the pulsed laser deposition with individual layer thickness ranging from 4 to 200 nm for the YBa2Cu3Oy and 10 to 200 nm for the La2-xCaxMn3Oy. The films are characterized by X-ray diffraction analysis, Raman spectroscopy, magnetic susceptibility and transport measurements. Whereas single heterostructures [single layer YBa2Cu3Oy and single layer YBa2Cu3Oy 50 nm thickness each] reproduce the intrinsic properties of the constituent materials rather well, the temperature dependence of the superconducting transition temperature of YBa2Cu3Oy [15 K] is very high. 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 mm e.g. show a reduced Curie temperature of 120K and the onset temperature of 60K. Lowering the temperature a resonance normal
state occurs at $T = 25K$. The experimental findings are discussed within the frame of a phenomenological model based on ferromagnetic interlayer coupling and superconducting proximity effect.

11:15 AM **III.10**

MICRON-THICK Sr-BASED (Hg,Pb)$_2$-1223 FILMS WITH $J_c$(77K) OF ABOVE 1.0$^6$ A/cm$^2$ S. O. Klimanov, S.V. Samoylenko, O. Yu. Gorenko, N.P. Kryukov, D.A. Emel’yanov, A. V. Linashenko, A. R. Kaul, Chemistry Department, Moscow State University, Moscow, RUSSIA; D.G. Andrianov, Institute GRIEDMET, Moscow, RUSSIA.

Sr-based (Hg$_{1-x}$Pb$_x$)$_2$Sr$_{2-y}$Ba$_y$Cu$_2$O$_x$ compound ($x \approx 0.3$, $y \approx 0.5$) 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 this composition. Hg-free precursor films with the thickness up to 1 micron were deposited by MOCVD and laser ablation on (001)LaAlO$_3$ substrates. Then, the annealing of the precursor films was carried out in sealed quartz ampoules together with the synthesized (Hg,Pb)$_2$-1223 ceramic pellets or pressed precursor of the same cation composition. If the (Hg,Pb)$_2$-1223 ceramics was used, the mixture of Hg Cu$_2$O (molar ratio 1:1) was also added to achieve the desired high vapor pressure of mercury. The (Hg,Pb)$_2$-1223 ceramic pellets was synthesized as described in [1-2]. The temperature of film annealing varied from 830 to 850$^\circ$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 CuHgO$_2$ decreased considerably as the deposition temperature of the precursor film increased from 150 to 600$^\circ$C. The MOCVD films prepared at 600$^\circ$C and annealed for 12h contained above 85% of (Hg,Pb)$_2$-1223 phase and very small amount of non-superconducting phases according to XRD. $T_c$ of 118K and $J_c$(77K, 0.1T) of 2*10$^6$A/cm$^2$ were determined by 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.