SYMPOSIUM II

Superconducting Materials–Properties, Crystal Chemistry, and Processing

November 28 – December 3, 1999

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* Invited paper

TUTORIAL

FTi: FUNDAMENTAL MATERIAL ASPECTS OF HIGH-TEMPERATURE SUPERCONDUCTORS
Sunday, November 28, 1999
1:00 - 5:00 p.m.
Room 200 (H)

The last decade has seen major advances in high-temperature superconductors. Several high-temperature superconducting devices have been designed which are based on the compounds $YBa_2Cu_3O_{7-x}$, $Bi_{2+x}Sr_{2-y}Ca_{1+y}Cu_2O_{8+\delta}$, and $(Bi,Pb)_{2+x}Sr_{2-y}Ca_{2+y}Cu_3O_{10+\delta}$, respectively. These compounds have in common the complexity of their crystal structure and multi-component chemistry which requires challenging materials-processing requirements for practical applications. Thus, a practical need exists for understanding the fundamental relations between physical properties, crystal chemistry, phase relations, and processing. The tutorial will investigate the relationship between features at the atomic level, such as crystal chemistry and crystal structure, and macroscopic physical properties. Phase diagrams of the compounds which are the basis for the advanced processing of the materials with controlled microstructure will be discussed. The tutorial will focus on the following topics:

- General remarks on high-temperature superconductors (history, applications)
- Crystal chemistry and crystal structure of $YBa_2Cu_3O_{7-x}$
- Correlation between oxygen content and critical temperature of $YBa_2Cu_3O_{7-x}$
- Phase equilibria of $YBa_2Cu_3O_{7-x}$, melt texture processing of $YBa_2Cu_3O_{7-x}$, coated conductors of $YBa_2Cu_3O_{7-x}$
- Crystal chemistry and crystal structure of $Bi_{2+x}Sr_{2-y}Ca_{1+y}Cu_2O_{8+\delta}$ and $(Bi,Pb)_{2+x}Sr_{2-y}Ca_{2+y}Cu_3O_{10+\delta}$
- Correlation between crystal chemistry and critical temperature of $Bi_{2+x}Sr_{2-y}Ca_{1+y}Cu_2O_{8+\delta}$
- Phase relations and tape processing of $Bi_{2+x}Sr_{2-y}Ca_{1+y}Cu_2O_{8+\delta}$ and $(Bi,Pb)_{2+x}Sr_{2-y}Ca_{2+y}Cu_3O_{10+\delta}$

Instructor: Peter J. Majewski, Max-Planck-Institut fuer Metallforschung

SESSION II1: CRYSTAL CHEMISTRY AND NEW MATERIALS I
Chair: Robert J. Cava
Monday Morning, November 29, 1999
Room 200 (H)

8:30 AM *II1.1
TAILORED OXYCARBONATES AND CUPRATES THIN FILMS : CRYSTAL CHEMISTRY AND SUPERCONDUCTIVITY OF SUPERLATTICES. Jean-François Hamet, Bernard Mercey, Trong Duc Doan, Bernard Raveau, Crismat, Caen, FRANCE; Oleg Lebedev, Gustaaf Van Tendeloo, Ruca, Antwerpen University, BELGIUM; Paul A. Salvador, Department of Materials Science and Engineering, Carnegie Mellon University, Pittsburgh, PA.

Oxycarbonate thin films built up of two different blocks, carbonate $B_{2-x}Ca_xCuO_2CO_3$ (B_2CC) and infinite layer structure $SrCuO_2$ (SCO) have been realized. The so-synthesized superlattices $(B_2CC)_m$ $(SCO)_n$ suggest the possibility to replace the reservoir block "B_2CC" by various other blocks involving an excess holes susceptible to migrate towards the SCO layers. The B_2CC block has then been replaced by the LBCO block ($La_4BaCu_5O_{13}$). The latter adopts the perovskite structure and exhibits indeed a metallic conductivity due to its hole excess. The realization of various superlattices $(LBCO)_m$ $(SCO)_n$ shows the great flexibility of these systems. Nevertheless, no superconductivity is detected. A structural analysis of those superlattices shows that additional oxygen is incorporated into the SCO layers, preventing superconductivity. The latter can be eliminated by argon annealing, but the resistivity is in fact increased due to the simultaneous reduction of the LBCO layer, which then does not play the role of hole reservoir. At this point of the investigation, it clearly appears that $CaCuO_2$ layers (CCO) should be introduced instead of SCO layers in order to avoid the presence of excess oxygen. Unfortunately, we observe that $(LBCO)_m(CCO)_n$ superlattices cannot be set up due to mismatch effects between the two LBCO and CCO lattices. A strategy is then developed, which allows new superlattices to be realized, using a "mirror" structure, (LBCO)m $(SCO)_n$ $(CCO)_{n'}$ $(SCO)_n$(LBCO)$_m$. Such superlattices show a significant decrease of the resistivity which appears most promising for the appearance of superconductivity.
New superlattices corresponding to the artificial stacking of $LaAlO_3$ (LAO) and $Yba_2Cu_3O_7$ layers have also been realized. The (YBCO)m (LAO)n superconducting superlattices exhibit T_c up to 85 K. The coupling between the YBCO superconducting layers is evidenced, T_c being directly related to the stacking sequence of the YBCO and LAO layers. HREM observations show an excellent crystallization and a perfect ordering of the layers, with a rugosity at the interface smaller than 20 $\AA$. Structural models, explaining the junction between LAO and YBCO layers are in progress.

9:00 AM *II1.2
STRUCTURAL ANOMALIES AT THE CROSSOVER BETWEEN THE UNDER DOPED AND OVER DOPED STATES IN HIGH-T_c SUPERCONDUCTORS. J.D. Jorgensen, D.G. Hinks, H. Shaked, Materials Science Division and Science and Technology Center for Superconductivity, Argonne National Laboratory, Argonne, IL; O. Chmaissem, B. Dabrowski, Dept. of Physics, Northern Illinois Univ., DeKalb, IL; J.L. Wagner, Dept. of Physics, Univ. of North Dakota, Grand Forks, ND; Y. Eckstein, A. Knizhnik, Dept. of Physics and Crown Center for Superconductivity, Technion, Haifa, ISRAEL.

The occurrence of a maximum T_c separating the under doped and over doped regimes is a unique feature of copper-oxide high-temperature superconductors. Although this behavior is believed to occur for all high-T_c superconductors, there are a limited number of systems in which it can be achieved because of difficulties in the synthesis chemistry. We have studied the crossover from the under doped to the over doped regime in two systems – a cation-substituted 123 compound and $HgBa_2CuO_4$ – and have observed structural anomalies at the maximum T_c in both cases. We conclude that these anomalies are a manifestation of a change in the electronic structure upon passing through the maximum T_c. Changes in the defect chemistry can also be associated with such a change in the electronic structure. Understanding this behavior is important for developing methods for extending the doping ranges of other high-T_c compounds.
This work is supported by the US Dept. of Energy, Basic Energy Sciences - Materials Sciences, contract W-31-109-ENG-38, and the NSF, Office of Science and Technology Centers, grant DMR 91-20000.

9:30 AM *II1.3
SUPERCONDUCTIVITY IN CARBIDE HALIDES OF RARE-EARTH METALS. R.W. Henn, R.K. Kremer, A. Simon, Max-Planck-Institut fuer Festkoerperforschung, Stuttgart, GERMANY.

Superconductors with layered crystal structures have attracted particular interest because of their unusual physical properties. In 1991 we discovered superconductivity in carbide halides of rare-earth metals $RE_2C_2X_2$, rare-earth RE beeing either Y or La, and halides X beeing Cl, Br or I. These compounds exhibit layered crystal structures with units of bilayers of close packed rare-earth metal atoms that are sandwiched by sheets of halogen atoms. The octahedral voids in the metal atom bilayers are occupied by C-C dumbbells. Early band-structure calculations suggest that covalency which involves antibonding C_2-π^* molecular orbitals and energetically neighboring RE-d states causes an electron delocalization. The particular electronic structure in the Y-C building units was proposed to be of importance with respect to the electronic and superconducting properties.
Here, we report on the experimental investigations of the superconducting state of the phases $Y_2C_2X_2$. In the mixed halide system $Y_2C_2Br_{2-x}I_x$, a sharp cusp in $T_c(x)$ was observed. The maximum transition temperature of T_c=11.5 K was found for $Y_2C_2Br_{0.5}I_{1.5}$. An explanation of the $T_c(x)$ dependence was given relying on band-structure calculations, the temperature dependece of the NMR Korringa relaxation in $Y_2C_2Br_2$, and the preassure dependence of the transition temperature in the $Y_2C_2Br_{2-x}I_x$ system: The Fermi-energy lies close to a maximum in the electronic density of states (DOS) and is shifted across that maximum depending on the halide radii and/or hydrostatic preassure. An analysis of $c_p(T)$ data reveal a strong electron phonon coupling and s-wave pairing in the $Y_2C_2X_2$ compounds. The characteristic parameters of the superconducting state were evaluated from magnetization and μSR experiments. The Ginzburg-Landau parameter indicates extreme type-II superconductivity. The coherence length exceeds the lattice constants by at least one order of magnitude. The anisotropy of the critical field $B_{c2}(0)$, which was measured on $Y_2C_2I_2$ single crystals, indicates an anisotropic but not a quasi-twodimensional superconducting order parameter.

10:15 AM *II1.4
SUPERCONDUCTING HG-BASED MIXED OXIDES AND OXYFLUORIDES. E.V. Antipov, A.M. Abakumov, M.G. Rozova, S.N. Putilin, K.A. Lokshin, D.A. Pavlov, V.A. Alyoshin, Moscow State Univ, Dept of Chemistry, Moscow, RUSSIA; A.M. Balagurov, D.V. Sheptyakov, Joint Institute for Nuclear Research, Dubna, RUSSIA.

$HgBa_2Ca_{n-1}Cu_nO_{2n+2+\delta}$ series exhibits the highest T_c among known superconductors thus providing a great attention to these compounds. Syntheses under high pressure and under regulated mercury and oxygen partial pressures, thermodynamics, structures and properties of different members of the series will be presented and discussed. There are two parameters influencing T_c in this family: a width of a perovskite slab (n) and a concentration (δ) of the extra oxygen. The increase of T_c with n occurs until the third member, while after that it decreases. All members of the series exhibit cupola shaped dependencies of T_c vs. δ. Neutron powder diffraction experiments were carried out for oxygenated and fluorinated Hg-1201 samples with different anion content and T_c. Fluorinated samples exhibit also the cupola shaped behavior for the T_c vs. δ. NPD showed twice the amount of extra fluorine in comparison with those for the oxygenated Hg-1201 phases with close T_c. Fluorination of Hg-1223 resulted in a slight increase of T_c in comparison with oxygenated material. Structures of fluorinated and oxygenated Hg-1223 were refined by NPD. The influence of pressure on the structure and T_c of Hg-1201 strongly depends on the doping level. An increase of the oxygen content on going from underdoped to overdoped state results in the larger compression of the apical Cu-O and Ba-O_{Hg} distances while the HgO_2 dumbbell as well as a distance between Ba and O from the (CuO_2) layers become pressure independent. These results together with the data for fluorinated materials allow to elucidate the structural features responsible for the T_c variation under pressure.

10:45 AM *II1.5
EFFECTS OF LEAD SUBSTITUTION ON BISCCO. Mikio Takano, Yasunori Ikeda, Zenji Hiroi, Iksu Chong, ICR, Kyoto Univ, Uji, Kyoto-fu, JAPAN; Yoshihiro Kusano, Jun Takada, Faculty of Engineering, Okayama Univ, Okayama, JAPAN; Masato Nishiyama, Keiichi Ogawa, Graduate School of Integrated Science, Yokohama City Univ, Yokohama, JAPAN.

Substitution of lead for bismuth shows several chemical and electronic effects in the BISCCO system. The presence of a partially molten phase including lead mediates the formation of the 2223 phase, the structural modulation period is elongated with increasing lead content for all the 2201-2223 phases, the crystal symmetry changes with lead content, resistivity measured normal to the CuO_2 planes becomes low, certain lead-containing fine particles precipitate on the surface of crystals when annealed at low temperatures in oxidizing atmospheres like the air but dissolve again into the crystals on annealing at high temperatures in the same atmosphere.
Two years ago we showed that the pinning effect is greatly improved in the heavily lead-substituted 2212 phase. More recent studies of the structure and defects done by using TEM and UHU-STM and of the effects of lithium on the formation process and structural and electronic properties will he presented.

11:15 AM *II1.6
HOMOLOGOUS SERIES OF LAYERED CUPRATES AND CHEMICAL CONTROL OF HOLE INHOMOGENEITY FOR TAILORING THE HIGH-Tc SUPERCONDUCTING PROPERTIES. H. Yamauchi, M. Karppinen, Tokyo Inst. of Tech., Mater. Structr. Lab., Yokohama, JAPAN.

The multi-layered crystal structures of high-Tc superconducting copper oxides are viewed as members of different homologous series. Correlation between spatial inhomogeneity of different dimensions over the crystals and the basic superconducting properties such as Tc, Hirr and the peak effect has been investigated. It is shown that, the hole distributions among the different layers and within the CuO_2 plane depend on the doping route applied. In general, (i) the more homogeneous the hole distribution along the piling direction of the different layers is, the more improved is the Hirr characteristics, while (ii) the more confined holes are in the middle of the CuO_2-plane stack, the higher is the Tc. Furthermore, (iii) some hundred-angstrom modulations are likely to cause the peak effect.

11:45 AM II1.7
NOVEL SUPERCONDUCTORS SYNTHESIZED BY ELECTROCHEMICAL Zn INTERCALATION IN β-ZrNCl AND RELATED COMPOUNDS. M.E. Arroyo y de Dompablo, E. Moran, and M.I. Alario-Franco, Facultad de Ciencias Quìmicas, Universidad Complutense, Madrid, SPAIN; F. Drymiotis, A.D. Bianchi, and Z. Fisk, National High Magnetic Field Laboratory, Florida State University, Tallahassee, FL.

The structure of β-MNX (M = Zr, Hf; X = Cl, Br) consists of an ordered stacking sequence of hexagonal XMNNMX layers held together by van der Waals interactions [1], allowing the intercalation of different guests into the host lattice. The intercalated A_xMNX compounds (A = alkaline metal, cobaltocene) are superconductors[2],with a critical temperature T_c of the superconducting transition that varies only little with the doping level x and is rather insensitive to the choice of the chemical species A for the doping. To our knowledge, no previous work has been reported on the intercalation of divalent ions into this class of compounds, so we have attempted to introduce Zn into β-ZrNCl, β-ZrNBr and β-HfNCl. The loading/unloading of zinc into β-MNX was carried out electrochemically through the potentiostatic discharge/charge of Zn/0.2M $Zn(CF_3SO_3)2$ + DMSO + PC (1:4)/β-MNX cells in the voltage window between 1 and 0 V against Zn. Noticeably different results from the previously studied lithium doping are obtained for zinc incorporation. In the case of β-MNCl an intercalation reaction takes place, leading to the final compound $Zn_{0.04}$ZrNCl with a Tc of 15 K. For β-MNBr an irreversible process is observed yielding the superconducting compound $Zn_{0.02(1)}$ZrNBr with a Tc of 14 K. However, attempts to obtain Zn_xHfNCl superconductors using this technique were unsuccessful. Superconductivity in *beta*-MNX appears at surprisingly low levels of electron doping for the case of zinc in comparison to the monovalent dopants. 1.-V.R. Juza and H. Friedrichsen, A. Anorg. Allg. Chem., 332, 173 (1964) A. Fogg, J. Evans and D. O'Hare, Chem. Commun., 2270 (1998) 2.-H. Kawaji, K. Hotehama and S. Yamanaka, Chem. Mater, 9, 2127 (1997) A.M. Fogg, V.M. Green and D. O'Hare, Chem. Mater, 11, 216 (1999) S. Yamanaka, S. Hotehama and H. Kawaji, Nature, 392, 580 (1998)

SESSION II2: CRYSTAL CHEMISTRY AND NEW MATERIALS II
Chair: Ruling L. Meng
Monday Afternoon, November 29, 1999
Room 200 (H)

1:30 PM *II2.1
STRUCTURES, PROPERTIES AND STABILITIES OF NOVEL COPPER-RICH OXIDES. Douglas A. Vander Griend, Kenneth R. Poeppelmeier, Sylvie Malo, Antoine Maignan, Northwestern University, Chemistry Dept, Evanston IL; Vinayak Dravid, Northwestern University, Materials Science Dept, Evanston IL.

The chemistry of cuprates pervades the field of perovskite superconductors. Many new solid state phases with $(ABO_3)_n$ stoichiometry have been found for which over half of the B-cations are copper, but which do not adopt the perovskite structure at ambient pressure. Archetypal examples $La_4Cu_3MoO_{12}$ and $La_3Cu_2VO_9$ crystallize in a hexagonal structure at ambient pressure which is a homeotype of $YMnO_3$. Under 6 GPa of pressure, both convert to a layered perovskite. The cuprate layers of the high pressure molybdenum phase, critical for high temperature superconductivity, disappear upon A-site doping as the cations mix. The stabilities of the different structures are compared as well as possible decomposition products. Magnetic properties and conductivities for doped and undoped samples reflect the stoichiometry and structural architecture.

2:00 PM *II2.2
CREATION OF THE BEST PERFORMANCE SUPERCONDUCTOR BASED ON Cu-1234 ($CuBa_2Ca_3Cu_4O_{12-y}$) SYSTEM. Hideo Ihara Electrotechnical Laboratory, Umezono, Tsukuba, Ibaraki , JAPAN and CREST, JST.

The purpose of this paper is to propose how to create the best performance superconductor based on the Cu- 1234 ($CuBa_2Ca_3Cu_4O_{12-y}$) system of ($CuBa_2Ca_{n-1}Cu_nO_{2n+4-y}$: n=3~6) family. The best performance superconductor should have a long coherence length along c- axis ($\xi_c > 1nm$), small penetration depth ($\lambda_c \sim 200nm$), low superconducting anisotropy ($\gamma \sim 1.5$), high T_c (>116 K = one and a half time of 77 K), high J_c { $50MA/cm^2$, (77 K, 0 T), 0.5 MA/cm^2 (77 K, 10 T)} and high H_{irr} (~ 30 T). The best performance superconductor will be realized by the modification of superconducting wave function (MSWF) and the development of new preparation techniques of thin film for the Cu-1234 system. The MSWF means the elongation of ξ_c based on the uncertainty principle and the transformation from d- wave to (d+is)-wave superconductivity. The (d+ is)-wave will be realized by a selective over-doping effect in the Cu-1234 system. The new thin-film preparation techniques are APE (amorphous phase epitaxy) and SAE (self-assembling epitaxy) methods by using structure stabilizer and reaction accelerator such as Tl. Our recent results shows a high T_c of 132 K and high J_c {20 M A/cm^2 (77 K, 0 T), 0.4 M A/cm^2 (77 K, 10 T)} for $Cu_{1-x}Tl_x$ − 1223 system. Further higher performance is likely to be achieved by the $Cu_{1-x}Tl_x$ − 1234 system. The best performance superconductor is necessary for wire, bulk and Josephson

junction and microwave device applications at 77 K, and it is essential for the construction of the energy and information super- highway in the 21st century.

2:30 PM *II2.3
MOLECULE AND CRYSTAL ENGINEERING OF HIGH-TC STRUCTURES AND DEVICES. John T. McDevitt, Department of Chemistry & Biochemistry, Texas Materials Institute, The University of Texas at Austin, Austin, TX.

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

3:30 PM *II2.4
STRUCTURE AND DEFECT STRUCTURE OF FLUORINATED SUPERCONDUCTING MATERIALS. Joke Hadermann, Oleg Lebedev, Artem Abakumov, Gustaaf Van Tendeloo, EMAT, University of Antwerp, BELGIUM.

Bulk superconductivity with Tc up to 94K has been induced by fluorination of non-superconducting $YBa_2Cu_3O_{6.11}$ using XeF_2 as a fluorination agent. HREM shows the presence of a new phase with c =1.3 nm within a matrix of the 123 structure. Fluorinated compounds exhibit a strong disorder along the c-direction. Microanalysis demonstrates that fluorine does enter the YBCO structure inducing a significant structural rearrangement for high level fluorinations. The structure of the ideally fluorinated $YBa_2Cu_3O_6F_2$ phase, has been determined from HREM observations. For $Y_2Ba_4Cu_7O_{14.09}$, the intercalation of fluorine is accompanied by a significant structural rearrangement and leads to an increase of Tc from 30K to 62K. Fluorene intercalation results in the formation of an octahedral arrangement of Cu1 atoms with a significant elongation of the apical Cu-O distances. The structure with a complete octahedral arrangement of the copper atoms only exists in limited regions; the c parameter in these areas is as high as 53.5$\AA$. In $La_2CuO_{4-x}F_y$ superconductivity is suppressed by the appearance of several (commensurate or incommensurate) modulated phases. In Hg-1201 the fluorination leads to an increase of Tc from 60K to 97K and allows to obtain this compound even in overdoped non-superconducting state. The double amount of fluorine was found by neutron diffraction in comparison with oxygenated samples with the same Tc, which strongly supports an ionic model of hole doping in the Hg-1201 compound.

4:00 PM II2.5
Abstract Withdrawn.

4:15 PM II2.6
EFFECTS OF CATION SUBSTITUTIONS ON Tc IN DIFFERENT HTS SYSTEMS. Francesca Licci, Andrea Gauzzi, Massimo Marezio, MASPEC Inst of CNR, Parma, ITALY.

The application of external pressure to cuprate superconductors may induce increases of T_c. In spite of numerous attempts, such effect could not be reproduced by applying chemical pressure, by substituting small for the large cations, typically Sr for Ba. The effect of these substitutions depends on the cuprate family, and, with the only exception of the $La_{2-x}(Ba_{1-y}Sr_y)_xCuO_4$ system, it has always resulted in decreases of T_c. In the La-Ba-Sr-Cu-O system T_c increases either for increasing y or for increasing external pressure. In addition, Locquet et al. recently showed that T_c in this system can be further enhanced by contracting the basal plane (through epitaxial growth of thin layers on a substrate with a smaller a parameter) and expanding the c parameter. Our studies of the crystal structure and superconducting properties of $Y(Ba_{1-x}Sr_x)_2Cu_3O_w$, as a function of x and w, showed that the Sr substitution in this system reduces the unit cell volume as does the external pressure. However, it also reduces the strain of the Ba/Sr layers which seems to be needed for optimizing the charge transfer between the chain and the planar Cu cations. The strain of the Ba layer seems to scale well with T_c, at least in the YBCO system. Negative dT_c/dx coefficients have also been measured in the Sr -substituted $Hg(Ba_{1-x}Sr_x)_2Ca_{n-1}Cu_nO_{2n+2+\delta}$ compounds. The dT_c/dx coefficient, on the contrary, is positive. Refinement data of $Hg(Ba_{1-x}Sr_x)_2CuO_{4+\delta}$ ($x \leq 0.5$), obtained by high-pressure synthesis, indicate that the Sr substitution reduces the unit cell volume and the interatomic distances, however, the critical temperature decreases. Also in this case a reduction of the strain at the large cation sites (Ba/Sr and Hg) is observed for increasing x. It should be interesting to try for a mercury-based cuprate and YBCO the anisotropic variation of the unit cell as that obtained by Locquet et al. for $La_{1.9}Sr_{0.1}CuO_4$.

4:30 PM II2.7
MAGNETIC AND ELECTRICAL PROPERTIES OF COBALT ANALOGS OF THE CUPRATE SUPERCONDUCTORS. R.J. Cava, S. Loureiro, N. McGlothlin, P. Khalifah and D. Young, Department of Chemistry and Materials Institute, Princeton University, Princeton NJ; R. Jin, Yu. Zadorozhny and Y. Liu, Department of Physics, Pennsylvania State University, State College, PA.

The Co analogs of Bi 2212 and the superconducting copper oxychlorides have been synthesized and their electronic and magnetic properties studied on small single crystals. For the 2212 related phases, metallic conductivity is observed down to very low temperatures in variants for which partial substitution of Pb for Bi has been made. No superconductivity is observed. The new single and double layer trivalent cobalt oxychlorides Sr_2CoO_3Cl and $Sr_3Co_2O_5Cl_2$ are reported for the first time. In addition to their electronic and magnetic properties, the results of electronic band structure calculations will be reported. This research is supported by Department of Energy grant DE-FG02-98-ER45706.

4:45 PM II2.8
MAGNETIC AND ELECTRONIC PROPERTIES OF Am AND Cm IN HIGH T_c RELATED COPPER OXIDE SYSTEMS.
S. Skanthakumar, L. Soderholm, C.W. Williams, Chemistry Division, Argonne National Laboratory, Argonne, IL.

The physical properties of Cm in $Cm_{2-x}Th_xCuO_4$, $CmBa_2Cu_3O_7$ and $Pb_2Sr_2Cm_{1-x}Ca_xCu_3O_8$, and Am in $Pr_{2-x}Am_xCuO_4$, have been studied using x-ray and neutron diffraction, magnetic susceptibility and x-ray absorption near edge structure experimental techniques. These Cm and Am analogs are isostructural with their rare earth counterparts. The experimental results consistently show that Cm in these compounds are trivalent whereas Am is tetravalent. Magnetic susceptibility experiments show evidence for anti-ferromagnetic ordering of Cm spins at 25 K in Cm_2CuO_4, 22 K in $CmBa_2Cu_3O_7$ and 15 K in $Pb_2Sr_2CmCu_3O_8$. Magnetic neutron diffraction data show that the Cm spins order with a simple antiferromagnetic structure in Cm_2CuO_4. The high magnetic ordering temperatures, which are higher than those of the other rare earths in corresponding compounds, indicate that the magnetic exchange interactions are very strong. Tetravalent Am inhibits the formation of $Am_{2-x}Ce_xCuO_4$, a result that is in contrast to a recent theoretical prediction that superconductivity can be induced in $Am_{2-x}Ce_xCuO_4$. This work is supported by the U.S. DOE, Basic Energy Sciences Chemical Sciences, under contract W-31-109-ENG-38.

SESSION II3: POSTER SESSION:
BULK AND FILMS
Chair: Timothy J. Haugan
Monday Evening, November 29, 1999
8:00 P.M.
Exhibition Hall D (H)

II3.1
INVESTIGATION OF THE PHASES FORMATION AND SUPERCONDUCTIVITY OF $Ag_{(1-x)}(HgBa_{1.9}Bi_{0.1}Ca_2Cu_3O_{8+\delta})_x$ ($0.05 \leq x \leq 0.4$). H.R. Khan, FEM, Materials Physics Department, Schwaebisch Gmuend, GERMANY and Dept of Physics, University of Tennessee, Knoxville, TN.

Ag substitution in Hg-based high T_c superconducting materials improves the chemical stability and superconducting properties (1). A series of materials of compositions $Ag_{(1-x)}(HgBa_{1.9}Bi_{0.1}Ca_2Cu_3O_{8+\delta})_x$ ($0.05 \leq x \leq 0.4$) were prepared directly from the oxide powders, Ag_2O, HgO, BaO, Bi_2O_3, CaO and CuO by mixing, grinding in a dry air glove box and pressing. The pressed tablets were placed in an evacuated quartz tube and given heat a treatment of 700°C / 10 h and slowly cooled to room temperature with a cooling rate of 1°C / min. Substitution of Bi with Ba in Hg-1223 material and addition of different amounts of Ag enhances the chemical stability and affects the morphology. Lattice parameters of the superconducting Hg-1223-tetragonal phase and the formation of other phases are investigated by X-ray diffraction technique. The Ag addition does not affect the critical transition temperature as measured by the temperature dependent electrical resistivity and magnetization. But the T-dependence of the resistivity above T_c depends on the Ag-concentration. Ag addition also affects

the superconducting volume. The formation of various phases together with Hg-1223 phase as well as morphology of the materials as a function of Ag-concentration in relation to superconductivity will be reported.
(1) H.R. Khan, J. of Superconductivity, vol. II, No. 1, 15 (1998)
This work was supported by the Deutsche Forschungsgemeinschaft under the grant no. DFG-Khan20/1-1.

II3.2
EFFECT OF HOLE FILLING BY La AND HOLE DOPING BY Ca ON THE SUPERCONDUCTIVITY OF $NdBa_2Cu_3O_{7-\delta}$.
Amish G. Joshi, M.V. Subbarao, D.G. Kuberkar and R.G. Kulkanri, Department of Physics, Saurashtra University, Rajkot, INDIA.

The structural and superconducting properties of La and Ca substituted $(Nd_{1-x}Ca_x)(Ba_{2-y}La_y)Cu_3O_z$ samples prepared under identical conditions have been investigated by X-ray diffraction, resistivity, a.c. susceptibility and oxygen content measurements. The observed lowering of T_c with increasing y in $Nd(Ba_{2-y}La_y)Cu_3O_z$ provides convincing evidence that the filling of holes by La^{3+} reduces hole concentration and suppresses superconductivity, varying smoothly through an orthorhombic to tetragonal transition with increasing y. This suppression in T_c can be compensated by an appropriate hole doping with Ca. The introduction of Ca for Nd in $(Nd_{1-x}Ca_x)(Ba_{1.6}La_{0.4})Cu_3O_z$ has shown that T_c increases from 0 K (x = 0.0) to 84 K (x = 0.4) closer to the value of 91 K for pure $NdBa_2Cu_3O_{7-\delta}$ (x = y = 0.0) with structural transition from tetragonal (x = 0.0) to orthorhombic (x = 0.4).

II3.3
STRUCTURAL AND SUPERCONDUCTING PROPERTIES OF THE $La_{3.5-2x}Y_xCa_{2x}Ba_{3.5-x}Cu_7O_z$ SYSTEM.
M.V. Subbarao, Amish G. Joshi, D.G. Kuberkar and R.G. Kulkanri, Department of Physics, Saurashtra University, Rajkot, INDIA.

The structural and superconducting properties of $La_{3.5-2x}Y_x$-$Ca_{2x}Ba_{3.5-x}Cu_7O_z$ ($0 \leq x \leq 0.7$) system are investigated by X-ray diffraction, neutron-diffraction, resistivity, a.c. susceptibility and oxygen content measurements. Samples with $0.1 \leq x \leq 0.7$ are superconducting and $T_c{}^{R=0}$ is between 27 and 79 K. Rietveld refinement of neutron diffraction data on five samples x = 0.0, 0.20, 0.40, 0.50 and 0.70 confirms (i) occurance of a single phase tetragonal structure withspace group P4/mmm, (ii) Ca and Y ions substitute onto the La sites with concomitant displacement of La onto Ba sites, and (iii) increasing x from 0.0 to 0.50 decreases La-O(3) bond length and increases Cu(2)-O(2) bond length with corresponding increase in T_c from 0 K to 79 K suggesting a correlation between bond lengths and T_c values.

II3.4
PROCESSING OF SUPERCONDUCTING COMPOSITES IN THE SYSTEMS Bi(Pb)-Sr-Ca-Cu-A-O (A=Al, Ga, In).
V.V. Poltavets, P.E. Kazin, O.N. Poltavets, A.A. Kovalevsky, Yu.D. Tretyakov, Chemistry Department, Moscow State University, RUSSIAN FEDERATION; M.Jansen, Max-Plank-Institut fur Festkorperforschung, Stuttgart, GERMANY.

Phase transformations in the systems Bi-Sr-Ca-Cu-A-O (A=Al, Ga, In) were investigated with the purpose to obtain two phases superconducting composites. The phase assemblages in the systems Bi-2212/A-rich phases were determined above and below the Bi-2212 melting point. Phases $Sr_{1.7}Ca_{1.3}Al_2O_6$, $BiSr_{1.5}Ca_{0.5}Al_2O_x$, $Bi_{0.2}SrCa_{0.8}Ga_2O_z$, $(Sr,Ca)_3Ga_2O_6$, $Sr_{0.6}Ca_{0.4}In_2O_4$ were determined as chemically compatible with Bi-2212. Changes in the Bi-2212 melting process were observed for $BiSr_{1.5}Ca_{0.5}Al_2O_x$ and $Bi_{0.2}SrCa_{0.8}Ga_2O_z$ addition: Bi-2212 + $BiSr_{1.5}Ca_{0.5}Al_2O_x$ ->L + $(Sr,Ca)_3Al_2O_6$ Bi-2212 + $Bi_{0.2}SrCa_{0.8}Ga_2O_z$ -> $Bi_{1.7}Sr_{2.3}Ca_{0.6}$ $Cu_{1.6}Ga_{0.4}O_z$ + L1 ->L2 +$(Sr,Ca)_3Ga_2O_6$ In the case of doped systems no cuprate and Cu-free phases were observed in the melt unlike the case of undoped system. The composite with the $Sr_{1.7}Ca_{1.3}Al_2O_6$ surplus exhibited enhanced flux pinning at increased temperatures. In order to form dense materials with submicron inclusion homogeneous oxide glass precursors were prepared with the composition Bi-2212/$Sr_{1.7}Ca_{1.3}Al_2O_6$. Phase transformations of the glass precursor were established. It was noted that the temperature interval between the glass transition and glass crystallization greatly increased in comparison with the undoped Bi-2212 glass. The composite prepared by glass-ceramic route consisted of dense Bi-2212 with 0.1-0.3 μm inclusions of $(Sr,Ca)_3Al_2O_6$ particles. It was established that $BiSr_{1.5}Ca_{0.5}Al_2O_x$ and $(Ca,Sr,Pb)_2In_2O_x$ phases were in equilibrium with Bi-2223 superconductor. They were mostly concentrated between the superconductor grains, but random submicron particles were detected in Bi-2223 lamellas. Increase in the Bi-2223/Bi-2212 ratio and acceleration of the Bi-2223 phase formation were observed for the $BiSr_{1.5}Ca_{0.5}Al_2O_x$ doped samples. Two phase ceramics containing Bi-2223 and $BiSr_{1.5}Ca_{0.5}Al_2O_x$ was obtained.
Work was supported by Russian Foundation of Basic Research (project 97-03-33249a), Russian Ministry of Science (project Composite).

II3.5
OXYGEN DIFFUSION MECHANISMS IN $YBa_2Cu_3O_{6+c}$ BASAL PLANES.
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A Monte Carlo simulation allows us to obtain the most often realized diffusion mechanisms in 2D ordered structures, like for the diffusion of oxygen ions in 123-YBCO high temperature superconductor. Starting from several diffusion mechanisms we show to what extent they depend on the temperature and concentration of the diffusing particles. For YBCO, we introduce a new possible jump for the O-diffusion process in the basal planes. Our results are compared with the ones proposed earlier on the basis of energy arguments. We find additional trajectories, different from those earlier proposed, though sensibly as reasonable.

II3.6
CRYSTALLOGRAPHIC STUDY OF THE HIGH- AND LOW-TEMPERATURE FORMS OF $Bi_2(Sr,Ca)_4O_x$.
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During processing of high temperature superconductors in the Bi-Pb-Sr-Ca-Cu-O (BSCCO) system, $Bi_2(Sr,Ca)_4O_x$ frequently appears as an impurity. $Bi_2(Sr,Ca)_4O_x$ was found to be in equilibrium with the 20K, 80K, as well as the 110 K superconductors in the BSCCO system. Therefore crystal chemistry and structural characterization of this phase is deemed important for the study of the BSCCO superconductors. $Bi_2(Sr,Ca)_4O_x$ exists in both high- and low temperature forms. The high-temperature form of the composition $Bi_{34.0}Sr_{49.5}Ca_{16.5}O_x$ (prepared at 860°C, yellow) shows a complicated x-ray diffraction pattern as compared to that of the low-temperature one, which was obtained by annealing the high-temperature sample in oxygen at 620°C (black). Combined Rietveld refinements of the low-temperature oxidized form $Bi_{34.0}Sr_{49.5}Ca_{16.5}O_x$ using both neutron and x-ray diffraction showed it to be indexable on a distorted ABO_3 perovskite lattice. The structure was found to have a monoclinic space group $P2_1/n$, with a= 8.38917(7)Å, b=5.99436(5)Å, c=5.89592(5)Å and β=89.965(3)°. There are two independent B sites, one of which is mostly populated by Bi^{5+}, and the other about equally by Bi^{5+} and Ca^{2+}. The A site appears to be populated solely by Sr^{2+}. All Bi's adopt distorted octahedral environment. The high-temperature reduced form was found to be also monocline with a space group Pc, and a=11.0896 (15) Å, b=5.9734(5) Å, c= 19.8216(26) Å, and β=101.504(11)°. Single crystal x-ray study showed the structure consist of two types of Bi ions. The first type has distorted octahedral coordination while the other one only coordinate to three oxygens. The latter Bi's are located around broad channels with lone-pair electrons concentrated in these channels.

II3.7
A NOVEL 02(n-1)n HOMOLOGOUS SERIES IN THE Ba-Ca-Cu-O SYSTEM AND A DERIVATIVE WATER-CONTAINING HOMOLOGOUS SERIES.
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Two novel homologous series of multi-layered superconductive copper oxides were successfully established. The 02(n-1)n phases (n = 2 - 4) in the Ba-Ca-Cu-O system were obtained from high-pressure synthesis under oxidizing conditions. The derivative phases formed spontaneously from the highly unstable as-synthesized 02(n-1)n phases when exposed to humid air. For each parent 02(n-1)n phase, the original unit cell of body-centered symmetry was found to expand along the c axis by ca. 5.7 Angstroms upon the phase transformation. For the n = 3 case, it was successfully demostrated by wet-chemical analysis that the parent phase was more highly oxidized than its derivative phase. From HRTEM images and TG-MS data it was concluded that the lattice expansion was primarily due to incorporation of water molecules, probably involving also additional protons from a redox reaction between the highly oxidized 02(n-1)n phases and water. Based on ND data, the high oxidation state of the parent 02(n-1)n phases is explained by peroxide-type nature of the oxygen atoms in the BaO layers.

II3.8
LATTICE DINAMICS OF OXYGEN AND CATALYTIC PROPERTIES OF LAYERED LANTHANUM CUPRATES.
Galina

N. Mazo, Stanislav N. Savvin, Ivan A. Koudriashov, Oleg A. Shlyakhtin, Moscow State Univ, Dept of Chemistry, Moscow, RUSSIA.

Extensive study of HTSC materials revealed that layered perovskite-type cuprates possess a number of valuable properties. Wide homogenety region with respect to oxygen, high oxygen mobility caused by high concentration of vacancies in anion sublattice, fairly low activation energies of oxygen diffusion make rather promising their application as heterogeneous catalysts of various redox reactions. As reaction activation energies and other electrophysical characteristics of oxide materials depend strongly upon the oxygen content, nonstoichiometry control (e.g. by varying synthesys conditions) makes it possible to manage activity and selectivity of catalysts based on these compounds. Several La-Sr cuprates $La_{2-x}Sr_xCuO_{4-d}$ (x=0.15; 0.3;1) were synthesized using ceramic and freeze-drying methods. Preparation conditions have been optimized in order to enhance specific surface area of powders. Oxygen mobility in layered cuprates has been examined using dynamic-thermal isotope exchange method. The data obtained by this method allowed to select three temperature regions which can be attributed to several solid state and boundary processes. It was found that processes of completely heterophase exchange occur at 385-440°C for x=0.15 and 385-425°C for x=0.3. Partially heterophase exchange starts at 440°C for x=0.15 and at 425°C for x=0.3. For compositions corresponding to x=0.15 and 0.3 processes of out- diffusion and desorption begin at 490°C. Samples with x=0.15 demonstrated good catalitic activity in the reaction of methane oxidation. Kinetics of the process was studied in the temperature range 250-600°C. This work was supported by Ministry of Science and Technical Politics of RF, project No 139/97 RUS.

II3.9

CONTROL OF THE GROWTH MORPHOLOGY AND TRANSPORT PROPERTIES OF NdBCO LARGE GRAINS. Wai Lo[1], N. Hari Babu[2], D.A. Cardwell[2] and K. Salama[1]; [1]Texas Center for Superconductivity and Department of Mechanical Engineering, University of Houston, Houston, TX; [2]IRC in Superconductivity, University of Cambridge, Cambridge, UNITED KINGDOM.

Bulk $Nd_{1+x}Ba_{2-x}Cu_3O_y$ (NdBCO) large grains fabricated by melt processing have been demonstrated to have higher T_c, faster growth rate and superior flux pinning properties than YBCO, and hence have better potential for applications in high current devices. These materials, however, are also known to have complicated processing conditions and difficulties in growth morphology and superconducting properties control. Such difficulties are originated from the sensitive dependancy of properties on x, as well as the high melting point of NdBCO such that no seed with similar lattice structure and higher melt point could be found. This presentation focuses on the manipulation of growth morphologies of NdBCO large grains by additives and thermal properties of the precursor materials. The evaluation of superconducting properties by both transport and magnetization measurements and the comparison between these results will also be discussed in detail. These superconducting properties will be correlated with the growth morphology of the NdBCO grains.

II3.10

THERMODYNAMICS OF OXYGEN INTERCHANGE AND OXYGENATION KINETICS OF $Nd_{1+x}Ba_{2-x}Cu_3O_z$ SOLID SOLUTIONS. E.A. Trofimenko, High School of Materials Science, N.N. Oleynikov, Yu.D. Tretyakov, Chemical Department, Moscow State Univ., Moscow, RUSSIA.

In this work thermodynamic features of $Nd_{1+x}Ba_{2-x}Cu_3O_z$ were analyzed with respect to oxygenation kinetics. Thermodynamical treatment of system $Nd_{1+x}Ba_{2-x}Cu_3O_z$-O_2 was carried out using literature data. Oxygen absorption was considered as two processes: (1) complete oxygenation of compound with minimal oxygen content and (2) mixing of terminal compounds. Thermodynamics of mixing was calculated in a frame of subregular solution model. It was found that for substituted Nd123ss interval of oxygen content breaks into two intervals with different oxygen behaviour. Character of these changes can be explained by the holes transfer processes. For a further kinetic study the thermodynamic diffusion factor $W(T,x,z)=d\ln A_O/d\ln C_O$ was calculated through a wide range of temperatures. The kinetic study consisted in a TGA investigation of oxygenation process of quenched solid solutions using a linear heating regime with rates 2, 5, 10, 20 K/min in air. The data were processed according to the Merjanov's sheme to obtain quasi-isothermal α-$d\alpha/dt$ dependencies. Samples of solid solutions with different substitution degrees (x=0.05, 0.45, 0.6) were made by a ceramic route using Nd_2O_3, $Ba(NO_3)_2$, CuO. Precursors were annealed at 1000°C in air and quenched into liquid nitrogen as followed by XRD study (Guinier chamber, lattice parameters calculation), SEM (particle size evaluation), and iodometric titration. It was found that for a large substitution degree x oxygenation started at a lower temperature. As soon as thermodynamic factor is taken into account, it allowes us to extrapolate the arrhenius plot $\ln(d\alpha/dt)$-1/T and to perform further data processing to quasi-isothermal conditions. The oxygenation process model $d\alpha/dt=B_0*\exp(-E_A/RT)*W(T,x,z)$ was suggested and its parameters were calculated for the investigated solid solutions.

II3.11

PRESSURE STUDIES ON STRONTIUM BISMUTATES. M. Nunez-Regueiro, S. Sanfilippo, CRTBT/CNRS, Grenoble, FRANCE; C. Bougerol-Chaillout, P. Bordet, Lab. Cristallographie/ CNRS, Grenoble, FRANCE; S. Kazakov, J. Pshirkov, E. Antipov, Chem. Dept., Moscow Univ., RUSSIA; and M. Hanfland, ESRF, Grenoble, FRANCE.

The bismutate superconductors are a widely studied system due to their superconducting properties. Although the apparent disproportionation of Bi atoms in $BaBiO_3$ is suggestive of non-standard superconductivity, studies on $Ba_{0.6}K_{0.4}BiO_3$ have shown conventional electron-phonon superconductivity. However, contrary to cuprates, where the diversity of systems has been capital to their correct understanding, the research on the bismuth materials have been limited to the Pb and K substituted compounds. We have recently succeeded in synthesizing a third variety of bismutates[1] based on the new oxide $SrBiO_3$. By partial substitution of K for Sr, we found superconductivity with T_c=12K in $Sr_{1-x}K_xBiO_3$ (x=0.45-0.60). We report here on the pressure behaviour of both the structure and superconductivity of these new superconductors. We find strong evidence for the passage under pressure from an underdoped to an overdoped material, and as in cuprates superconductors the superconducting transition temperature follows a parabolic dependence with charge carriers density.

[1] S.M. Kazakov et al. Nature 390, 148 (1997)

II3.12

PARTIAL SUBSTITUTION OF COPPER BY TIN IN La(2)CuO(4): THE La(2)Cu(1-x)Sn(x)O(4+x) SOLID SOLUTION. Georges Denes, Glenn Taylor and Brent Thompson, Concordia University, Dept of Chemistry and Biochemistry, Laboratory of Solid State Chemistry and Mossbauer Spectroscopy, and Laboratories for Inorganic Materials, Montreal, Quebec, CANADA; Krzysztof Ruebenbauer, Institute of Physics and Computer Science, Pedagogical University, Cracow, POLAND.

One of the criteria for high temperature superconductivity in cuprates is the presence of excess oxygen in their structure. This occurs together with oxidation of some of the copper(+2) to the trivalent state. In the present work, we have carried out oxygen enrichment by substituting some of the copper(+2) by a higher oxidation state metal, namely tetravalent tin. Tin has the convenience of offering a powerful local probe, the tin-119 nuclide, which opens up the possibility of studying specifically the tin site(s) by Mossbauer spectroscopy. It was found that up to 12% of the copper could be substituted by tin to give a disordered La(2)Cu(1-x)Sn(x)O(4+x) solid solution. Mossbauer spectroscopy showed that the material contains two distinct tin(IV) sites, that are crystallographically very different. One is a moderately distorted [SnO6] octahedron, whereas the other is much more distorted. Nearly octahedral coordination is expected for tin(IV) in oxides, whereas the square pyramidal and the square planar coordinations commonly found for copper(II) are unheard of for tin(IV) in an oxide matrix.

II3.13

C-AXIS COPPER DISTORTIONS IN THE $YBA_2{}^{63/65}CU_3O_7$ SUPERCONDUCTOR BY THE ISOTOPE DIFFERENCE PAIR DENSITY FUNCTION. Despina Louca, G.H. Kwei, Los Alamos National Laboratory, Los Alamos, NM; B. Dabrowski, Z. Bukowski, Department of Physics, Northern Illinois University, DeKalb, IL.

The local atomic structure of the $YBa_2Cu_3O_{6.92}$ (YBCO) superconductor with isotopically pure ^{63}Cu and ^{65}Cu was studied, to investigate the nature of the lattice effects during the phase transformation. The analysis was done using the neutron differential pair density function (DPDF) technique. The DPDF provides an atom specific PDF, enabling the distinction between overlapping atom pair correlations in complicated systems such as the present one. It utilizes the contrast in the scattering amplitude enhanced by the different neutron scattering lengths for ^{63}Cu and ^{65}Cu in two isotopically pure samples with identical compositions. Evidence for coupling of the local lattice distortions to the superconducting transition is provided. Copper displacements in the superconducting planes are most likely the cause of the double Cu2 (planar copper) - O4 (apical oxygen) distance correlation observed in the vicinity of T_C. In addition, the bifurcation of the Cu2 - O4 pair correlations is temperature dependent. This effect originates from distortions along the c-axis of the crystal of the Cu2 ions and not of the O4, as no distortions are observed in association with the connecting pair, the Cu1 (chain)-O4.

Secondary lattice anharmonicities in the chains are also seen, but these arise mostly from defects in the vicinity of oxygen vacancies.

II3.14
CRYSTAL STRUCTURE AND LATTICE DYNAMICS OF $ND_{1+X}BA_{2-X}CU_3O_Z$ SOLID SOLUTIONS. Valery Petrykin, Masato Kakihana, Materials and Structures Laboratory, Tokyo Institute of Technology, Yokohama, JAPAN; Pedro Berastegui, Inorganic Chemistry Arrhenius Laboratory, Stockholm University, Stockholm, SWEDEN; Sten Eriksson, Department of Inorganic Chemistry, University of Goteborg, Goteborg, Studsvik Neutron Research Laboratory, Uppsala University, SWEDEN.

Structure and lattice dynamics of $Nd_{1+x}Ba_{2-x}Cu_3O_z$ solid solutions (x = 0.0-0.9) were investigated using high quality ceramic samples prepared via chemical solution route. Crystal structure of newly reported insulating compound $Nd_2BaCu_3O_z$ was established from powder X-ray and neutron diffraction data sets. It was shown that the superlattice appears as a result of Nd ordering in Ba sites of neodymium based solid solutions. The phonon assignment for this phase was carried out using results of lattice dynamics calculation based upon shell model for the refined crystal structure and experimental Raman spectra collected in different scattering geometry by micro setup. Structural information and Raman spectroscopy data were used for calculation of free energy of $Nd_{1+x}Ba_{2-x}Cu_3O_z$ solid solutions as a function of neodymium concentration at room temperature. In addition, from structural information, Raman spectroscopic data and chemical analysis it was assumed that possible scenario of superconductivity suppression in the neodymium based solid solution is associated with the redistribution of holes between different orbitals of Cu in conducting plane rather than between charge reservoir and conducting planes. This assumption was tested on another type of compound from the RE-123 family.

II3.15
HOMOGENEITY AREA OF $Bi_2Sr_2Ca_{1-x}R_xCu_2O_{8+d}$ (R = Nd, La) SOLID SOLUTIONS. A.V. Knotko, D.V. Korolyov, A.V. Garshev,V.I. Putlayev, Dept of Chemistry, Moscow State Univ, Moscow, RUSSIA.

Creation of precipitates in bulk matrix to act as pinning centers is a perspective way to improve critical current density in high temperature superconductors. It is possible to generate such precipitates via partial decomposition of supersaturated solid solution derived from a superconducting phase. To control this process one should go by a knowledge of stability field for these solutions. The objective of present work consisted in determination of homogeneity area of $Bi_2Sr_2Ca_{1-x}R_xCu_2O_{8+d}$ (R = Nd, La). Semi-quantitative XRD analysis was used to determine phase composition of specimens quenched from various temperatures. Annealing time appropriate to attain equilibrium in powder mixture was evaluated through kinetics measurements. It was found that lower temperature limit of stability field of Bi-2212 based solid solutions in multiphase mixture is 750° C. Monophase solid solutions exist in relatively narrow temperature interval. This interval is significantly larger in the case of Nd-doped Bi-2212. It stretches to x=0.45 while temperature is within 825° - 880° C. Solubility limit of Nd corresponds to x=0.75 at 850 C. In the case of $Bi_2Sr_2Ca_{1-x}La_xCu_2O_{8+d}$ solid solutions the maximum substitution was achieved at x = 0.3 and 875° C. Features of homogeneity fields for both solutions are discussed in terms of ionic radii of the dopants.

II3.16
USING SOFT X-RAY SPECTRA TO DETERMINE VALENCE STATE OF TRANSITION METALS IN THE ELECTRON MICROPROBE. Karoline Mueller, James K. Meen, Don Elthon, Univ of Houston, Dept of Chemistry and Texas Center for Superconductivity, Houston, TX; Gene Ulmer, Temple Univ, Dept of Geology, Philadelphia, PA.

The majority of methods for determining valence states of metals are indirect, macroscopic, or both. We describe an electron microbeam method of direct determination of the valence state of first-row transition metals. The method uses relative intensities of peaks in the soft L x-ray spectra of these elements. The pioneering work of Fischer (1964) showed that the Lβ- Lα intensity ratio is a function of the valence state for first-row transition elements. More recently, Hofer et al. (1994) applied a similar technique to Fe in which excitation was by electron microbeam. The measured Fe Lβ-Lα intensity ratios for wustite, magnetite, and hematite, and for the magnetite-hercynite spinel series strongly correlate with the average Fe oxidation state in each group. We obtained similar results on Fe oxides and on the chromite- picrochromite spinel series. The calibration curves obtained are, however, not applicable to all Fe-bearing oxides. Standard materials with similar chemical compositions and crystal structures to those of the unknowns must be used for calibration. The technique is applicable to all first-row transition metals. Examples of its application to oxides containing Mn or Cu will be emphasized.

Mixed-valence manganites are of special interest due to their colossal magnetoresistance. They are a prime candidate for application of L x-ray analysis as the crystal structure does not change with doping level and the chemical composition can be varied in a controlled manner. We have previously shown that liquids in the BSCCO system contain Cu(I) even though coexisting solids contain entirely divalent Cu. This observation is of great importance for the synthesis of phase-pure superconductors. Only by determining the valence state of Cu in BSCCO liquids can the full phase relations of these superconductors be unraveled. Cu L spectra for copper oxides and BSCCO glasses will be presented.

II3.17
LOCAL AND LONG - RANGE STRUCTURAL CHANGES IN THE ND1+XBA2-XCU3OZ HTSC SYSTEM. E.A. Goodilin, I.S. Bezverkhiy, Yu.D. Tretyakov, Inorganic Materials Lab., Chemistry Faculty of the Moscow State University, Moscow, RUSSIA; V.V. Petrykin, M. Kakihana, Materials and Structures Lab., Tokyo Institute of Technology, Yokohama, JAPAN; J. Hester, Australian National Beamline Facility, Tsukuba, JAPAN.

Study of the Nd1+xBa2-xCu3Oz system has led to great progress in preparation of HTSCs with advanced properties. At the same time, the system demonstrates a number of surprising features related to ordering/disordering phenomena at different x and z, which may lead to better control of bulk material properties. In the present work phase relations for the whole system Nd-Ba-Cu-O are studied by the XRD, DTA, EPMA, and AES ICP techniques, and special attention has been given to the typical structural changes occurring during the largest possible shift of x (0..1) and z (0..1) values in the series of light rare-earth REE1+xBa2-xCu3Oz compounds (REE=La, Nd, Sm, Pr) as probed by X-Ray, neutron, electron diffraction, EXAFS, Raman scattering spectroscopy, Mossbauer spectroscopy, and susceptibility measurements. Possible structural transitions at x 0.3 and x 0.6 within the homogeneity field of Nd1+xBa2-xCu3Oz are discussed in the framework of Nd and Ba atomic ordering and a subsequent oxygen sublattice reconstruction. New anomalous examples of a disordered low-Tc quasicubic NdBa2Cu3Oz phase and a superstructural ordered orthorhombic Nd2BaCu3Oz compound, both with no tetra-ortho-transition, are analyzed on the basis of structure refinement and the behavior of Cu-O, Nd-O and Nd-Cu polyhedra in the compounds during oxygenation. The oxygen nonstoichiometry, a charge transfer model and superconducting properties have been determined for the Nd1+xBa2-xCu3Oz phase as well. Finally, interrelations of the structural evolution and physical properties are reconsidered to improve the possibility of high-quality rare-earth-containing HTSC production. This work is suported by RFBR (98-03-32575a). Part of this work was performed at the Australian National Beamline Facility with support from the Australian Synchrotron Research Program, which is funded by the Commonwealth of Australia under the Major National Research Facilities Program.

II3.18
THERMAL ANALYSIS AND THERMODYNAMICS OF (Ca,Sr)-PLUMBATES. L.P. Cook, R. Klein and W. Wong-Ng, NIST, Gaithersburg, MD.

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

II3.19
PHASE EQUILIBRIA AND CRYSTAL CHEMISTRY OF THE SrO-R_2O_3-CuO SYSTEMS, R=LANTHANIDES. W. Wong-Ng, J. Dillingham, T. Haugan, Q. Huang, L.P. Cook, H. Brown, MSEL, NIST, Gaithersburg, MD; X.L. Chen, Chinese Academy of Science, Beijing, CHINA.

In the past decade, a great deal of phase equilibrium research has been conducted on the BaO-R_2O_3-CuO systems (R=lanthanides and Y) due to the presence of high-temperature superconductors such as $Ba_2RCu_3O_{6+x}$, $Ba_2RCu_4O_x$ and $Ba_4R_2Cu_7O_x$. Substitution of the alkaline earth element Ba by Sr may provide further insights regarding the crystal chemistry and phase equilibria of the high T_c phases. Although the analogs of the high T_c phases as mentioned above were not found in these Sr-systems under ambient pressure, the Sr-doped $Sr_{14}Cu_{24}O_{41}$ phase has been reported to have interesting properties, such as anomalous microwave and magnetic properties, and $Sr_2RCu_3O_x$ can become superconducting under high oxygen pressures. This paper investigates the trend of phase formation, and the crystal chemistry of various series of compounds, and subsolidus

phase relationships in the SrO- R_2O_3-CuO systems. A comparison of these phase diagrams with the Ba-analogs will also be made.

II3.20
BOND-VALENCE ANALYSIS OF $YBa_2Fe_3O_8$. Isabella Natali Sora, INFM and University of Brescia, Dept of Mechanical Engineering, Brescia, ITALY; Anthony Santoro, Qing Huang, NIST, Center for Neutron Research, Gaithersburg, MD.

The nuclear and magnetic structures of $YBa_2Fe_3O_8$ have been determined from neutron powder diffraction data (Q. Huang et al., Phys. Rev. B 45, 9611 (1992)). The nuclear structure was found to have an atomic configuration very similar to that of $YBa_2Cu_3O_x$. Bond valences were calculated from the observed bond lengths using the formalism and the bond valence parameters of Brown and Altermatt (Acta Cryst. B 41, 244 (1985)) and Brese and O'Keeffe (Acta Cryst. B 47, 192 (1991)). The results of these calculations showed significant departures of the bond valence sums of some atoms from the expected values (e.g. V(Ba) = 2.5 v.u. rather than 2.0 v.u., and V(Y) = 2.6 v.u. rather than 3.0 v.u.). Since the structure was determined very precisely, with excellent agreement between observed and calculated intensities, these discrepancies cannot be attributed to errors in the experimental results, but rather may be due to structural strains. The bond valence analysis confirms this conclusion. In fact, the bond distances calculated from the theoretical bond valences (I.D. Brown, Zeit. Kristall. 199, 255 (1992) ; M. O'Keeffe, Structure and Bonding 71, 161 (1989)) are incommensurate under the constrains imposed by the site symmetries of the atoms. Under these conditions the presence of structural strains is unavoidable. In the actual structure the atoms occupy positions that minimize these strains and resulting in coordination polyhedra of the cations significantly stretched or compressed.

II3.21
ELECTRICAL TRANSPORT PROPERTIES OF BIPOLARONIC Ti_4O_7 UNDER HIGH PRESSURE. Miguel Monteverde, Carlos Acha, Laboratorio de Bajas Temperaturas, Dept de Física, FCEYN., Univ de Buenos Aires, Buenos Aires, ARGENTINA; Manuel Núñez-Regueiro, CRTBT, CNRS, Grenoble, FRANCE; Alois Kuhn, Facultad de Ciencias Experimentales y Técnicas, Univ San Pablo Boadilla del Monte, SPAIN; Miguel A. Alario Franco, Facultad de Ciencias Quimicas, Univ Complutense, Madrid, SPAIN.

Motivated by the discussion about the bipolaronic nature of high T_c superconductivity, we have studied the electrical transport properties of Ti_4O_7, whose carriers, for temperatures below a metal-insulator transition, were identified as bipolarons. We measured the resistivity of Ti_4O_7 twinned single crystals as a function of temperature (R(T)) for a pressure span (P) up to 230 kbar. We studied current-voltage (I-V) characteristics applying both a conventional DC and a pulsed technique. Depending on current direction and for $P > 20$ kbar we observed a semiconducting-like behavior with a $\ln \frac{1}{T}$ and a $\ln \frac{1}{V}$ dependence at low temperatures, or a metallic conductivity with a characteristic carrier interacting T^2 law. A phenomenological relation was established to describe our results, that are discussed within the framework of electrical transport of (bi)polarons scattered whether by disorder or by interacting with other carriers.

II3.22
MAGNETIC CONTRIBUTIONS TO MICROWAVE LOSSES IN CUPRATE SUPERCONDUCTORS. S. Sridhar, Z. Zhai, N. Hakim, P.V. Patanjali, C. Kusko, Northeastern University, Department of Physics, Boston, MA.

The origin of the absorption of microwaves is of importance from fundamental reasons as well as for applications. Despite the very strong evidence suggesting a d-wave order parameter from a variety of experiments, the microwave data are not explainable in their entirety in terms of a pure d% -wave order parameter, or even one with mixed symmetry. We have carried out extensive measurements on a variety of cuprate (superconducting and non-superconducting), nickelate and manganite oxides. An important theme that emerges from the data is the presence of peaks in the microwave absorption with varying temperature. The presence of similar peaks in superconducting and non-superconducting materials strongly suggests that they are magnetic in origin. This is because the parent compounds for the cuprates and nickelates are anti-ferromagnetic insulators. Hole doping leads to an inhomogeneous state with separation into magnetic and charge stripes. The formation of stripes does lead to peaks in the absorption in $La_{\frac{5\%}{3}}Sr_{\frac{1}{3}}NiO_4$. These results therefore suggest that in the superconductors, the superconducting contribution is overwhelmed by a magnetic contribution. Support for these observations from measurements on $\%YBa_2Cu_3O_{7-\delta}$ with varying O concentration and in $Hg:1223$ and $Hg:1201$ is discussed. We thank A. Erb (U. of Geneva), A. Revcolevschi (U. Paris-Sud, Orsay), D. Colson (Saclay), A. Maignan (Caen) and S. Cheong for providing samples used in this work. Work supported by NSF-9711910 and AFOSR-5710000349.

II3.23
FERROMAGNETISM AND HIGH Tc SUPERCONDUTIVITY IN RuSr2GdCu2O8. D.Z. Wang, S.X. Yang, Y. Tu, Z.F. Ren, J.I. Oh, Y. Sun, H.I. Ha and M.J. Naughton, Boston College, Dept of Physics, Chestnut Hill, MA; Michael De Marco, SUNY at Buffalo, Dept of Physics, Buffalo, NY and Buffalo State College, Buffalo, NY; Jingyu Lao, J.H. Wang, SUNY at Buffalo, Dept of Chemistry, Buffalo, NY; D.T. Verebelyi, M. Paranthaman, T. Aytug, D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN; Michael Haka, Steve Toorongian, SUNY at Buffalo, Dept of Nuclear Medicine, Buffalo, NY.

Bulk samples of the presumed hybrid ferromagnet-high Tc superconductor RuSr2GdCu2O8, with different Tc's, have been synthesized by solid state chemical reaction. TEM and powder XRD show that the RuSr2GdCu2O8 compound is the dominate phase. The highest zero-resistance temperature measured via four-probe resistivity is close to 40K. 99Ru Mossbauer effect measurements at 4K reveal a hyperfine magnetic field of around 70T. This is the first use of the Ru Mossbauer effect without any additional impurity to demonstrate an internal magnetic field in a high Tc superconductor.

II3.24
RIETVELD REFINEMENTS OF (R0.5Y0.5)Ba2Cu3O7-X (R=HO,DY, GD, EU, SM,ND). Eric J. Peterson, William L. Hults, Michael Simpson, Stephen R. Foltyn and Quanxi Jia, Los Alamos National Laboratory Superconductivity Technology Center, Los Alamos, NM.

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

II3.25
STRUCTURAL CHANGES IN $YBa_2Cu_3O_6$.3 AND $YBa_2Cu_3O_7$.0 DUE TO PHOTODOPING. Lamine Dieng, Trevor A. Tyson, New Jersey Institute of Technology; Steven Tidrow, Army Research Laboratory.

$YBa_2Cu_3O_{6+x}$ (YBCO), is known to undergo changes in resistivity when exposed to optical radiation. However the origin of the changes in transport are not well understood. In previous work [1] a local distortion of CuO_x chains was found to coincide with hole transfer seen in the absorption spectra of the photodoped (x=0.4) system. We have performed a systematic x-ray absorption study of samples in the insulating and superconducting regions. We find significant structural changes in samples from both regions- including the superconducting region. Structural parameters (bond lengths and bond correlations) are extracted to give a picture of the photoinduced structural deformations. This research is funded by NSF CAREER grant DMR-9733862 [1] Tyson *et al.*, Physica C 292, 163 (1997).

II3.26
THE INFLUENCE OF ANNEALING ON SUPERCONDUCTING GLASS OF THE Bi-SYSTEM OBTAINED BY MELTING-QUENCHED METHOD. Cláudio Luiz Carvalho, Keizo Yukimitu, Victor C.S. Reynoso, João C.S. Moraes, Universidade Estadual Paulista, Dept of Physics and Chemistry, Ilha Solteira, SP, BRAZIL; Paulo N.L. Filho, Group of Superconductivity, Universidade Federal de São Carlos, Dept of Physics, São Carlos, SP, BRAZIL.

Bi-system superconducting have been studied intensively because presents crystallographic phases with differents transition temperature. 2201, 2212 and 2223 are the principal phases with critical temperatures around 30K, 85K and 110K, respectively. The most important between them is 2223, but this phase require long heat treatments, it is unstable and to solve this problem it is necessary to add Pb in the composition. Then, the ideal composition is known as BPSCCO (1.6;0.4;2;2;3). Differentsmethods have been used to obtain this material like conventional, coprecipitation, evaporation method, etc, but all of them spend too much time mixing oxides, exist

problems with the stoichiometrymainly with Pb because it is a volatile element. In this work, we used glass route and controlled atmosphere to obtain the 2223 phase. After obtain a kind of vitreous ceramic material and submitted it at differents thermal treatments, X-ray diffraction, energy dispersive X-ray, electric and magnetic measures were used to characterize it and we have observed the presence of 2212 and 2223 phases for short and long time annealing, respectively.

II3.27
LOW SUPERCONDUCTING ANISOTROPY AND HIGH DIMENSIONALITY OF (Cu,X)-12n(n+1). Yasumoto Tanaka, Kosuke Tanaka, Akira Iyo, Madoka Tokumoto, Hideo Ihara, Electrotechnical Laboratory, Tsukuba, JAPAN and CREST of JST; Takeyo Tsukamoto, Science Univ of Tokyo, Dept of Applied Physics, Tokyo, JAPAN; Minoru Ariyama, Science Univ of Tokyo, Dept of Physics, Noda, JAPAN; Shiko Miyashita, Kazuyasu Tokiwa, Nobuya Ichioka, Tsuneo Watanabe, Science Univ of Tokyo, Dept of Applied Electronics, Noda, JAPAN and CREST of JST.

The anisotropy and the dimensionality of the copper based cuprate superconductor (Cu,X)-12n(n+1) was estimated by the normal state conductivity, temperature dependence of H_{c2}, torque measurement and flux-flow resistivity where X is Tl or C or vacancies or etc. The latter three among the four techniques had given the superconducting anisotropy. The estimated anisotropy was slightly varied between $\sim$ 1.6(in the case of applying the second technique to Cu-1234) and $\sim$ 12 (in the case of applying the third technique to (Cu,Tl)-1223) depending on X and the techniques. The origin of the discrepancy and the reliability of the values are discussed because there are some conditions like the sample dimensions and strength of the applied field to give the reliable value. We pay attention to the relationship between the low anisotropy (and high dimensionality) and the properties of the charge reservoir layer. We discuss if the superconducting pair potential works between the holes resident at different CuO_2 sheets or different superconducting blocks.

II3.28
CHARACTERIZATION OF SUPERCONDUCTING MATERIALS AND THEIR CRYSTAL AND CHEMICAL FEATURES USING STANDARD REFERENCE MATERIALS. Boris N. Kodess, V.K. Ovcharov, L.A. Butman, I.L. Kommel, VNIIMS-Gosstandart of Russia, Moscow, RUSSIA; ICS&E Department of Crystal Metrology; E.M.Roysenblatt, Division of Electron Materials, Donetsk, UKRAINE.

There is a strong relation between physical properties and composition within the homogeneity region for both conventional (for example A15 and C15 compounds) and new high-T_c superconductors. Samples of the same composition of HTSC materials were measured dozens times. But the results were usually different even within one institution for macro and micro ranges of the length scale as well as for measurements of atom structure and electron density distribution. To minimize typical errors an interlaboratory experiment has been conducted on measurement of chemical composition, interplane distances, lattice parameters and intensity ratio for a set of pure elements and compounds uncluding supeconducting silicide vanadium and yttrium cuprates. The X-ray data has been collected from a number of set-ups (12-16 laboratories) using single crystals and powder X-ray and neutron radiation. An experiment was conducted also for powder samples of Si-V and Y-Ba-Cu-O systems to determine their composition (mass ratios of major components and impurities) using various physical and chemical methods (more 30 methods of analysis). Such Standard Reference Materials and associated reference X-ray patterns provide high accuracy and establish basis for reproduction of experimental results. For example, details of kinetics and phase evolution of melt-textured samples were investigated for YBCO with high I_c and T_c. Precision experiment data yields fine details of distribution and/or arrangement of atoms of components of these materials, including sublattice of vacancies. As a result of series of experiments we established chemical tendencies of changes in properties if changes in composition or special treatment or external influence are applied. Also electron densities distributions, charge transfer and atomic thermal vibration for each atoms for all crystallographic positions of stoichiometric and non-stoichiometric V_3Si and $YBa_2Cu_3O_{6+x}$ have been determined from diffraction data with a higher level of precision.

II3.29
PHASE TRANSFORMATION AND DISTRIBUTION IN MECHANICALLY DEFORMED BSCCO SUPERCONDUCTOR TAPES. Sean Li, Nayang Technological Univ, Dept of Materials Engineering, SINGAPORE; Hoani Cooper, Wei Gao, Auckland Univ, Dept of Chemical and Materials Engineering, NEW ZEALAND.

Mechanical deformation has widely been used to produce textured microstructure to achieve high critical current density in the Bi2223 superconducting tapes. But the effect of mechanical deformation on the phase transformation in the BSCCO system has not been well understood. In the present research, the effects of mechanical deformation on the kinetics of Bi2212 to Bi2223 phase transformation have been investigated in detail. The results showed that the Bi2223 phase abundance decreased with increasing mechanical deformation (thickness reduction) from 20-60%, and then increased with further increasing deformation ratios above 60%. The 60% deformation produced the lowest abundance of the Bi2223 phase. These phenomena can be explained with the different deformation energies that are dominant in the different deformation process. In the relatively low mechanical deformation regime (up to 60%), an increase in the deformation ratio results in a higher interface energy that is against the nucleation of the Bi2223 phase. In the relatively high mechanical deformation regime (above 60%), an increase in deformation provides higher strain energy which acts as a driving force, increasing the nucleation and phase formation of the Bi2223 phase.
The effects of mechanical deformation on the distribution of Bi2223 phase abundance across the thickness of the tapes were also studied. The phase profiles were different for the tapes with different deformation ratios. For the tapes with high deformation ratios, the Bi2223 phase abundance is maintained to a high level from the surface to a certain depth; while the concentration of the Bi2223 phase dropped sharply to a low level underneath the surface in the tapes with a low mechanical deformation. It is suggested that the Bi2223 phase profile is related to the grain alignment distribution and the density of the materials in the tape.

II3.30
MAGNETIC ARRANGEMENT AND REVERSIBLE MAGNETISATION IN PLANE MAGNETIC FIELD OF $Bi_{2.1}Sr_{1.9}Ca_{1.0}Cu_{2.0}O_x$ SINGLE CRYSTAL. G.D. Gu, Advance Electronic Materials Group, School of Physics, University of South Wales, Sydney, AUSTRALIA; R. Puzinik, Institute of Physics, Polish Academy of Sciences, Warszawa, POLAND; N. Koshizuka, Superconductivity Research Laboratory International Superconductivity Research Center, Tokyo, JAPAN.

One of the most important intrinsic properties of high Tc Bi-system superconducting materials is its extremely high anisotropy. The magnetic properties in the plane of the crystals has not been known for lack of the large size single crystals. The large, high quality single crystals of Bi-2212 phase superconductors have been grown by floating zone method. The field dependence of magnetisation of the Bi-2212 single crystal of 5*3.6*0.4 mm^3 and 5.5*5*1.3 mm^3 were studied in-plane magnetic field by a 7 tesla SQUID magnetometer (Quantum Design) equipped with the sample rotation system. One unusual phenomenon observed in the experiment is that thc absolute value of magnetisation in a wide field range of $H_{c1}<=H<=H_{c2}$ increases with increasing field. We compare the above results with several proposed theory models and find that the results is agreement with the laminar structure approximation theory for the magnetisation distribution inside superconductor. We also discover that the negative susceptibility under a high external field of 7 tesla does not vanish up to temperature as high as the temperature of 140 K, which is significantly higher than the bulk superconducting transition temperature Tc=88K of the crystal. The high field negative susceptibility above the superconducting transition temperature Tc indicates that the superconducting transition temperature in the layered cuprate is affected by a peculiar interplay between doping and pair breaking scattering.

II3.31
RADICAL CHANGE IN THE APPROACH TO SEMICONDUCTOR-METAL-SUPERCONDUCTOR TRANSITION IN SOLIDS. Valery P. Kisel, Institute of Solid State Physics, Chernogolovka, RUSSIA.

Charged ion conductivity, electroplastic and electrical breakdown effects evidenced for the crusial role of deformation mechanisms in the electric properties of crystalline (CS) [1] and organic (OS) [2] solids. This approach allows one to describe the insulator-metal-superconductor transition (IMST) through the increase of crystal work-hardening (WH) which decreases the plastic-deformation losses for nucleation and motion of charges thus lowering the electrical resistivity of CS, OS [1]. The works [2-5] directly evidenced for the strict correlation between the mechanical (WH, structural instabilities) and electrical properties of solids. The same features of the motion of deformation units in CS [6] and OS [7] confirm the universality of WH-mechanisms in CS and OS. The change of resistance in strained (due to hydrostatical pressures, inhomogeneous phases, compacted powder, etc.) samples closely varies with the hardening of soft crystals and the softening of hardened samples [2-8]. The sharp increase of WH at low temperatures means the sudden decrease in electrical resistivity (the IMST), the higher WH of crystal prompts the higher transition temperatures of low-temperature WH abrupt rise and of the insulator- metal-superconductor transition and vice versa. The remarkable finding of this work is the scaling of mechanical [9-10] and electrical parameters of superconductors in

different scales of observation, which confirms the new approach to IMST.

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II3.32
Abstract Withdrawn.

II3.33
THE NONCONTACT METHOD OF CRITICAL CURRENT DENSITY MEASUREMENT IN HTSC SINGLE CRYSTALS.
Kh. R. Rostami, Institute of Radio Engineering & Electronics, Moscow, RUSSIA.

One of the central tasks at synthesis and further applications of HTSC single crystals in the applied purposes is the maximum precise and quick measurements of their critical currents. We design a simple method for a critical current density determination in HTSC single crystals of the arbitrary form, according to which the J_c value is determined on measurements of axial allocation of an entrapped magnetic field apart from a sample. According to our research which have been carried out on $YBa_2Cu_3O_{7-\delta}$ and $Bi_2Sr_2CaCu_2O_{8+\delta}$ single crystal samples of the various forms and sizes, density of a critical current in them can be exactly defined from the expression.
$J_c=(c/\pi h)(B_p\text{-}B_{c-1})/Arsh\ (R/h)$
where B_p - the value of an external magnetic field B, at which B_{tr} reaches saturation, B_{c1}- the first critical magnetic field of a sample, h- the thickness of a sample, R - reduced radius of a sample and c - the speed of light. The value B_p was determined from the dependence B_{tr} on B for the mode ZFC and B_{c1} was determined at the moment of detection of a non-zero B_{tr} signal by the sensor located near to a central part of the sample surface. In the measurements we utilized the Hall sensor with a size of the working area 0.15 on 0.45 mm with sensitivity 10 mkV*Gs^{-1}. The compensating circuit allowed us to register fields with 10^{-2} Gs accuracy. Usage of more responsive sensor of a smaller size will allow to raise accuracy of definition of the value J_c.

II3.34
Abstract Withdrawn.

II3.35
MODEL FOR SUPERCONDUCTOR STRIP SURFACE-BARRIER AT A LOW FIELD.
D. Agassi and R.J. Cullen, Naval Surface Warfare Center, Carderock Division, Bethesda, MD.

Flux pinning and entry/exit to/from a thin film in the presence of a weak magnetic field is controlled by the film's surface barrier. This barrier depends on the film's intrinsic physical parameters and, more importantly, on its geometrical dimensions. For the one-dimensional configuration of a field parallel to a semi-infinite superconductor planar surface, this barrier has been discussed a while ago[1]. On the other hand, for a thin supercondutor film, where the external-field field-lines wrapped around the film imply a two-dimensional geometry, the analysis is more involved. By adopting a new approach, a divergence-free expression is obtained for the underlying screening current. The results are compared to alternative approaches which entail a divergence at the film's edge[2]. With regard to the screening current distribution (at the film's surface), comparison with the 'canonical' result[2] shows that the new approach compares well with the latter provided the evaluation point is not too close to the film's edges. The associated surface barrier, however, entails a new qualitative feature which will be presented and discussed. A corollary is an expression for the film's vortex penetration-field and the total screening current carried by half of the film.

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II3.36
INDUCED SUPERCONDUCTIVITY AT LONG DISTANCE AND PROXIMITY EFFECT IN SNN′ STRUCTURES.
A. Gama Goicochea, M.J. Holcomb and W.A. Little, Stanford University, Stanford, CA.

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

II3.37
EFFECT OF SINTERING ATMOSPHERE ON THE WEAK LINK BEHAVIOUR OF YBCO SUPERCONDUCTORS.
Lokesh Chandra Pathak, S.K. Das, S.K. Mishra, National Metallurgical Laboratory, Jamshedpur, INDIA.

The transport properties of the superconducting materials are affected by the weak-link characteristics of the grainboundaries. The weak-link behaviour has been investigated extensively during last one decade. However, several aspects of the weak link formation is not been studied. In this paper the role of sintering atmosphere on the weak-link characteristics of bulk YBCO superconductors is discussed. The pellets were sintered at 960°C for 1h in argon, air and oxygen atmosphere and subsequently oxygenated at 500°C for 20h and cooled down to room temperature at the rate of 1°C/min. The weak-link behavior has been studied by analyzing the critical current densities near the transition temperature. A detailed investigation of the chemical composition across the grain boundaries was carried out to understand the weak-link behavior. The variation of critical current densities near the superconducting transition temperatures indicated that the weak-link characteristics were changed from S-I-S to S-I-N-S and S-N-S as the sintering atmosphere varied from argon to air and oxygen respectively. As discussed earlier, SEM/EDS studies were carried out in the samples to understand the weak link behavior which showed a continuous variation of composition across the grainboundaries when the samples were sintered either in argon or air. Whereas, no variation of chemical composition was observed in the samples sintered in oxygen atmosphere. The widths of the grain-boundaries were also found to increase with the decrease of pO2 in the sintering atmosphere. The EDS analyses indicated the presence of excess Ba and Cu in the grainboundaries of the samples sintered in air and argon atmosphere. From our earlier studies it was known that the sintering is enhanced with the decrease of pO2. The possibility of the formation of Ba-Cu rich liquid phase at the grainboundaries was possibly the cause for the enhanced sintering of the samples and weak link characteristics of the samples.

II3.38
SINTERING CHARACTERISTICS OF DOCTOR BLADE CAST YBCO-Ag TAPES.
Lokesh Chandra Pathak, S.K. Mishra, National Metallurgical Laboratory, Jamshedpur; D. Bhattacharya, Materials Science Centre, Indian Institute of Technology, Kharagpur; K.L. Chopra, Thin Film Lab. Dept of Physics, Indian Institute of Technology, Delhi, INDIA.

The fabrication and subsequent processing of high temperature superconducting powders is an integral part for its possible application in industries. Amongst the several techniques, tape casting of ceramic powders by doctor blade is important and has been investigated extensively during the last one decade particularly the Y-Ba-Cu-oxide superconductors. However, little effort has been made to study the sintering characteristics of these doctor blade cast tapes. In this paper, the binder removal, densification and grain-growth characteristics of YBCO-Ag (10 mol%) has been discussed. Submicrometre sized YBCO(123)-Ag (10 mol%) powder was prepared by a solution combustion technique using a chemical pyrophoric reaction. The powder was calcined and slurry was prepared by adding binder, plasticiser and defloculating agent. The slurry was deaired and subsequently tape cast by a doctor blade technique. The decomposition characteristics and the dimensional changes were investigated using thermal analyser and thermomechanical analyser (TMA). The tapes were sintered for 10 h at temperatures ranging from 600 to 940°C. The average grain sizes of the tapes after sintering were also measured. The superconducting properties of the sintered tapes were measured after oxygenation at 500°C for 20h. Thermal analyses and TMA studies indicate that the binder removal from the tapes leads to shrinkage of the tapes. The generation of volatile gases during binder removal and the subsequent flow of these gases through capillaries has lead to shrinkage of the tapes. Anisotropic shrinkages

of the tapes during binder removal is also observed. The sintering studies of the YBCO-Ag tapes showed two stages of sintering and grain growth behaviour. The apparent activation energy of sintering has been estimated to be 57±1 kJ/mol below 800°C and 327 ± 6 kJ/mol above 800°C. Whereas, The apparent activation energy for grain growth is estimated to as, 252±1 kJ/mol below 900°C and 2317±45 kJ/mol above 900°C. The grain growth has been observed to enhance after attaining 80% of the theoretical density.

II3.39
COMPARISON OF THE MICROSTRUCTURE TO THE CRITICAL CURRENT DISTRIBUTION UNIFORMITY ACROSS JOSEPHSON JUNCTIONS. Michael W. Carmody, Argonne National Laboratory and Northwestern University; K.L. Merkle, Argonne National Laboratory; L.D. Marks, Northwestern University.

Critical current vs. applied magnetic field measurements were measured across 24 degree, 45 degree and interface engineered ramp-edge YBCO Josephson junctions. The critical current distributions along the length of the boundary were calculated using a phase retrieval algorithm. The uniformity of the various current distributions are discussed in terms of microstructure features along the boundary analyzed by high resolution electron microscopy.

II3.40
MEASUREMENT AND ANALYSIS OF HARMONIC POWER FROM $YBa_2Cu_3O_{7-\delta}$ MICROWAVE RESONATORS. Durga P. Choudhury, Physics Department, Northeastern University, Boston, MA and Air Force Research Laboratory, Hanscom Air Force Base, Bedford, MA; John S. Derov, Air Force Research Laboratory, Hanscom Air Force Base, Bedford, MA; S. Sridhar Physics Department, Northeastern University, Boston, MA.

Harmonic generation measurements as a function of input power and temperature were carried out on five different suspended microwave resonators patterned out of high quality $YBa_2Cu_3O_{7-\delta}$ film on a $LaAlO_3$ substrate with a fundamental frequency of about 4.3 GHz. The third harmonic power P_3 shows a subtle nonlinear dependence on the fundamental power P_1 on a log-log scale. Fit to a straight-line yields slopes of P_3 against P_1 between 1.5 to 3, contrary to elementary calculations that predict a slope of exactly 3. It is shown that power dependence of the insertion loss and third harmonic power generated from higher order terms in the nonlinear impedance could account for the discrepancy quantitatively without resorting to any additional ad-hoc assumptions. Results of measurement of fifth harmonic power and power dependence of surface reactance are presented as confirmations of the conjecture. A small second harmonic power is also observed that does not show anomalous power dependence within the experimental accuracy. The temperature dependence of the harmonic power, however, does not seem to be describable by any of the existing theories. Relationship of the data to material parameters will be discussed along with various nonlinear phenomenological and microscopic models for high temperature superconductors. Work supported by NSF-9711910 and AFOSR-5710000349.

II3.41
EFFECTS OF He IRRADIATION ON THE MIXED STATE OF 123 SUPERCONDUCTING THIN FILMS. D. Arias, Dpto. Fisica Aplicada III, Univ. Complutense, E.M. Gonzalez, E. Luna, Dpto. Fisica de Materiales, Univ. Complutense; Z. Sefrioui, J. Santamaria, Dpto. Fisica Aplicada III, Univ. Complutense, J.L. Vicent, Dpto. Fisica de Materiales, Univ. Complutense, Madrid, SPAIN.

Irradiation with high energy heavy ions is a powerful and well known tool to study the mixed state, mainly phenomena related with pinning. The effect of irradiation by lower energy and lighter ions produces a very different scenario. In this case, the main effect could be due to Oxygen disorder, mainly in the chains. The $YBa_2Cu_3O_7$ films were obtained by dc magnetron sputtering in pure Oxygen pressure (3.4 mbar) on (100) $SrTiO_3$ substrates, with film thickness of 50 nm. The films were irradiated with He ions, using a microelectronic implanter, at a dose of 4 10^{14} cm^{-2} and 80 keV, at 7° to avoid channeling. Due to the irradiation the T_c is decreased from 90 K up to 62 K. (I,V) and resistivity vs. the angle (between the substrate and the applied field direction) curves were measured in the mixed state with applied magnetic fields up to 90 kOe. The anisotropy behavior and critical scaling laws obtained from the data are compared with the results in Oxygen deficient and fully oxygenated films.

II3.42
POWER HANDLING IN AG-DOPED YBCO: MICROWAVE MICROSCOPY AND DEVICE MEASUREMENTS. Hans M. Christen, K.S. Harshavardhan, S.D. Silliman, Neocera, Inc., Beltsville, MD; Wensheng Hu[1], A.S. Thanawall, B.J. Feenstra[2], F.C. Wellstood, and S.M. Anlage, Center for Superconductivity Research, University of Maryland, College Park MD; [1]present address: Hughes Network Systems, Germantown, MD; [2]present address: Philips Research Laboratories, Eindhoven, THE NETHERLANDS.

The effect of silver doping on the power handling capacity of $YBa_2Cu_3O_{7-x}$ thin films is investigated by both microscopic (imaging) and macroscopic measurement (device characterization) techniques. Coplanar resonators (5.5 GHz) are fabricated using PLD-grown $YBa_2Cu_3O_{7-x}$ targets with different doping levels of silver. Nonlinearitites are characterized through intermodulation distortion (IMD) of two nearby pure tones. Spatially resolved IMD measurements are performed using a cryogenic scanning near field microwave microscope, and the results are compared to the macroscopic (device level) measurements. The influence of the silver doping is studied for both types of IMD observations.

II3.43
THE KOSTERLITZ-THOULESS TRANSITION IN ULTRATHIN YBCO FILMS. Igor Grekhov, Lyuba Delimova, Ivan Liniichuk, Ivan Veselovsky, Ioffe Inst, RAS, Dept of Solid State Electronics, St-Petersburg, RUSSIA.

One of the possible factors responsible for the observed depression of the critical temperature in ultrathin YBCO films can be the Kosterlitz-Thouless (KT) transition associated with the thermal dissociation of vortex-antivortex pairs above a temperature T_{kt}, below the mean-field temperature T_{co}. To study the KT transition, we have made experiments on 6nm-thick YBCO films deposited by laser ablation on a $SrTiO_3$ substrate (YBCO/STO) and on an YBaCuNbO buffer layer (YBCO/buffer). The YBaCuNbO buffer [1] allows an increase in the critical temperature of a 6nm-thick YBCO film to a value above 77K. The analysis of the measurements showed that the current-voltage characteristics and the resistive transitions follow the KT prediction for both the YBCO/STO and the YBCO/buffer. The comparision between the theory and the experiment allowed us to calculate the superconducting carrier density, the vortex dielectric constant, and the effective penetration depth. The KT parameters were found to be T_{kt}=34.8K and T_{co}=55K for the YBCO/STO and T_{kt}=75.5K, and T_{co}=80.2K for the YBCO/buffer. A transition width gives information about the changes of vortex-antivortex interaction with pair scale. Significant difference in T_{kt} values for the YBCO/STO and the YBCO/buffer we believe to indicate the absence of the vortex pairs with a large separation distance in the YBCO/buffer; so pair breaking starts at higher temperature than for the YBCO/STO. Indeed, an ultrathin YBCO deposited on YBaCuNbO buffer we found experimentally to have a lot of voids with a typical distance among them of about 100 nm. That appears to prevent the bounding of the vortex-antivortex pairs with a separation larger than 100 nm. [1] I. Grekhov, et al., Physica C, 286, 18-24 (1997).

II3.44
PHASE COMPETITION BETWEEN Y_2BaCuO_5 AND Y_2O_3 PRECIPITATES IN Y-RICH YBCO THIN FILMS. U. Scotti di Uccio, F. Miletto Granozio, Dip. Scienze Fisiche, Universita di Napoli Federico II, Napoli, ITALY; F. Tafuri, Dipartimento di Ingegneria, Seconda Universita di Napoli, Aversa (CE); O.I. Lebedev, K. Verbist, G. Van Tendeloo, EMAT, University of Antwerp (RUCA), Antwerp, BELGIUM.

Segregation and competition of Y-based impurities in Y-rich biepitaxial YBCO samples is investigated. The analysis of distribution and microstructure of the precipitates is performed by High Resolution Electron Microscopy. Y_2O_3 precipitates are found in (103) YBCO films grown on (110) $SrTiO_3$ substrates. On the same biepitaxial samples, Y_2BaCuO_5 precipitates are found in (001) YBCO films deposited on an intermediate (110) MgO seed layer. Agglomeration of Y_2BaCuO_5 precipitates is observed near the edge in the MgO seed layer. The experimental data are discussed in the context of phase competition, nucleation, epitaxy and surface migration in the Y-Ba-Cu-O system. It is deduced that the change of supersaturation and surface energy, also governed by the favorable epitaxy of the secondary phase with the substrate or the YBCO matrix, plays a major role in determining the segregation of eitherY_2BaCuO_5 or Y_2O_3 precipitates.

II3.45
SYSTEMATIC VARIATIONS OF ELECTRONIC STRUCTURE IN Bi2212 SINGLE CRYSTALS WITH Pb- AND CARRIER DOPING LEVELS. J. Shimoyama, T. Motohashi, Y. Nakayama, T. Yamada, K. Otzschi, K. Kitazawa and K. Kishio, Department of Superconductivity, University of Tokyo, Tokyo, JAPAN.

We have reported that the flux pinning properties of Bi(Pb)2212 single crystals were systematically improved by increasing Pb-doping level up to its solubility limit, ~25% of Bi site [1,2]. The introduced inhomogeneity corresponding to the distribution of Pb ions is believed to contribute to the enhancement of pinning force in the present

system. On the other hand, intrinsic effect by Pb-doping on the electronic structure, which is indispensable for quantitative discussions on their flux pinning properties, has not been systematically understood yet. In the present study, we have performed various electrical transport measurements, such as resistivity along each crystallographic axis, Hall coefficient and E-J charactenstics, for oxygen controlled Bi(Pb)2212 single crystals. Through the careful resistivity measurements, we have found that the resistivity anisotropy (out-of-plane/in-plane) systematically decreases with an increase of Pb-doping level. In addition, the resistivity anisotropy always decreases with an increase of oxygen content in each Pb composition. Analyses made on the relationship between irreversibility lines and resistivity anisotropy revealed that the irreversibility lines of Bi(Pb)2212 with various Pb- and carrier doping levels can be expressed by a unique function containing only resistivity anisotropy and T_c as material parameters. Hall coefficient of the crystals annealed under the same condition systematically decreased with increasing Pb-doping level, suggesting the substantial carrier doping effect of Pb-doping. By comparing the Hall coefficient and resistivity anisotropy of oxygen overdoped pure Bi2212 and nearly carrier optimally doped Bi(Pb)2212 [Pb/(Bi+Pb)∼22%], we found that the latter crystal has much lower resistivity anisotopy, while it has smaller carrier concentration. This strongly indicates that the Pb-doping essentially decreases electrical anisotropy in the present system.
[1] I. Chong et al., Science 276 (1997) 770.
[2] J. Shimoyama et al., Physica C281 (1997) 69.

SESSION II4/L2/O2: JOINT SESSION:
BIAXIALLY TEXTURED SUBSTRATES FOR
HIGH-Tc COATED CONDUCTORS
Chairs: Ron Feenstra and James M.E. Harper
Tuesday Morning, November 30, 1999
Room 200 (H)

8:30 AM *II4.1/L2.1/O2.1
HIGH-J_c, YBCO CONDUCTORS FABRICATED BY EPITAXIAL DEPOSITION OF YBCO ON ROLLING ASSISTED BIAXIALLY TEXTURED SUBSTRATES (RABiTS). A. Goyal, R. Feenstra, M. Paranthaman, F.A. List, D.F. Lee, D.P. Norton, P.M. Martin, D. Verebelyi, X. Cui, E.D. Specht, D.B. Beach, T. Chirayil, C. Park, D.M. Kroeger and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

Progress made in the fabrication of Rolling assisted biaxially textured substrates (RABiTS) and epitaxial deposition or formation of HTS on such substrates is reported. Significant progress has been made in the fabrication of non-magnetic, strengthened, biaxially textured metal templates, deposition of oxide and other buffer layers and in the fabrication of long length substrates and superconductors. Ni-Cr alloy substrates fabricated using thermomechanical processing show a single orientation cube texture (∼100%) with sharp in-plane and out-of-plane textures, essentially identical to that obtained for pure Ni. High J_c's exceeding 1 MA/cm^2 have been demonstrated on epitaxially grown YBCO films on RABiTS using Ni-Cr as the starting template. Tensile tests and magnetic hysteresis and susceptibility measurements show that the substrates have greatly reduced magnetic properties compared to Ni, and are significantly stronger. In the area of buffer layer development, significant progress has been made in the formation of single orientation oxide buffer layers on Ni using sol-gel processes. A variety of Re_2O_3 type materials have been fabricated in this manner. The buffer layers are dense and crack-free and electron backscatter diffraction patterns show that the films have a high crystalline quality. High J_c's exceeding 1 MA/cm^2 have been demonstrated on such substrates using intermediate vapor deposited buffer layers. Progress made in the area of long length deposition using both vapor deposition and sol-gel will be reported. Efforts are underway to fabricate longer length superconductor samples exceeding l0cm and results obtained will also be summarized.
*Research sponsored by U.S. Department of Energy under contract DE-AC05 96OR22464 to Lockheed Martin Energy Research Corporation.

9:00 AM *II4.2/L2.2/O2.2
INCLINED SUBSTRATE DEPOSITION BY EVAPORATION OF MAGNESIUM OXIDE FOR COATED CONDUCTORS. Markus Bauer, Ralf Metzger, Robert Semerad, Paul Berberich, Helmut Kinder, Technische Universität München, Physik Department, Garching, GERMANY.

Thin films of $YBa_2Cu_3O_7$ (YBCO) must be highly textured in order to have good superconducting properties. One way to achieve this is to grow textured buffer layers on arbitrary polycrystalline substrates by inclined substrate deposition (ISD). This was first proposed by Hasegawa et al. using pulsed laser ablation. We use evaporation techniques to make the ISD process scalable to large areas and high production rates. Buffer layers of MgO are deposited at very high rate either by e-beam or simply by thermal reactive evaporation on substrates of hastelloy or stainless steel inclined by typically 40°. This leads to columnar growth with biaxial texture, improving with thickness up to 2 μm. YBCO films grown on these buffers are highly textured with FWHMs around 8°. The CuO_2 planes of the YBCO are typically tilted with respect to the surface by 20° towards the direction of vapor incidence. Therefore the critical current density is anisotropic, with lower j_c along the vapor direction and twice as much across. This direction must be chosen along the tape for coated conductors. The highest j_c we have reached so far is 0.8 MA/cm^2, resistively measured. To understand the texturing mechanism we have carried out Monte Carlo simulations. These confirm the columnar growth mode. We find that the preferred orientation arises from two effects, namely biased hopping of the particles due to their initial momentum, and mutual shadowing of the colums selecting the fastest growing orientation. Issues of magnetic anisotropy, production rate, upscaling of tape length, and cost will be addressed.

9:30 AM *II4.3/L2.3/O2.3
ION BEAM INDUCED GROWTH STRUCTURE OF FLUORITE TYPE OXIDE FILMS FOR BIAXIALLY TEXTURED HTSC COATED CONDUCTORS. Yasuhiro Iijima, Mariko Kimura, Takashi Saitoh, Fujikura Ltd., Material Technology Lab., Tokyo, JAPAN.

The achievement of sharp biaxial alignment of Yttria Stabilized Zirconia (YSZ) films by off-normal ion-beam-assisted deposition (IBAD) produced a hopeful application as flexible HTSC coated conductors using metallic substrates. Quite high-J_c values were successfully achieved by removal of intergranular weaklinks in Y-123 films on the YSZ templates. Till now 2-3 m length Y-123 tapes were fabricated using random polycrystalline Ni-based alloy tapes coated with textured YSZ layers. Ar^+ ion bombardment had significant effects on the crystalline structure of the YSZ films; to align a $< 100 >$ axis with the substrate normal, and a $< 111 >$ axis with the bombarding beam axis. Those two effects were induced simultaneously at room temperature and resulted in peculiar biaxially textured structure without epitaxial relationship to substrates. This paper discuss the alignment mechanism with the structural properties of several fluorite or related type oxide films including YSZ, CeO_2, Y_2O_3, etc., based on several proposed models. Films were formed on polycrystalline Ni-based alloy by dual ion beam sputtering method. Growth structures were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), atomic force microscopy (AFM) etc. Peculiar structural evolution of the crystalline orientation was observed and its development was well described by an exponential equation which agreed with Bradley/s selective growth model. It could be explained as a collaboration among in-plane and out-of-plane anisotropic growth of surface crystallites, and also homoepitaxial growth onto crystalline surface beneath, both induced by Ar^+ ion bombardment. Very smooth surfaces were observed by AFM imaging with a roughness of 2-3 nm and a peculiar ripple structure. The surface topographic structure was discussed by relating to Ressler/s surface binding energy model without using ion channeling. Because the energy of assisting ions were too low as 200 - 300 eV, the origin of azimuthal aligning effect is still under controversy.

10:30 AM *II4.4/L2.4/O2.4
BIAXIALLY TEXTURED BUFFER LAYERS ON LARGE-AREA POLYCRYSTALLINE SUBSTRATES. H.C. Freyhardt[1,2], J. Dzick[1,3], S. Sievers[1,3], J. Hoffmann[1], K. Thiele[1], F. Garcia-Moreno[1,2], A. Usoskin[2] and Ch. Joss[1]; [1]Institut fuer Materialphysik, Universitaet Goettingen, Goettingen, GERMANY, [2]ZFW: Zentrum fuer Funktionswerkstoffe GmbH, Goettingen, GERMANY, [3]Kabelmetal Electro GmbH, Hannover, GERMANY.

Biaxially textured buffer layers on polycrystalline metallic or ceramic substrates are required as templates for high-current-carrying HTS films, particularly coated YBCO films. In this contribution we report on our present understanding of the mechanisms governing the ion-beam-assisted deposition (IBAD) process employed for the preparation of textured YSZ as well as CeO_2 and Gd-doped CeO_2. On Ni, Hastelloy, SS as well as on ceramic tapes IBAD buffers could be produced with high-quality in-plane textures characterized by a FWHM of considerably smaller than 20°. Two systems were used, one with two 11 cm sputter sources and a 21 cm Kaufman ion source for the assisting beam and a second one with 11 cm sources for sputtering and the assisting beam. Furthermore, the IBAD process is now developed to an extend to allow the coating of large-area substrates (up to 20 cm x 50 cm) with well textured buffer films. Maximum current densities of PLD-YBCO layers on IBAD-buffered substrates reach values up to 2 MA/cm^2.
In parts supported by the German BMBF, kabelmetal electro GmbH and Siemens AG under grants 13 N 6924/6 and 13 N 6482, respectively.

11:00 AM *II4.5/L2.5/O2.5
METHODS EMPLOYED FOR PRODUCING HIGH-QUALITY ION-BEAM DEPOSITED CUBIC OXIDE TEMPLATE FILMS ON METAL SUBSTRATES. P.N. Arendt, J.R. Groves, S.R. Foltyn, Q.X. Jia, H.H. Kung, T.G. Holesinger, E.J. Peterson, P.C. Yashir, M.R. Fitzsimmons, R.F. DePaula, J.Y. Coulter, Y. Fan and M. Ma, Los Alamos National Laboratory, Los Alamos, NM.

Ion-beam assisted deposition was used to fabricate biaxially aligned cubic zirconia or magnesia films on flexible metal substrates. These films are used as templates for heteroepitaxially deposited YBCO films. The quality of the crystalline texture of the template films has a direct influence on the superconducting properties of the final YBCO films. We describe our efforts to fabricate high-quality templates on small-area substrates processed in stationary mode and meter-long substrates processed in continuous mode. Cubic zirconia templates were deposited on the meter-long substrates and magnesia templates were deposited on the small-area substrates. Our best phi scan FWHM values for the films on the meter-long tapes are 12.6 degrees for the template and 6.1 degrees for the overcoated YBCO. This meter-long tape had self-field, 75 K, superconducting critical current of 122 amps. Our best phi scan FWHM values for the films on small area substrates are 5.6 degrees for the template and 3.6 degrees for the overcoated YBCO. We will also describe x-ray grazing incidence measurements of the topmost layers of the template films as well as TEM measurements of the film microstructure.

11:30 AM II4.6/L2.6/O2.6
LEVEL SET SIMULATION OF ION BEAM ORIENTED MgO GROWTH. Xingquan Li, Dept. of Physics, University of Michigan, Ann Arbor, MI; Peter S. Smereka, Dept. of Mathematics, University of Michigan, Ann Arbor, MI; David J. Srolovitz, Princeton Materials Institute, Princeton University, Princeton, NJ; Giovanni Russo, Dept. of Mathematics, University of L'aquila, L'aquila, ITALY.

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

11:45 AM II4.7/L2.7/O2.7
QUANTITATIVE RHEED ANALYSIS OF BIAXIALLY-TEXTURED POLYCRYSTALLINE MgO FILMS ON AMORPHOUS SUBSTRATES GROWN BY ION BEAM-ASSISTED DEPOSITION. R.T. Brewer, J.W. Hartman and Harry A. Atwater, California Institute of Technology, Dept of Applied Physics, Pasadena, CA.

We have developed a computer simulation based on analytic calculation of RHEED patterns in the kinematic approximation for mosaic polycrystalline films for given values of electron beam incidence angle, polycrystalline texture, in-plane orientation distribution, and grain size. Although RHEED is most accurately modeled using a dynamical scattering model, the computational efficiency of kinematical scattering lends itself to development of a model suitable for real time control of biaxially-textured film growth by ion beam-assisted deposition (IBAD). Using the simulation results, we can quantitatively determine how RHEED spot shapes and relative intensities depend on the mosaic film characteristics. RHEED patterns taken at 15 keV with incidence angle in the range 1-5 degrees from 10 nm thick nominally [100]-textured MgO films grown on amorphous Si_3N_4 films by IBAD were analyzed by comparing experimental RHEED spot shapes and relative intensities with those predicted by the simulation results. For some films, an additional 200 nm thermally-grown MgO homoepitaxial layer was grown on top of the IBAD MgO layer. Results are also compared to X-ray rocking curve film analysis, and the quantitative correlation between biaxial texture and model-based RHEED analysis will be discussed.

SESSION II5: PHASE EQUILIBRIA, THERMODYNAMICS AND KINETICS
Chair: Winnie Wong-Ng
Tuesday Afternoon, November 30, 1999
Room 200 (H)

1:30 PM *II5.1
SUBSTITUTION FOR Ba BY Pr, La, AND Eu IN $Eu(Ba_{1-x}Re_x)_2Cu_3O_{7+d}$ SOLID SOLUTION. R.W. McCallum, K.W. Dennis, A. O'Connor and M.J. Kramer, Ames Laboratory, Iowa State University, Ames, IA; Youwen Xu, Mankato State University, Mankato, MN; S.K. Malik, Tata Institute of Fundamental Research, Bombay, INDIA; W.B. Yelon, University of Missouri Research Reactor, Columbia, MO.

Except for the $Pr_{1+x}Ba_{2-x}Cu_3O_7$ (Pr123ss), all other light $RE_{1+x}Ba_{2-x}Cu_3O_7$ (RE123ss) exhibit superconductivity with varying degrees of RE substitution on the Ba site. Previous study of Nd123ss system has shown that Pr is a well-behaved trivalent ion when nominally substituted for Ba. Neutron diffraction results demonstrated that Pr substitution in Nd123ss structure occurs partially on the Nd site and partially onto the Ba site. To try to differentiate the effect of charge from structural distortions arising from RE substitutions on the Ba site, we choose a solid solution based on $Eu(Ba_{1-x}RE_x)_2Cu_3O_{7+d}$, (RE = Eu, La, or Pr) because Eu has an ionic size (0.950Å) considerably smaller than that of trivalent Pr (1.013Å) or La (1.061Å) unlike Nd whose ionic radii (0.995Å) is nearly identical to Pr, resulting in a tendency for RE site exchange. If ionic radii is the controlling factor for site preference, Pr and La should have a greater tendency to substitute for Ba rather than Eu. Neutron diffraction results show that the larger RE remains on the Ba site while Eu remains on the RE site. The contraction of the lattice with increasing x is systematic with ionic radii, the La showing the least sensitive and Eu the most sensitive. However, DC magnetization shows that the depression of T_c with x is less pronounced with La and more pronounced with Pr as compared to Eu. If the valence state of the RE on the Ba site alone is the sole factor in hole localization, there should be no effect of varying RE on Ba site in suppressing T_c. However, the size of the RE could come into play either due to non-random substitution or varying charge localization due to differences in RE-O bond lengths. Both of these hypotheses will be discussed in light of crystal chemistry and magnetic behavior.

2:00 PM *II5.2
PHASE EQUILIBRIA AND MELT PROCESSING OF Re-Ba-Cu-O CERAMICS. Gernot Krabbes, Wolfgang Bieger, Peter Schaetzle, Guenter Fuchs, IFW-Institute of Solid State and Materials Research Dresden, Dresden, GERMANY.

The paper will present the thermodynamic approach to modifications of the melt texturing process for large single grain RE-Ba-Cu-O bulk materials up to 2 inch diameter, trapped fields larger than 1 T (at 77 K) and 9 T (at 44 K) on the top face of a YBaCuO cylinder, levitation forces of 100 N at 77K (or 20 N/cm^2 related to the top face of the 25 mm SmCo permanent magnet) and the critical current densities at 77 K of $45kA/cm^2$ (YBaCuO) or $70kA/cm^2$ (NdBaCuO) in absence of an applied field and the appearing J_c peak near 2 T applied field. Univariant equilibria define the framework for the process model which takes into account the growth of 123 phases by primary crystallization whereas 211(or 422for Nd) is dissolved in a certain distance from the growing front. The entrapment of remaining 211 particles into the growing crystal can be considered by defining an effective distribution coefficient of 211 between the melt suspension and the solidified bulk.
Then considerable alterations of the process route can be realized by choosing appropriate values for the thermodynamic variables as component activities, concentrations or oxygen partial pressure.
A solid solution $RE_{1+y}Ba_{2-y}Cu_3O_x$ is typical for RE = La, Nd, Sm due to partial substitution of Ba by RE ions. The superconducting properties (T_c,J_c) deteriorate with increasing y. The expansion of the stability field was found to depend on the oxygen potential. The optimal conditions for melt processing of these materials have been achieved either by controlling the composition by monovariant phase equilibrium (realized by admixing appropriate secondary phases) or by controlling $P(O_2)$.
It will be shown that phase stabilities and properties can also be influenced by annealing after growth as well as by chemical doping.

2:30 PM *II5.3
PHASE FORMATION, THERMAL EQUILIBRIUM AND TEXTURING IN Ag/Bi(2223) AND Ag/R.E.(123) TAPES. Renè Flükiger, Dept. Phys. Cond. Matter, Univ. of Geneva, Geneve, SWITZERLAND.

In Ag/Bi,Pb(2223) tapes, the phase relationships are strongly influenced by the heating ramp, the reaction history and the cooling conditions. A recent high temperature diffraction study at the high flux neutron reactor in Grenoble confirmed the formation of the Bi,Pb(2223) phase by nucleation and growth. During the cooling process, the phase Bi,Pb(2223) was found to remain stable, i.e. the phase limits are almost temperature independent. At the same time, however, a newly formed, non aligned Bi(2212) phase was observed, as a result of the decomposition of the Bi(2201) and 14/24 phases. The consequences for the jc values are discussed. We have recently found that the Pb content inside Bi,Pb(2223) filaments shows a continuous decrease with reaction time, correlated to an enhancement of Tc from 107.8 to 109.2 K after 200h in air at 838°C. The question is discussed,

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

3:30 PM *II5.4
SOLIDIFICATION PROCESSING OF RE-SYSTEM HIGH-Tc SUPERCONDUCTING MATERIALS. Teruo Izumi, Yuichi Nakamura, *Takateru Umeda and Yuh Shiohara, Superconductivity Research Laboratory, ISTEC, Tokyo, JAPAN .*Department of Materials Science and Metullurgy, Tokyo, JAPAN.

Solidification processing and crystal growth on HTSC are reviewed. Continuous progress has been achieved and some of them have recently led to the applications. In this paper, the progress of the solidification processing, understandings of crystal growth mechanisms and developments to applications are discussed. The solidification processes of HTSC oxides are classified into two groups, which are the growth from semisolid and that from solution. The peritectic reaction with diffusion through the liquid for fabricating RE-123 crystals was discovered in the series of research of unidirectional solidification processing. This idea has been widely utilized for understandings of the RE-123 crystal growth mechanism. Recently, the pushing/trapping phenomena of RE211 particles at the crystal growth interface have been observed firstly in the bulk growth, which have resulted in causes for the non-steady state growth. The semisolid processes are suitable for not only investigating the mechanism but also obtaining higher Jc. Actually, the high Jc have been reported in the samples grown by several different semisolid processes. On the other hand, solution growth has been recognized to be much suitable for realizing high crystallinity. Large single crystals with higher crystallinity were realized by optimizing the growth conditions in the crystal pulling. The single crystal wafer is expected as the substrate for the electronic device applications. LPE method is another process to attain high crystallinity. Recently, the self-refinement in the crystallinity during the growth was observed. The preferential dissolution/growth of the grains in the initial stage was responsible to the refinement. The LPE process is also expected to obtain higher Jc values, which can be realized by introducing micro-defects as a flux pinning center at substrate/film interface. This idea has been started to apply for the developments of the coated tape conductors. This work was supported by New Energy and Industrial Technology Development Organization.

4:00 PM II5.5
DIFFUSION OF Cu IN Ag SHEATH MATERIAL OF BSCCO TAPES. Peter J. Majewski, Andre Aubele, Fritz Aldinger, Max-Planck-Institut fuer Metallforschung, Stuttgart, GERMANY.

The Ag sheath material of the tapes has been found to dissolve up to 0.3% Cu. Experiments with Ag-(Bi,Pb)2223 diffusion couples show that the diffusion coefficient of Cu is high enough to penetrate the Ag sheath. When Ag is alloyed with Mg the diffusion coefficient of Cu appears to be higher. As (Bi,Pb)2223 tapes consist of about 80% of Ag this aspect is of great importance for the stoichiometry of the ceramic material, because during processing the Cu content of the ceramic decreases due to diffusion of Cu into the Ag sheath material. Experiments with Ag-Bi2212 diffusion couples show comparable results.

4:15 PM II5.6
THE EFFECT OF UNDERCOOLING ON THE GROWTH RATE OF NdBCO GRAINS IN REDUCED O_2 PARTIAL PRESSURE. N. Hari Babu, IRC in Superconductivity, University of Cambridge, Madingley Road, Cambridge, UNITED KINGDOM; W. Lo, Dept of Mechanical Engineering and Texas Center for Superconductivity, University of Houston, Houston,TX; D.A. Cardwell, Y. Shi, IRC in Superconductivity, University of Cambridge, Cambridge, UNITED KINGDOM.

The growth rate of $NdBa_2Cu_3O_7$ - $0.15Nd_4Ba_2Cu_2O_{10}$ composite in a 1 % O_2 in N_2 atmosphere as a function of undercooling temperature has been investigated in detail using isothermal top seeded melt growth. The grain growth rate parallel to the {100} {001} planes, R_a and R_c, have been observed to increase with increasing undercooling. In addition, R_c has been found to exceed R_a at all undercooling temperatures. Single, large grain NdBCO superconductors have been fabricated on the basis of this and complementary studies performed at fixed undercooling temperature from which the homogeneous nucleation of satellite grains has been eliminated.

4:30 PM II5.7
PHASE EQUILIBRIA OF Ag WITH Pb-BSCCO 2223 UNDER VARIOUS OXYGEN PRESSURES. L.P. Cook and W. Wong-Ng, Ceramics Division, NIST, Gaithersburg, MD.

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

4:45 PM II5.8
PHASE RELATIONS IN THE $BiO_{1.5}$-SrO-CaO-CuO SYSTEM. Vance J. Styve, Marie-Laure Carvalho, Joel Geny, James K. Meen, Don Elthon, University of Houston, Department of Chemistry and Texas Center for Superconductivity, Houston, TX.

A systematic study of phase equilibria in the quaternary $BiO_{1.5}$-SrO-CaO-CuO is being conducted to determine chemographic relationships between various superconducting and non-superconducting phases. Previous research on superconductors within the system and its subsystems have concentrated on maximizing modal content while minimizing amounts of impurity phases. Complete knowledge of the phase equilibria within key regions can play an important role in determining the optimal conditions for synthesizing superconducting materials. In order to fully understand the phase relations of the quaternary, knowledge of bounding binary and ternary systems is vital. Examining the subsystems shows that most of the phase volume is occupied by primary phase volumes of alkaline earth oxides and alkaline earth cuprates, leaving very little phase volumes of bismuth-bearing phases. This agrees with our previous study which showed CaO (with minor amounts of dissolved SrO) to be the liquidus phase over the entire range of f(O2) for compositions of the homologous series $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ (n=1,2,3 includes three known superconducting phases). Only for small n and high oxygen fugacities are compositions in the primary phase volume of a bismuth-bearing phase. Projection of phase relations from binary and ternary subsystems into the quaternary show that the primary volumes of bismuth bearing phases is confined to a limited region from $BiO_{1.5}$ to ~60% (Ca,Sr)O and ~20% CuO. We will discuss the projection of the SrO-CaO-CuO and $BiO_{1.5}$-CaO-CuO ternaries into the quaternary and examine supersolidus phase relations, modal proportions of liquid and crystalline phases, and reaction relationships of liquids and solids at various compositions, oxygen fugacities and temperatures. Some recorded examples of melting experiments conducted in a heating stage will be demonstrated and relevant chemographic relationsips will be discussed.

SESSION II6: CRITICAL CURRENTS
Chair: Stephen J. Pennycook
Wednesday Morning, December 1, 1999
Room 200 (H)

8:30 AM *II6.1
CRITICAL CURRENTS IN EPITAXIAL YBCO FILMS: ISSUES RELATED TO COATED CONDUCTORS. D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

Approaches to the fabrication of second-generation, coated conductor HTS wire involve the epitaxial deposition of YBCO films. These goals are similar to those of previous efforts to develop high-quality thin films for electronic devices, although the physical characteristics and properties requirements of coated conductors are somewhat different, mostly due to the need to economically achieve large overall current densities in the presence of substantial magnetic fields. Systematic studies of epitaxial films, deposited under a variety of controlled conditions that simulate the several less-than-ideal constraints posed by tape substrates and scalable deposition processes, can provide useful insights for understanding and optimizing the properties of coated conductors. Here we describe how individual isolated effects of low-angle grain boundaries, vicinal substrate surfaces, anisotropic strains, and film growth rates, thickness, and processing approaches can affect the critical current densities. In some cases the resulting defect structures are beneficial, providing flux pinning sites that enhance the high-field transport properties. Results will be discussed in relation to their consistency with the observed properties of short-length coated conductors. Research sponsored by the USDOE under Contract No. DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

9:00 AM *II6.2
TRANSPORT PROPERTIES OF BICRYSTALS AND GRANULAR COATED CONDUCTORS. Jan E. Evetts, Dept of Materials Science and IRC in Superconductivity, University of Cambridge, UNITED KINGDOM.

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

9:30 AM II6.3
THE ORIGIN OF HIGH CRITICAL CURRENTS IN $YBa_2Cu_3O_{7-\delta}$ THIN FILMS. B. Dam, J.M. Huijbregtse, F.C. Klaassen, R.C.F. van der Geest, G. Doornbos, J.H. Rector and R. Griessen, Faculty of Sciences, Division of Physics and Astronomy, Vrije Universiteit, Amsterdam, NETHERLANDS.

The high critical current densities in $YBa_2Cu_30_{7-\delta}$ thin films, point to the importance of *strong* pinning along extended defects. So far, however, it was not at all clear which type of defect is operative. Recently we found a clear correlation between the number of linear defects as revealed by wet etching (not only the screw dislocations!) and the characteristic magnetic field B* [1]. All our films sofar can be characterised by a certain characteristic field B* up to which j_c remains constant. While B* is proportional to the linear defect density n_{dial} we find no correlation with j_c. We can reproducibly manipulate n_{dial} by changing the PLD growth conditions. It appears that in PLD-grown films there is a correlation between the island size and the defect density. The growth process also enforces a self organised short-range order of the linear defects. The radial defect distribution function approaches zero at small distances, which enhances the efficiency of these pinning sites. By repeated etching and Atomic Force Microscopy (using markers to identify a specific film area) we are also able to obtain a depth profile of the linear defect density. We find that in 150nm films more than 70% of the defects is formed during growth. It appears that the length distribution and the spatial distribution of linear defects is the same in all our films. By annealing the films after deposition we find that some linear defects are annihilated. As a result both the density and the distribution of linear defects is changed. $YBa_2Cu_3O_{7-\delta}$ films offer therefore an attractive possibility to investigate vortex matter in a superconductor with tailored disorder. For technological applications the challenge is to find ways to increase the dislocation density, preferably keeping the short-range order intact.
[1]. B. Dam et al.: to be published in Nature June 1999

10:15 AM *II6.4
ADJUSTING GRAIN BOUNDARY TRANSPORT PROPERTIES FOR APPLICATIONS OF HIGH-TC SUPERCONDUCTORS. H. Hilgenkamp, R.R. Schulz, C.W. Schneider, B. Goetz, A. Schmehl, H. Bielefeldt, J. Mannhart, Exp. Phys. VI, Electronic Correlations and Magnetism, University of Augsburg, Augsburg, GERMANY.

The electronic properties of grain boundaries play a central role in many applications of high-Tc superconductors. Over the years, basic electronic properties of the grain boundaries have been studied in great detail by numerous groups, but a comprehensive understanding of the mechanisms controlling their properties is still in development. Here, we point out the important influences of the dx2-y2 symmetry component of the order parameter and of space-charge layers associated with band bending at the interface [1]. These space-charge layers can be altered to optimize the properties of grain boundaries for a given application, e.g. in large current carrying conductors or in electronics. This is demonstrated by enhancing the critical current densities and reducing the normal state resistivities of grain boundaries to unprecedented values, by appropriately doping the superconductor [2]. [1]: H. Hilgenkamp and J. Mannhart, Appl. Phys. Lett. 73, 265 (1998). [2]: A. Schmehl, B. Goetz, R.R. Schulz, C.W. Schneider, H. Bielefeldt, H. Hilgenkamp and J. Mannhart, to be publ. in Eur. Phys. Lett. (1999).

10:45 AM II6.5
CRITICAL CURRENT AND LOW ANGLE GRAIN BOUNDARIES IN YBCO COATED CONDUCTORS. D.T. Verebelyi, R. Feenstra, A. Goyal and D.K. Christen, Oak Ridge National Laboratory, Solid State Divison, Oak Ridge, TN; C. Prouteau, School of Metallurgy & Materials, University of Birmingham, Edgbaston, Birmingham, UNITED KINGDOM; P.N. Arendt, Los Alamos National Laboratory, Superconductivity Technology Center, Los Alamos, NM.

Biaxial texturing gives coated conductors the advantage of having primarily low angle (θ<10°) grain boundaries (GB). There is consensus that high-angle, YBCO [001] tilt boundaries reduce J_c exponentially with increasing misorientation angle. We find at 77 K, $J_c(\theta) = J_c(0)\ \exp(-\theta/3.2)$ for bicrystal GB's greater than 7°. This is expected because the GB is disordered and therefore weakly-linked. Alternatively, low angle GB's have periodic dislocations separated by strongly linked material. This low angle regime would therefore be expected to have a weaker than exponential dependence on θ. We pursue this low angle region by analyzing the best YBCO films on IBAD and RABiTS, with J_c>1.5 MA/cm^2. Electron backscattering images allow mapping of the GB misorientations and a "threshold" value of θ is assigned where pervasive current transport occurs along the sample. We find the J_c values for the coated conductors appear to also closely follow the exponential dependence on θ found for high angle GB's. Research co-sponsored by the DOE Division of Materials Sciences, the DOE Office of Power Technologies, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

11:00 AM II6.6
THE FORMATION OF YBCO [001] TILT GRAIN BOUNDARY STRUCTURES AND THEIR EFFECT ON THE TRANSPORT PROPERTIES. J.P. Buban, Y. Pan, N.D. Browning, University of Illinois at Chicago, Dept of Physics, Chicago, IL.

Atomic resolution scanning transmission electron microscope (STEM) observations have shown that [001] tilt grain boundaries in perovskite structured systems show similar atomic arrangements. In view of the similarity between the bulk structures, i.e. they are perovskites, this may not be surprising, but it does suggest that the boundary structures may be controlled by the same parameters that control the formation of the bulk crystal. For perovskites, the bulk crystal structure can be formed from consideration of the close packing of the [111] planes. In the case of YBCO, the hexagonal arrangement of the [111] Ba-O and Y-O planes form packing layers that alternate with [111] Cu planes. By consideration of how these planes interact at an [001] tilt boundary and making allowances for strain and stoichiometry, the experimentally observed structures can be reproduced. The interaction of the close packed planes at the [001] tilt grain boundaries leads naturally to the formation of 2x1 reconstructions in the boundary core on either the Y-Ba or Cu-O columns in the [001] projection, and the under- coordination of copper to oxygen. Whether this under- coordination is interpreted as causing the formation of localized donor states or to a decrease in the hybridization between copper and oxygen, the net effect is a reduction in the number of charge carriers at the boundary. As the number of these reconstructed sites increases linearly with misorientation angle, the width of this carrier depletion zone also increases linearly with misorientation angle and creates a tunnel barrier for high-angle grain boundaries. Calculating the tunneling of the current across such a barrier using Ginzburg-Landau theory generates a good match with various experimental measurements of the critica

11:15 AM *II6.7
MICROSTRUCTURE IN YBCO COATED CONDUCTORS. S.E. Babcock, Chau-Yun Yang, Yuehong Wu, Dept of Materials Science and Engineering, Univ of Wisconsin, WI; A. Goyal, R. Feenstra, D.P. Norton, Oak Ridge National Laboratory; S.R. Foltyn and P.N. Arendt, Los Alamos National Laboratory.

A key to both understanding and optimizing YBCO coated conductors lies in understanding the microstructural detail of the oxide layers. The interrelationships among the choices of buffer layers, deposition methods, microstructure, and physical properties are all central issues. This talk will summarize and compare the microstructural features we have observed in a variety of coated conductor samples with a view toward better understanding the critical current density and the path and distribution of current in the superconductor. The samples studied include YBCO deposited by both the barium fluoride precursor method and pulsed laser deposition on both IBAD and RABiTS substrates aid YBCO deposited on YSZ, CeO_2 and Yb_2O_3 cap buffer layers. This work is funded by the DOE University Partnership Initiative through Oak Ridge National Laboratory and the NSF MRSEC at the University of Wisconsin-Madison.

11:45 AM II6.8
TRANSMISSION ELECTRON MICROSCOPY CHARACTER-

IZATION OF THE MICROSTRUCTURE AND GRAIN BOUNDARY STRUCTURE IN YBCO COATED CONDUCTORS. H. Kung, S.R. Foltyn, P.N. Arendt, T.G. Holesinger, R.M. Dickerson, Q.X. Jia and M.P. Maley, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

The benefit of in-plane grain alignments has been well documented in YBCO and other high Tc superconductor (HTS) bicrystals, in which high critical current density (Jc) has been observed across small angle grain boundaries. In-plane alignment can be obtained by providing a textured template, such as a buffer layer via the ion beam assisted deposition (IBAD) technique. Critical current densities exceeding 1 MA/cm2 at 75K, zero field, have been reported by the Los Alamos team in these textured YBCO films. These findings illustrate the potential in controlling in-plane grain alignment to approach large-scale HTS applications. In this study, we have investigated the microstructure, composition, and grain boundary structure in YBCO coated conductors by transmission electron microscopy. Specifically, the local chemistry, habit planes, misorientations, and dislocation structures of grain boundaries have been characterized. The effect of substrate roughness, choice of buffer layer, and processing parameters in affecting the grain size and grain boundary characteristics will be examined. In addition, the different types of grain boundary dislocations affecting the critical current transport will be discussed.

SESSION II7: COATED CONDUCTORS
Chair: Paul N. Arendt
Wednesday Afternoon, December 1, 1999
Room 200 (H)

1:30 PM *II7.1
RELATIONSHIP BETWEEN YBCO THICKNESS AND THE CRITICAL CURRENT OF IBAD YSZ-BASED COATED CONDUCTORS S.R. Foltyn, P.N. Arendt, Q.X. Jia, L.R. Kinder, T.G. Holesinger, H. Kung, M. Paffett, J.F. Smith, T.N. Taylor, Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM.

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

2:00 PM II7.2
EPITAXIAL GROWTH OF OXIDE FILMS ON TEXTURED Ni SUBSTRATES STUDIED BY X-RAY MICROBEAM DIFFRACTION. J.D. Budai, N. Tamura, D.P. Norton, C. Park, J.-S. Chung, G.E. Ice, B.C. Larson, J.Z. Tischler, A. Goyal, Oak Ridge National Laboratory, Oak Ridge, TN; W.P. Lowe, Howard University, Washington, DC.

One approach to fabricating high-current superconducting wires involves the epitaxial growth of oxide buffer layers (YSZ/CeO_2) and YBaCuO films on rolling-assisted biaxially textured Ni substrates (RABiTS). We have investigated the microstructure of these heteroepitaxial multilayers using synchrotron x-ray microbeam diffraction at the Advanced Photon Source. White and monochromatic x-ray beams were focussed to sub-micron size, enabling high-resolution, grain-by-grain diffraction studies of the orientation and strain in each epitaxial layer. The use of x-ray microbeams provides measurements from a large number of different vicinal grains grown under identical conditions on a single sample; i.e. it enables a combinatorial approach. At typical growth temperatures (600°-800°C), we find that successive layers are not strictly epitaxial; rather, each heteroepitaxial layer exhibits a crystallographic tilt with respect to the underlying vicinal layer. The tilt angle typically depends linearly on the local miscut angle with a ratio that is consistent with a model based on elastic deformation at step ledges. In contrast, growth at low temperatures (450°C), yields approximately aligned layers, suggesting that reduced kinetics suppress the influence of the step ledges. *Managed by Lockheed Martin Energy Research Corp. for the U.S. Department of Energy under contract DE-AC05-96OR22464. Measurements using the MHATT-CAT beamline at the Advanced Photon Source operated by Argonne National Laboratory.

2:15 PM II7.3
PULSED LASER DEPOSITION OF YBCO THICK FILMS FOR COATED CONDUCTOR APPLICATIONS. Gregory Kozlowski, Rand Biggers, Iman Maartense, Timothy Peterson, David Dempsey, John Jones, John Busbee, Richard Kleismit, Materials and Manufacturing Directorate, AFRL, OH; Rama Nekkanti, John McDaniel, Paul Barnes, Charles Oberly, Propulsion Directorate, AFRL, OH; Mike Tomsic, Marvis White, and Asok Sarkar, Plastronic, Inc., Tipp City, OH.

Coated-conductor technology is being developed world wide as a potential technique to process YBCO conductors for high current applications in high-magnetic-field electric power devices at liquid nitrogen temperatures. Our approach to this task is to deposit biaxially textured YBCO onto buffered metallic substrates (Ni or Ni alloy) which have different degrees of in-plane and out-of-plane alignments by using pulsed laser ablation. The main goal of our effort is to maximize the critical current density of the coated conductors. Our best result so far is $2 \times 10^6 A/cm^2$ (equivalent to a direct current value of 55A at 77K) passed through a $0.3\mu m$ thick YBCO film with a $CeO_2/YSZ/CeO_2$ arrangement of buffer layers on a Ni substrate. CeO_2 and YSZ have been deposited by using laser ablation with emphasis on optimal processing in terms of deposition rate and thickness of these buffers. Many issues are addressed to produce this important result. Among them, buffer-layer arrangement and oxygenation process in YBCO films are discussed. In addition, chemical and structural analyses (SEM, EDAX, X-rays, electron back-scatter Kikuchi patterns) are provided to obtain a correlation between the physical and chemical properties of YBCO films.

2:30 PM *II7.4
CONVERSION KINETICS OF OXYFLUORIDE-DERIVED YBCO FILMS. Michael J. Cima, Igor Seleznev and Mani Gopal, Massachusetts Institute of Technology, Dept. of Materials Science and Engineering, Cambridge, MA.

Epitaxial thin films of $YBa_2Cu_3O_{7-x}$ (YBCO) high temperature superconductor have been prepared by metal organic deposition (MOD). Single crystal lanthanum aluminate and buffered nickel single crystal were used as substrates for single MOD coatings. Kinetics of fluoride removal from oxyfluoride films during high temperature ($T>700°C$) heat treatment was studied at different $P(O_2)$, temperature, and moisture levels of the furnace atmosphere. Influence of buffer layer on fluoride removal rate during high temperature heat treatment was also investigated. Thin film YBCO coatings were derived from a trifluoroacetate precursor solution. Buffers for single crystal nickel samples had a two-layer architecture and were made by the e-beam deposition. The precursor films were converted into glassy solid by heating to temperatures $T<400°C$ under controlled conditions. Conversion to YBCO films is carried out at temperatures greater than 700°C while exposed to water vapor. These films were quenched after different times in varying amounts of water vapor. Quenched samples were then dissolved in nitric acid solution. The concentration of residual fluoride in these samples was determined by a fluoride selective electrode. It was shown that different levels of moisture strongly influence kinetics of conversion. Our observations also indicate that buffer layer had mostly no influence on rate of fluoride removal, during conversion into crystalline YBCO.

3:30 PM II7.5
REEL-TO-REEL DIP-COATING UNIT FOR FABRICATING LONG RABiTS FOR HTS COATED CONDUCTORS. M. Paranthaman, T.G. Chirayil, J.S. Morrell, D.B. Beach, F.A. List, A. Goyal, X. Cui, D.F. Lee, E.D. Specht, D.M. Kroeger, R. Feenstra, D.P. Norton, and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

Bulk solution techniques have emerged as viable, potentially low-cost non-vacuum methods to produce long length conductors. In this technique, typically the precursor solution is either spin-coated or dip-coated onto the substrate and heat-treated in a furnace to obtain dense crystalline film. By using the sol-gel alkoxide route, we have recently shown that buffer layers such as gadolinium oxide and ytterbium oxide can be grown epitaxially on $\{100\}<001>$ textured-Ni substrates. To demonstrate the feasibility of producing meter lengths of the sol-gel buffered-Ni substrates, we have constructed a reel-to-reel dip-coating unit. This unit is capable of producing buffered substrates up to 10 meters per hour. We will present our results related to the scale-up of the sol-gel process. We will also discuss in detail about the choice of our buffer layers and their microstructures. Attempts will be made to grow YBCO films on these dip-coated buffers.

3:45 PM II7.6
SOLUTION DEPOSITION OF YBCO AND BUFFER LAYERS FOR COATED CONDUCTORS. P.G. Clem, M.P. Siegal, J.A. Voigt, E.L. Venturini, E.R. Byhoff, Sandia National Laboratories, Albuquerque, NM.

Solution deposition of oriented coated conductors on RABiT or IBAD substrates is a potential method for rapid, low cost production of superconducting tapes. Deposition of YBCO(001)//SrTiO3(100)// substrate structures has been accomplished on substrates including LaAlO3 (100), RABiT Ni (200), and CeO2 (200), with Jc (80K) 1 MA/cm2, and YBCO thicknesses up to 1.8 um by solution deposition. Strategies for effective integration of buffer layers and thick YBCO films will be presented, including understanding of intermediate phase formation and multilayer processing. Structure property relationships, including buffer layer epitaxial quality effects on superconductor Jc and XRD properties will also be discussed. Sandia is a multiprogram laboratory operated by Sandia Corp., a Lockheed Martin Company, for the US Dept. Of Energy under contract DE-AC04-94A185000.

4:00 PM II7.7
GROWTH REGIMES AND NUCLEATION OF THICK HIGH-J_c YBCO FILMS ON FLEXIBLE METALLIC SUBSTRATES. V.F. Solovyov, L.J. Wu, H. Wiesmann, Y. Zhu, M. Suenaga, Brookhaven National Laboratory, Upton, NY; D.P. Norton, Oak Ridge National Laboratory, Oak Ridge, TN; K.R. Marken, Oxford Superconducting Technology, Carteret, NJ.

We present an experimental study of growth mechanism and nucleation of superconducting properties of 1-5 μm thick YBCO layers on flexible metallic substrates for coated conductor applications. We analyze factors influencing the growth kinetics and nucleation of thick post-annealed films on CeO_2 buffers. Nucleation of non-epitaxial YBCO grains due to chemical interaction of the YBCO and the buffer is considered as a major limitation on J_c of these films. Two processes are assumed to be of importance for stable growth of c-axis oriented thick films: diffusion of H_2O vapor down to the growth front and diffusion of HF to the film surface. A simple model of the film growth is proposed, which suggests possible ways of improving thick films critical current. Using a modified growth technique we obtained good properties for 1 and 3 μm thick YBCO layers on RABiTS tapes, T_c = 91 K, J_c (77 K, 1T HIIc) = 1.1×10^5 A/cm^2 for a 3 μm layer on a RABiTS tape and 0.8×10^5 A/cm^2 for a 5 μm deposit. The transport properties of thick film samples are analyzed along with TEM and X-ray data to establish a correlation between J_c and the structural features of the YBCO layer and the substrate-YBCO interface.

4:15 PM *II7.8
LARGE AREA YBCO FILMS FOR RESISTIVE FAULT CURRENT LIMITERS. B. Utz, R. Nies, W. Schmidt, B. Seebacher, H.-W. Neumueller, Siemens Corporate Technology, Erlangen and Munich, GERMANY.

A very promising application of HTS is the resistive type fault current limiter (FCL) designed to enhance power quality in power distribution networks. Based on the superconducting transition of YBCO thin films, this device is a challenge especially to material science and engineering of HTS materials. While high critical current densities of large area YBCO films are required for power levels above 1 MVA, the homogeneity of both j_C and thickness proves to be crucial as well. Polycrystalline YSZ substrates are coated at room temperature with a YSZ buffer biaxially textured by ion beam assisted deposition (IBAD) using ion beam sputtering. The subsequent YBCO layer is grown at 650°C by thermal coevaporation (TCE) of the metal constituents Y, Ba and Cu while periodically oxidizing the film in a high oxygen partial pressure pocket. Substrates up to 20x20 cm^2 in size can be coated simultaneously at YBCO growth rates of 10-50 nm/min. The films are characterized in terms of crystallinity and morphology by XRD and SEM. The superconducting properties are mapped using a scanning inductive j_C measurement setup. Key figures on HTS quality and FCL performance are presented.

4:45 PM II7.9
SUPERCONDUCTING THALLIUM OXIDE FILMS FROM ELECTRODEPOSITED PRECURSORS. R.N. Bhattacharya, H.L. Wu, Y.-T. Wang, R.D. Blaugher, National Renewable Energy Laboratory, Golden, CO. Y. Tu, D.Z. Wang, Z.F. Ren, Dept of Physics, Boston College, MA. D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

Electrodeposition offers a promising and economic approach for fabricating high-temperature superconducting (HTS) wire or tape for the Tl-oxide superconductors. The Tl-oxides, moreover, offer the potential for operation at 77K in practical magnetic fields of 3-5 T, which is supported by measurements on the irreversibility behavior of the Tl-1223 single layer compound with Pb, Bi, and Sr substitution. Along with Tl-1223, we are also investigating chromium doped Tl-oxide (1212) superconductors. Recent advances in the development of biaxial textured electrodeposited films on Ag coated single crystal substrates will be discussed. We are initially studying the processing conditions and proper buffer architecture using pulse laser deposition (PLD) technique to achieve biaxially textured films with high transport properties which will then be applied to the low cost electrodeposition technique. At present we are making two layers elctrodeposited films to increase the thickness and also the uniformity of the films. . The transport measurement of the electrodeposited Tl-Bi-Sr-Ba-Ca-Cu-O (1223) and Tl-Cr-Sr-Ca-Cu (1212) films showed critical current density of about 100,0000 A/cm^2 at 76K in zero field.

SESSION II8: HTS FILM GROWTH
Chair: Ron Feenstra
Thursday Morning, December 2, 1999
Room 200 (H)

8:30 AM *II8.1
GROWTH MECHANISM OF HG-HTS FILMS IN CATION-EXCHANGE PROCESSES. J.Z. Wu, Y.Y. Xie, T. Aytug, A. Gapud, R. Aga, S.L. Yan and L. Fang, Department of Physics & Astronomy, Univ. of Kansas-Lawrence, KS; D.K. Christen and J.R. Thompson, Oak Ridge National Lab; Oak Ridge, TN; M. Siegal, Sandia National Lab, Albuquerque, NM.

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

9:00 AM *II8.2
INTERVAL PULSED LASER DEPOSITION OF REBaCuO. Dave H.A. Blank, Guus J.H.M. Rijnders, Horst Rogalla, University of Twente, Dept of Applied Physics, Low Temperature Division, THE NETHERLANDS.

With the introduction of the possibility to use Reflective High Energy Electron Diffraction (RHEED) at standard Pulsed Laser Deposition (PLD) pressures, it became possible to study the growth of oxide materials under different oxygen and temperature conditions. In this contribution we applied this technique to study the growth of the high temperature superconductor, using modified etch treated single terminated SrTiO3 single crystal substrates. In addition to the RHEED oscillations another phenomenon is observed, typical for PLD. The pulsed way of deposition leads to discontinuities in the intensity of the diffracted pattern. This is caused by the mobility of the deposited material from a disordered distribution till an ordered one and leads to a characteristic exponential slope with characteristic relaxation time constants. These time constants give extra information about relaxation, crystallization, and nucleation of the deposited material during growth. From these results (intensity oscillations as well as relaxations), a new approach to deposit these complex oxide materials will be introduced. The basic idea of this so-called interval deposition is to deposit an equivalent of one unit cell of material in such a short time that no coalescence in larger islands can occur, followed by a relaxation time before the next unit cell layer is deposited. This interval deposition leads to an imposed layer by layer growth. The latest results on the infinite layer structure as well as REBaCuO-superconductors will be presented.

9:30 AM *II8.3
ADVANCED VAPOR PHASE MANUFACTURING PROCESSES FOR HIGH TEMPERATURE SUPERCONDUCTOR TAPES. Robert H. Hammond, Luke S.-J. Peng, Weizhi Wang, William Jo, Arjen Janssens, M.R. Beasley, Stanford University, Stanford, CA.

The program at Stanford University to develop the processes and the

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

10:30 AM II8.4
EFFECTS OF PULSED-LASER DEPOSITION REGIMES ON FLUX-PINNING PROPERTIES OF YBCO THIN FILMS.
C. Cantoni, D.T. Verebelyi, D.K. Christen and D.P. Norton, Oak Ridge National Laboratory, Solid State Division, Oak Ridge, TN.

In thin YBCO films pinning results from strong extended defects that form during the growth process. In the pulsed-laser deposition technique the pulse repetition rate used during film growth is reported to influence film morphology and crystallinity. We observed a significant difference in the pinning properties of YBCO films grown by PLD using different deposition rates. YBCO films of the same thickness were grown under the same deposition conditions except for the pulse repetition characteristics. Pulse rates were varied between 0.1 and 100 Hz, and modulated in bursts separated by a time delay. All deposited films have high T_c and a J_c(H = 0 T, T = 77 K) $\geq$ 3 MA/cm^2. The data show a systematic suppression of J_c in large applied field for films grown at low pulse rates compared to films grown using repetition rates $\geq$ 4 Hz. The irreversibility field is also suppressed by more than 2 T for the films grown at lower rates. These data suggest that the time constant for some atomic scale agglomeration, dissociation, and diffusion processes occurring during film growth is the order of seconds, and that films grown at lower rates develop with a lower density of flux-pinning defects. We will present systematics of the critical current, pinning energy, and irreversibility line for films grown in different deposition regimes. Research co-sponsored by the DOE Division of Materials Sciences, the DOE Office of Power Technologies, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

10:45 AM *II8.5
GROWTH OF SUPERCONDUCTOR-RELATED LAYERED RUTHENATES AND LAYERED TITANATES. D.G. Schlom, J.H. Haeni, M.A. Zurbuchen, Y. Jia, A.H. Carim, Dept of Materials Science and Engineering, Penn State Univ, University Park, PA; W. Tian and X.Q. Pan, Dept of Materials Science and Engineering, Univ of Michigan, Ann Arbor, MI; L-N. Zou and Y. Liu, Dept of Physics, Penn State Univ, University Park, PA; G.W. Brown and M.E. Hawley, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

We have grown epitaxial films of layered ruthenates (Sr2RuO4, Sr3Ru2O7, and Ba2RuO4) and layered titanates (Sr2TiO4, Sr3Ti2O7, Sr4Ti3O10, Sr5Ti4O13, and Sr6Ti5O16) and studied their structural and transport properties. These layered perovskite phases are all in the An+1BnO3n+1 Ruddlesden-Popper homologous series with A = Sr or Ba and B = Ru or Ti. High purity Sr2RuO4 single crystals and doped SrTiO3 are well-known superconductors, but epitaxial films of Sr2RuO4 as well as the effect of reducing the dimensionality of SrTiO3 from 3-D to 2-D (i.e., Sr2TiO4) have never been reported. The film synthesis conditions will be discussed in the context of the relevant phase diagrams and thermodynamic stability. The use of in situ growth monitors, specifically atomic absorption spectroscopy (AA) and RHEED intensity oscillations, to achieve these structures, all of which have never before been prepared as epitaxial films, will be described. By monitoring changes in the RHEED intensity oscillations as the strontium and titanium are sequentially deposited, the Sr:Ti ratio can be adjusted to within 1% of stoichiometry. Furthermore, the presence of a beat frequency in the intensity oscillation envelope allows the adjustment of the strontium and titanium fluxes so that a full monolayer of coverage is obtained with each shuttered dose of strontium or titanium. It is only with such accurate control that we have been able to prepare the n = 1 to 5 Srn+1TinO3n+1 phases. Resistivity versus temperature measurements show that several of these materials exhibit metallic conductivity. However, superconductivity has not been observed (yet) in any samples.

11:15 AM II8.6
TRANSPORT PROPERTIES OF DOPED SPIN LADDER GROWN BY MOLECULAR BEAM EPITAXY. M. Lagues, M. Dorget, J.B. Moussy, C. Partiot, X.Z. Xu and C. Deville Cavellin Surfaces et Supraconducteurs, Ecole Suprieure de Physique et de Chimie Industrielles - Universite Paris, Paris, FRANCE.

Spin Ladder compounds are grown by molecular beam epitaxy under atomic oxygen presssure, using real time control by the RHEED intensity. The growth of these $MCu_2O_{3+\delta}$ films is controlled on MgO and $SrTiO_3$ substrates for M = pure Ca, pure Sr or Ca_xSr_{1-x}. The detailed structure is studied by four-circle XRD and HRTEM. The structure of $SrCu_2O_{3+\delta}$ is extremely close to the structure of the $SrCu_2O_3$ bulk compounds grown under high pressure by other teams: in both films and bulk compounds the ladder planes exhibit the same structure, while the stacking is slightly different. To our knowledge there is no other report for the synthesis of $CaCu_2O_{3+\delta}$ spin ladders which seems to be difficult to stabilize as a bulk compound. In all these cases the films contain pure phases of two-leg ladders. The copper valence and the charge transfer were measured from X-ray Fluorescence near the L3 copper edge for these films and for $Sr_{14}Cu_{24}O_{41}$ and $Sr_2Ca_{12}Cu_{24}O_{41}$ compounds by purpose of comparison. According to the preparation process, the charge transfer of the films vary in the range 0.1 to 0.25, which are of the same order of magnitude as the values measured respectively for $Sr_{14}Cu_{24}O_{41}$ and for $Sr_2Ca_{12}Cu_{24}O_{41}$. The transport properties were of the $SrCu_2O_{3+\delta}$ compounds show localization in agreement with possible variable range hopping, while $CaCu_2O_{3+\delta}$ Spin Ladders are metallic for T>150K and show localization below 150K. The possibility will be discussed that the transport properties are determined by the charge transfer but also by the 3D structure and the disorder level.

11:30 AM II8.7
STRUCTURAL PROPERTIES OF $BaCuO_2/CaCuO_2$ INFINITE-LAYER SUPERLATTICES GROWN BY PLD. L. DeCaro, C. Giannini, M. Nacucchi, L. Tapfer, Pastis-CNRSM, Brindisi, ITALY; G. Balestrino, P.G. Medaglia, G. Petrocelli, INFM, Universita di Roma Tor Vergata, Roma, ITALY.

The stoichiometry and structural properties of $BaCuO_2$ and $CaCuO_2$ thin crystalline films and of $(BaCuO_2)/(CaCuO_2)$ superconducting superlattices (SL), grown by the pulsed laser deposition technique on (100)STO substrates, are investigated by x-ray diffractometry and electron microprobe (EPMA). Simulations of the x-ray diffraction (XRD) and x-ray specular reflectivity (XSR) data, considering the microanalytical data, lead to an accurate structure refinement of the investigated films. By varying the structure factor and, consequently, the chemical composition of the hypothesized structure a precise and quantitative description of the structural configuration of the investigated SL was obtained. We found that the $BaCuO_2$ compound can be deposited in the infinite layer phase, even if the real structure can present many percents of copper vacancies in the CuO_2 planes. In the $BaCuO_2$ layers of the investigated superconducting SLs the Cu/Ba ratio less than one. Here, however, the copper vacancies are localized in the CuO_2 planes between barium atoms and are not present in other CuO_2 planes. Non-stoichiometric compounds with an excess of oxygen are compatible with the experimental data, indicating the $Ba_2Cu_{2-x}O_{4-y}$ layers as the reservoir charge blocks in the superconducting SLs.

11:45 AM II8.8
HOW MUCH CAN T_c BE INCREASED IN EPITAXIAL $La_{1-x}Sr_xCuO_4$ THIN FILMS? J.W. Seo[1,2], J. Perret[1,2], J. Fompeyrine[2], H. Siegwart[2], P. Martinoli[1] and J.-P. Locquet[2]; [1]Université de Neuchâtel, Institut de Physique, Neuchâtel, SWITZERLAND, [2]IBM Zurich Research Laboratory, Rüschlikon, SWITZERLAND.

The critical temperature T_c of a superconductor can be increased under hydrostatic pressure. This pressurized state is unstable and T_c returns to its ambient value upon pressure relief. Using epitaxial strain, the pressure state induced in a film can be stabilized. A recent study showed that by optimizing the strain, T_c of $La_{1.9}Sr_{0.1}CuO_4$ (214) thin films can be doubled to 49 K, compared to the bulk material. This increase of T_c is mainly determined by the sign and amount of strain (+0.5%), i.e. compressive strain, whereas tensile strain decreases T_c. However, what are the limits of this T_c change? The actual amount of strain is determined by the microstructure, e.g. surface morphology, defects and interface structure. To understand what limits the elastic deformation process, the influence of each component must be studied. We systematically investigate the structural and physical properties of 214 thin films grown on different substrates. In particular, we focus on the effect of the substrate material & its surface morphology and correlate the film

microstructure with the changes in resistivity and T. Preliminary results suggest that about twice as much compressive strain compared to above could be induced locally in 214 films.

SESSION II9: BSCCO PROCESSING AND PROPERTIES
Chair: Peter J. Majewski
Thursday Afternoon, December 2, 1999
Room 200 (H)

1:30 PM *II9.1
CURRENT 'ROADBLOCKS' FACING Bi-2223 WIRE DEVELOPMENT. G.N. Riley, Jr. American Superconductor Corporation, Westborough, MA.

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

2:00 PM II9.2
ON TEXTURE DEVELOPMENT IN HIGH-Tc SUPERCONDUCTORS. E. Cecchetti, P.J. Ferreira and J.B. Vander Sande, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA.

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

2:15 PM II9.3
THE PHASE FORMATION OF THE HIGH-Tc SUPERCONDUCTING Bi(Pb)-2223 COMPOUND STUDIED BY ELECTRON MICROSCOPY TECHNIQUES. Oliver Eibl, Department of Applied Physics, University of Tubingen, Tubingen, GERMANY; Bernhard Fischer, Vacuumschmelze GmbH, Hanau, GERMANY.

The (Bi,Pb)2Sr2Ca2Cu3O10 phase formation has been studied in Ag-sheathed multifilamentary tapes. The samples prepared for the series were pre-characterized by the critical current density jc, ac-susceptibility and x-ray diffraction. The formation of the Bi(Pb)-2223 phase occurs at initial stages of the heat treatment, in which (Bi,Pb)2Sr2CaCu2O8 , alcaline earth cuprates and Pb compounds act as the precursor phases. These initial stages of the heat treatment were studied intensively by EDX in the SEM and TEM, electron spectroscopic imaging (ESI) and high-resolution TEM. The obtained data show that the growth of the Bi(Pb)-2223 compound occurs in a two-dimensional way by transforming one or several atomic layers of composition Bi(Pb)-2212 into the composition Bi(Pb)-2223. Ca and Cu correlation diagrams obtained by quantitative EDX microanalysis are particularly useful for understanding the intergrowth phenomena, which is the typical microstructure in intermediate stages of the heat treatment, however, which is mostly ignored in x-ray diffraction studies. We determine the volume fractions of the secondary phases by EDX elemental mapping and subsequent quantitative multi-phase analysis by the concentration histogram (CHI) method. Finally, these data are compared to the chemical composition of the Bi(Pb)-2212/2223 crystallites. In the microstructure of the totally processed tapes the Bi(Pb)-2223 crystallites contain 5-10% intergrown lamellae of Bi(Pb)-2212 and the volume fraction of all secondary phases is about 4%, the minimun detectable fraction of secondary phases by the applied method is much smaller, around 0.1 vol%. With a combination of these methods a quantitative understanding of the phase formation is achieved. A detailed model for the Bi(Pb)-2223 phase formation is presented which is based on the layer-by-layer growth and summarizes the microstructural findings. The critical current density of the tapes clearly correlates with the Cu mole fraction and the variances of the various mole fractions of the Bi(Pb)-2223 crystallites.

2:30 PM II9.4
BSCCO LIQUIDS CONTAIN Cu(I). PHASE RELATIONS AND CONSEQUENCES FOR GROWING PHASE-PURE BSCCO SUPERCONDUCTORS. James K. Meen, Karoline Mueller, Vance J. Styve, Univ of Houston, Dept of Chemistry and Texas Center for Superconductivity, Houston, TX.

Most Cu-bearing crystalline materials that coexist with liquids in the Bi_2O_3- SrO-CaO-CuO system under oxygen contains Cu with a valence of at least two. CuO is a stable crystalline phase up to 1117 C in oxygen. Nevertheless, BSCCO liquids (and liquids in cuprate subsystems) contain Cu(I) as well as Cu(II) at temperatures well below 1117°C. Evidence for the presence of Cu(I) includes direct analysis of quenched glasses for Cu(I) and Cu(II) by iodometry and microbeam techniques, indirect determination of the Cu valence state by analysis for cations and oxygen, and chemographic analysis of the CuO liquidus surface. The liquidus for CuO when projected from a multicomponent system to pure CuO will not, in this case, reproduce the melting temperature of CuO. The presence of Cu(I) requires that phase relations be considered in the quinary Bi_2O_3-SrO-CaO-CuO-Cu_2O system. Liquids that have a composition of Bi:Sr:Ca:(total Cu)= 2:2:1:2 all contain some univalent Cu at all $p(O_2)$ values at 1 atm. Variation in $p(O_2)$ causes a liquid that has a Bi-2212 composition to move along a line denoting variation in CuO: Cu_2O. If the composition line cuts the primary phase volume of Bi-2212 at some value of $p(O_2)$, the liquid will crystallize entirely to Bi-2212 at that value of $p(O_2)$ and a unique temperature. If, on the other hand, the line does not intersect the primary phase volume of Bi-2212 at an appropriate oxygen pressure, the liquid cannot crystallize entirely to Bi-2212. Similar considerations hold for Bi-2223 and other cuprates.

2:45 PM II9.5
PRESSURE DEPENDENCE OF IRREVERSIBILITY LINE IN BSCCO. Mark E. Reeves, Department of Physics, The George Washington University, Washington, DC; Marc Raphael, Department of Physics, Catholic University of America, Washington, DC; Earl Skelton, Chris Kendziora, Naval Research Laboratory, Washington, DC.

One of the important problems of high-temperature superconductivity is to understand and ultimately to control fluxoid motion. We present the results of a new technique for measuring the pressure dependence of the transition to superconductivity in a diamond anvil cell. By measuring the third harmonic of the *ac* susceptibility, we determine the onset of irreversible flux motion. This enables us to study the effects of pressure on flux motion. The application of pressure changes interplanar spacing, and hence the interplanar coupling, without significantly disturbing the intraplanar superconductivity. Thus we are able to separate the effects of coupling from other properties that might affect the flux motion. Our results directly show the relationship between lattice spacing, effective-mass anisotropy, and the irreversibility line in $Bi_2Sr_2CaCu_2O_{8+\delta}$. Our results also demonstrate that an application of 2.5 GPa causes a four-fold decrease in the effective-mass anisotropy.

3:30 PM *II9.6
SPATIAL VARIATIONS IN COMPOSITION AND STRUCTURE IN BI-2223 MULTIFILAMENTARY TAPES. Terry G. Holesinger, John F. Bingert, Los Alamos National Laboratory, Los Alamos, NM; Qi Li, Ronald D. Parrella, Martin W. Rupich and Gilbert N. Riley, Jr., American Superconductor Corp., Westborough, MA.

Structural and compositional defects that may act as potential current limiting mechanisms (CLMs) were examined in Bi-2223 tapes from American Superconductor by transmission electron microscopy (TEM). Of particular interest in this work are the length scales at which the CLM's would operate, correlations with transport

properties, and the time scale for the development of CLM's during processing. Potential CLM's were divided into three categories based on whether they operated within a colony structure (microscale), between adjacent colony structures (mesoscale), or between different regions of the filamentary structure (macroscale). Particular attention was placed on two areas where complimentary work with lead composition studies and magneto-optical imaging (MOI) of crack structures had shown relationships to the transport properties. Compositional studies in the TEM show heterogeneity in composition throughout the tape and, in particular, lead depletion in Bi-2223 around the lead-rich 3221 phase. Examination of through-process tapes was used to identify the process steps during which CLM's developed within the tapes. The formation of crack structures and their partial healing in the final heat treatments were examined. Secondary phases present in the tapes during intermediate deformation are prime sources for formation of large, extended crack structures. Smaller, localized crack structures were also found which result from the failure of the matrix during mechanical deformation. Roughening of the silver sheath/BSCCO interface during intermediate deformation was found to increase the number of high-angle grain boundaries in the colony structure next to the silver sheath.

4:00 PM II9.7
OBSERVATION OF LOCAL PHASE CHANGES AT GRAIN BOUNDARIES IN AG-SHEATHED BSCCO TAPES.
Kyosuke Kishida, Nigel D. Browning, Dept of Physics, Univ of Illinois at Chicago, Chicago, IL.

Grain boundaries contained within the superconducting filaments of highly textured $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10}$(Bi-2223)/Ag composite tapes are known to strongly influence the overall transport of large currents. Although several models have been proposed to describe the macroscopic current pathways in these wires, the underlying mechanism controlling the properties of individual grain boundaries has yet to be clarified. In order to elucidate the exact role of grain boundaries in these tapes, systematical studies of the atomic structure and chemistry changes that occur at the boundaries are required. For this purpose, the microstructure of Bi-2223/Ag multifilament tapes have been investigated by scanning transmission electron microscopy (STEM). In particular, use of the high-angle annular dark field or Z-contrast technique has allowed direct atomic resolution images of grain boundary structures to be obtained.
The majority of the grain boundaries observed in these tapes are (001) pure twist boundaries. These boundaries are seen to be located in the middle of the double BiO layers and are atomically flat. However, it is important to note that although no amorphous layers have been observed at these boundaries, local phase variations near the grain boundaries are often observed, i.e. 1/2 - 2 unit cells of Bi-2212 or Bi-2234 phase are formed on either both or one side of the boundaries. This trend of forming the boundary plane on the Bi-O double layers, accompanied by periodical phase variations is also observed at low-angle asymmetric c-axis tilt boundaries, where one side of the boundary is parallel to the (001) plane. Such local phase variations and the distortion of the Bi-O double layers at the boundaries may have a significant impact on the local transport properties. Electron energy loss spectroscopy (EELS) analysis of these boundaries will also be discussed in relation to the effect of the observed structure variations on the local carrier concentration.

4:15 PM II9.8
LOCAL VARIABILITY OF Jc IN HIGH Jc MULTIFILAMENTARY Ag/Bi2223 TAPES. X.Y. Cai, J. Jiang, A. Polyyanskii, Y.H. Wu, S.E. Babcock and D.C. Larbalestier, Applied Superconductivity Center, University of Wisconsin, Madison WI; R.D. Parrella, Q. Li, M.W. Rupich, G.N. Riley, Jr., American Superconducter Corporation, Westborough, MA.

We have extracted filaments from multifilamentary Ag/Bi2223 tapes with very high Jc(0T,77K) values of 50 - 60 kA/cm^2. Using a lasser cutter to section the filament bundle, we can establish the positions of filaments within the cross section of the tape and,where useful,isolate particular filaments and sections of filaments for local measurements of Jc(H), defined by Ic/A,where A is the cross sectional area measured with an error $< 5\%$. Local Jc(0T,77K) values are rather variable, values ranging from 20 - 100 kA/cm^2, different by a factor of 5. Both self-field and in-field Jc(H) show an inverse linear dependence on the thickness of filament section, suggesting that thin layers near the interface of Ag/Bi2223 are still the major conductive regions even though the filaments are already $<$10 micrometer thick. Local variability of microstructure is large, suggesting that there is still a large opportunity for further improvement of Jc in today's state of the art high Jc tapes.

4:30 PM II9.9
DIRECT OBSERVATION OF THE GROWTH OF RIBBON-LIKE THIN FILMS OF Bi-2212. Shunichi Arisawa[1,2], Hanping Miao[2], Akira Ishii[1], Yoshihiko Takano[1], Yoshimasa Satoh[3], Hiroki Fujii[1,2], T.Hatano[1], Kazumasa Togano[1]; [1]Nat. Res. Inst. for Metals. Tsukuba, JAPAN, [2]CREST, Japan Science and Tech. Coop. Tokyo, JAPAN, [3]Univ. of Tsukuba, JAPAN.

We have demonstrated that ribbon-like single crystalline Bi-2212 films can be grown on Ag substrates by a very simple process[1]. In this paper, we will present the in-situ observation of the growth of the ribbon-like films by high temperature optical microscopy and the improvement of the substrates. Small pellets of the Bi-2212 were put on silver substrates. In a high temperature microscope, the samples were heated up to 905 C and were kept at the temperature for 10 minutes. They were then gradually cooled down to 850-860 C and kept for several hours followed by furnace cooling. The heat treatment was carried out under 1 atm of oxygen. Very thin ribbon-like films of Bi-2212 crystals grew on the Ag substrates by this simple method. The ribbon-like films showed the superconducting transition at the onset temperatures of 70-80K. By the $in-situ$ observation of the growth of the ribbon crystals, it was found that most of the ribbons grew within some 10 minutes when the starting materials were melted. During the slow cooling, a little retreat of the ribbons was seen and this retreat was strongly dependent on the heat treatment schedule. Further, the improvement of the substrate will be shown. In our previous work, the surfaces of the heat-treated substrate went very rough by the re-crystallization of the silver. We have succeeded in reducing the roughness by adding Pd in the Ag substrate. [1] Arisawa et al., Physica C 314 (1999)155

4:45 PM II9.10
EFFECT OF COOLING RATE ON THE MICROSTRUCTURE AND SUPERCONDUCTING PROPERTIES OF $Bi_{2-x}Pb_xSr_2CaCu_2O_y$/Ag COMPOSITE TAPES. H. Fujii, H. Kitaguchi, H. Kumakura, K.Togano, National Research Institute for Metals, Tsukuba, JAPAN; Y. Hishinuma, Advanced Materials Research and Development Center, Meisei University, Hino, JAPAN.

It is known that Ca_2PbO_4 is one of the main secondary phases acting as an obstacle for supercurrent path in $Bi_{2-x}Pb_xSr_2CaCu_2O_y$ ((Bi,Pb)-2212)/Ag tapes. We initially investigated the stability region of Ca_2PbO_4 in oxygen partial pressure (P_{O2})-temperature (T) diagram using bulk (Bi,Pb)-2212 samples. With increasing temperature and decreasing P_{O2}, Ca_2PbO_4 became less stable and Pb in Ca_2PbO_4 dissolved into Bi-2212 phase. Above 1100 K, Ca_2PbO_4 was not observed under P_{O2}=0.21 atm for x=0.2. These results indicate that rapid cooling from high temperature is required to avoid the segregation of Ca_2PbO_4. Taking these results into account, we investigated the effect of cooling rate on the microstructure and superconducting properties of (Bi,Pb)- 2212/Ag tapes of x=0.2 heat treated under P_{O2}=0.21 atm. For the tapes obtained by rapid cooling from 1103 K where Ca_2PbO_4 is not present, the amplitude dependence of the real part of ac susceptibility was smaller and the J_c values were higher than those of the sample obtained by a furnace-cooling. These results suggest that rapid cooling is effective to reduce the segregation of Ca_2PbO_4 at the grain boundaries and to improve the grain connectivity.

SESSION II10: POSTER SESSION:
FILM GROWTH AND COATED CONDUCTORS
Chair: David K. Christen
Thursday Evening, December 2, 1999
8:00 P.M.
Exhibition Hall D (H)

II10.1
THE INFLUENCE OF AN ELEVATED MAGNETIC FIELD ON THE TEXTURE FORMATION OF MELT-PROCESSED BI-2212. E. Cecchetti, P.J. Ferreira and J.B. Vander Sande, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA.

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

II10.2
DEFORMATION AND CRITICAL CURRENT DENSITY OF

Ag-Bi2223 TAPES. H. Wu, P. Skov-Hansen, W.G. Wang, Z. Han and P. Vase, Nordic Superconductor Technologies, DENMARK.

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

II10.3
EVOLUTION OF CURRENT-CONTROLLING FACTORS IN Ag/BiSCCO TAPES THROUGHOUT THE OPIT PROCESS. V.Beilin, A. Goldgirsh, E. Yashchin, M. Roth, I. Felner, The Hebrew University of Jerusalem, Jerusalem, ISRAEL; A. Polyanskii, D. Larbelestier, University of Wisconsin, Applied Superconductivity Center, Madison WI.

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

II10.4
CORRELATION BETWEEN SIZE/STRAIN PARAMETERS AND CRITICAL CURRENT DENSITIES IN BSCCO/Ag TAPES. Fernando Rizzo, Bojan Marinkovic, Alexander Polasek, Pontifícia Universidade Catölica do Rio de Janeiro, Departamento de Cincia dos Materiais e Metalurgia, Rio de Janeiro, BRAZIL; Xia Sike, Mauricio Lisboa, Eduardo Serra, Centro de Pesquisas de Energia Elétrica Eletrobras, Rio de Janeiro, BRAZIL.

Size/strain evolution and critical current densities, were studied in BSCCO/Ag tapes filled with two different precursor powders. The precursor powders were obtained by conventional so-called 'one-powder' technique and, also, by 'two-powder' processing route. The phase content of precursor powders is already well established. The size/strain investigations were performed after each step of thermomechanical treatment, using X-ray diffraction line-broadening approach. Convolution-fitting method was used for obtaining physical (intrinsic) line broadening. Size/strain parameters were analyzed by 'double-Voigt' method. Existence of low-angle boundaries inside grains were considered as a potential flux pinning sites. Potential presence of chemical inhomogenity in 2212-BSCCO and 2223-BSCCO were monitored by strain behavior over column length orthogonal to investigated diffracting planes.

II10.5
SYNTHESIS AND CHARACTERIZATION OF THALLIUM BASED 1212 FILM WITH HIGH TRANSPORT CRITICAL CURRENT DENSITY. Jingyu Lao, J.H. Wang, SUNY at Buffalo, Department of Chemistry, Buffalo, NY; D.Z. Wang, S.X. Yang, Y. Tu, H.L. Wu, Z.F. Ren, Boston College, Department of Physics, Chestnut Hill, MA; D.T. Verebelyi, M. Paranthaman, T. Aytug, D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN; R.N. Bhattacharya, R.D. Blaughger, National Energy Renewable Laboratory, Golden, CO.

A new type of Cr doped Tl-1212 film has been successfully synthesized on $LaAlO_3$ substrate. The Tl contained precursor film was deposited by PLD method, and annealed in static air with Tl contained pellet to become superconductive. The T_c of the film is in the range of 94 - 100 K, and the transport J_c of the film reaches 1.5 x 10^6 A/cm^2 at 77 K and self field. According to our knowledge, this is the highest transport J_c at 77 K for Tl-1212 film up to now. XRD θ-2θ scan, Ω scan and Φ scan shows the dominant, epitaxial growth of 1212 film on the substrate. TEM analysis verifies the high quality of the film.

II10.6
SURPRISING THERMAL STABILITY OF Hg-1212 FILMS. M.P. Siegal, D.L. Overmyer, E.L. Venturini Sandia National Labs, Albuquerque, NM; J.Z. Wu and Y.Y. Xie, Dept. of Physics & Astronomy, Univ. of KS, Lawrence, KS.

We studied the high-temperature thermal stability of high-quality Hg-1212 thin films by incrementally annealing samples in a pure oxygen ambient from 200 to 800C. The films for this study were grown via the cation-exchange process where a high-quality Tl-1212 or Tl-2212 film acts as a precursor. Typical as-grown Hg-1212 films have Tc > 120 K and Jc ~ 1 MA/cm2 at 100 K. By monitoring surface morphology, x-ray diffraction microstructure, and Jc (5K) from magnetization measurements, we find that Hg-1212 films are stable to temperatures > 750C, and in the absence of the necessary Hg-oxide overpressure for their growth! This is even more surprising given the severe degradation of the Tl-superconductor precursor films at 700C in the same oxygen ambient, due to Tl-oxide volatilization. The onset of this degradation in Tl-superconducting films generates defects associated with significant enhancements in flux pinning. Comparisons will be made with Hg-1212 films annealed to similar conditions. *Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. DOE under Contract DE-AC04-94AL85000.

II10.7
REVERSAL OF UNDER-DOPED TO OVER-DOPED Tl2Ba2CuO6 THIN FILMS. D.Z. Wang, S.X. Yang, Y. Tu, Z.F. Ren, J.I. Oh, Y.Sun, H.I. Ha and M.J. Naughton, Boston College, Dept of Physics, Chestnut Hill, MA; Jingyu Lao, J.H. Wang, SUNY at Buffalo, Dept of Chemistry, Buffalo, NY.

Epitaxial superconducting thin films Tl2Ba2CuO6(Tl-2201) with different doping levels have been synthesized. The films were made by rf magnetron sputtering and post-deposition annealing. The doping levels were controlled by varying the oxygen content. At first, the as-grown Tl-2201 thin films were annealed gradually from the initially over-doped state, through the optimally doped state, to the under-doped state in flowing argon or a vacuum. Then, the same samples were brought back, through the optimally doped state, to the over-doped state in air or oxygen. Upon addition and depletion of oxygen there was no structural change measured by XRD. Transmission electron microscopy bright-field images and selected area diffraction patterns showed that the tetragonal structure didn't change either.

II10.8
FABRICATION OF $YBa_2Cu_3O_x$ THICK FILM ON NON-TEXTURED METALLIC SUBSTRATE BY ELECTRO- PHORETIC DEPOSITION. R.L. Meng, J. Hildebrand, J. Cmaldalka, D. Pham and C.W. Chu, Dept of Physics and Texas Center for Superconductivity, Univ of Houston, Houston, TX.

A practical electrophoretic process has been applied to deposit $YBa_2Cu_30_x$ thick films on a non-textured Ag alloy substrate. X-ray diffraction patterns show a high degree of c-axis texturing perpendicular to the surface. There is no observable reaction between the substrate and the YBCO thick films. A sharp transition with an onset temperature of 90 K and a zero-resistance temperature of 87 K indicates the high quality of the thick films. A transport J_c of up to 7-9 x 10^4 A/cm^2 has been achieved repeatedly at 77 K and zero field. The microstructure and physical properties of the YBCO thick films strongly depend on the process conditions, which will be discussed.

II10.9
MICROSTRUCTURE DEVELOPMENT AND INTERFACE STUDY IN PERITECTICALY GROWN $YBa_2Cu_3O_x$ TAPES ON SILVER ALLOY SUBSTRATE. Donglu Shi, X. Wen and D. Qu, Department of Materials Science and Engineering, University of Cincinnati, Cincinnati, OH.

A series quenching experiments have been carried out to study the initial nucleation and growth of YBCO on silver alloy substrate. Although the substrate has a random orientation, the YBCO grains have been found to be highly in-plane textured. The work is motivated to study the nucleation mechanism of YBCO from the liquid state during the peritectic solidification. Our results show that during peritectic reaction, YBCO grains precipitate along the surface of the silver alloy substrate which acts as the nucleation site. As the liquid wets the substrate, the precipitation of YBCO cannot assume a upward growth normal to the substrate surface due to liquid surface tension. As a consequence, they epitaxially grow at a high rate upon quenching along the substrate surface, which minimizes the system energy. Our x-ray data show a consistent evolution of YBCO precipitation as the quench temperature is lowered from slightly above peritectic point to 950°C. Corresponding microstructure study results will also be presented.

II10.10
MICROWAVE PROPERTIES OF A NOVEL SUBSTRATELESS RESONATOR USING SINGLE DOMAIN $YBa_2Cu_3O_x$. Donglu Shi and D. Qu, Department of Materials Science and Engineering, Altan Ferendeci, Dept. of Electrical and Computer Engineering, David Mast, Dept of Physics, University of Cincinnati, Cincinnati, OH.

Large single domain $YBa_2Cu_3O_x$ materials have been successfully fabricated with superb RF properties by employing the seeded-melt growth (SMG) method. The SMG YBCO samples have been treated with flowing oxygen in a wide range of temperatures up to 700°C. After oxygenation, the surface resistance has been measured at 12.95 GHz in a magnetic field up to 1.9 T. Our experimental data show sharp superconducting transitions and magnetic field dependence below T_c. Based on a new design, a cavity resonator has been constructed using the single domain $YBa_2Cu_3O_x$ processed by SMG. The cavity consists of a hollow cylindrical cup covered by a polished plate, which operates in the TM_{010} mode. All cavity parts are made of single-domain $YBa_2Cu_3O_x$ without any dielectric materials. The measured Q has reached a high value of 10,200 at 18.4 GHz. Our experimental data show great promise in the development of RF components using single domain high temperature superconductors.

II10.11
MICROWAVE PROPERTIES OF SCREEN-PRINTED Bi2223 THICK FILMS ON DIELECTRIC CERAMIC $Ba(Sn,MgTa)O_3$. A. Oota and D. Washimoto, Toyohashi University of Technology, Tempaku-cho, Toyohashi, JAPAN; Y. Kintaka, T. Tatekawa, N. Matsui, H. Tamura and Y. Ishikawa, Murata MFG Co. Ltd., Nagaokakyo, Kyoto, JAPAN.

Screen-printed Bi2223 thick films are prepared directly on both sides of dielectric disk $Ba(Sn,MgTa)O_3$ (abbreviated as BSMT) with relative dielectric constant ε,=24. Microwave properties of the films such as unloaded quality factor Q_u and surface resistance R_s are measured as a function of temperature in the range between 20 and 130 K on a TM_{010} mode, while checking the incident-power dependence. In an attempt to suppress chemical reactions, the nominal composition of superconducting paste is changed and the influence on microwave properties (in particular on the incident-power dependence) is discussed through multi-phases studies from a micro-structural point of view. The present best data for the Q_u value at 10dBm are 30,000 at 70K and 90,000 at 20K, which correspond to the R_s value of 0.8 and 0.3 mΩ, respectively. Dual-mode resonator operating at 1.8 GHz on a TM_{110} mode is under construction using 25 mm-cubes of BSMT with Bi2223 thick films as electrodes and the result will be presented.

II10.12
MICROWAVE SURFACE RESISTANCE OF SCREEN-PRINTED Bi2212 THICK FILMS. M. Tanaka, A. Oota, Toyohashi University of Technology, Totohashi, Aichi, JAPAN.

Microwave surface resistance (R_s) of screen-printed $Bi_2Sr_2CaCu_2O_x$ (Bi2212) thick films on Ag substrate subjected to the partial-melt solidification process was investigated using the dielectric resonator method at 10.7GHz in the TE_{011} mode. Film thickness and heat treatment conditions on the partial-melt solidification process influence the R_s value of thick films through changes in micro-structures therein. Low R_s value of 1.6m at 20K was obtained by heating the 27 m-thick film up to the maximum temperature at 880°C, subsequent slow cooling at 4°C/h from 880 to 860°C, isothermal annealing for 10 hours at 860° and final cooling to room temperature. To our knowledge, this R_s value is the lowest record for Bi2212 thick films subjected to the partial-melt solidificaton process. From a microstructural point of view, the dominating factor for the R_s value of Bi2212 thick films was investigated. The film with low R_s value shows the large grain size, small amount of impurity phases and also a high degree in grain alignment.

II10.13
THE EFFECT OF NANO-PARTICLES ADDITION ON FLUX PINNING IN HIGH TEMPERATURE OXIDE SUPER-CONDUCTORS. Gui-Wen Qiao, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, CHINA.

To increase the magnetic flux pinning ability of high Tc oxide superconductors, several kinds of nano-particles were added into the oxide superconductors and the Tc and Jc were measured. The experiments showed that the addition of proper amounts of nano-Cu, nano-MgO, nano-SiC and nano-ZrO_2 ultrafine powders can increase the critical current density Jc of the HTSCs. SEM and HREM observations showed that the nano-MgO and nano-SiC particles were solved into the HTSC matrix and caused local segregation of chemical elements and lattice distortions which may play a role of flux pinning centers and contribute to increasing of Jc. But nano-Al_2O_3 particles can not be solved into the matrix and keep their original sizes and shapes which may not contribute to the flux pinning.

II10.14
CONTROL OF STACKING FAULT CONFIGURATIONS IN MELT TEXTURED YBCO: A PATH TOWARDS HIGH CRITICAL CURRENTS. J. Plain, Institut de Ciencia de Materials de Barcelona (CSIC), Barcelona, SPAIN, and Universite de Poitiers, Lab de Metallurgie Physique, Poitiers, FRANCE; F. Sandiumenge, T. Puig, Institut de Ciencia de Materials de Barcelona (CSIC), Barcelona, SPAIN; J. Rabier, Universite de Poitiers, Lab de Metallurgie Physique, Poitiers, FRANCE; X. Obradors, Institut de Ciencia de Materials de Barcelona (CSIC), Barcelona, SPAIN.

YBCO (1:2:3) is metastable under a wide area of P(oxygen)-T space covering typical processing conditions. Extrinsic Cu-O stacking faults (SFs) are nucleated due to a local phase transformation to the 1:2:4 phase. Motivated by the high potential of dislocations as pinning centers, we explore different routes to develop SF configurations exhibiting a high perimeter to surface ratio in order to maximize the length of partial dislocation. Two forces are required for the nucleation and growth of such SFs. (1) A thermodynamic one, F1, which measures the distance from the stability line of 1:2:3 at which the sample is processed, and (2) a mechanical one, F2, necessary to move the bounding partials on (001). In the present experiments F2 results from an isostatic pressure due to the anisotropy of the composite. Under 200 MPa in Ar at 300°C it is found that F2 is dominant, the total SF area remains almost constant, but owing to the different SF energies on both twin domains, a selective re-organization of pre-existing SFs to a low energy configuration in one twin domain occurs resulting in an increase of the total partial dislocation length. A 100% increase of the critical current is achieved by this procedure. On the other hand, at 400°C and 100 bar oxygen F1 dominates over F2. Accordingly the SF area is increased through the nucleation of new SFs at 2:1:1 interfaces, which owing to the strong non-equilibrium conditions, expand across twin walls and display irregular dendritic-like shapes. In this case critical currents are augmented by 150%. Pinning mechanisms are discussed on the basis of transmission electron microscopy observations and inductive magnetization measurements with the magnetic field applied parallel to the c-axis.

II10.15
NOISE CHARACTERISTICS OF ASYMMETRIC MULTI-JUNCTION HTS RF-SQUID MAGNETOMETER AND GRADIOMETER ON BICRYSTAL SUBSTRATES. M. Fardmanesh, K. Barthels, J. Schubert, Institut fuer Schicht- und Ionentechnik (ISI), Forschungszentrum Juelich, Juelich, GERMANY.

Asymmetric multi-junction YBCO rf-SQUID magnetometer and gradiometer have been made of 200 nm thick patterned (standard photolithography) PLD YBCO films on the symmetric bi-crystal $SrTiO_3$ substrates. Low 1/f noise performance has been obtained for the rf-SQUIDs (magnetometers and gradiometers) on the bi-crystal substrates by implementing a design principle avoiding large area films on the bi-crystal grain boundary of the substrates. Washer rf-SQUID magnetometers are made with a design principle same as for a dc-SQUID on bi-crystal substrates using different slit structure and asymmetric junctions with width ratios of 1/2, 1/3, and 1/4. The 1/f noise behavior of the above devices while is lower compared to that of the conventional designs, show a dependence on the width of the larger junction, increasing with the increase of the junction width. A new multi-junction design for gradiometers is also presented implementing the same principle using asymmetric junctions while avoiding any large YBCO film weak links on the substrate grain boundaries.

II10.16
STEP-EDGE STRUCTURE DEPENDENCE OF 1/F NOISE IN rf-SQUIDs AND THE EFFECT OF IBE PARAMETERS. J. Schubert, Y. Zhang, W. Zander, M. Fardmanesh Institut fuer Schicht- und Ionentechnik (ISI), Forschungszentrum Juelich, Juelich, GERMANY.

Step-edge junction rf-SQUID gradiometers and magnetometers are made using PLD YBCO films on $LaAlO_3$(100) and $SrTiO_3$(100) substrates. Effects of the step-edge structure, prepared by different IBE parameters, as well as the influence of reused substrates on the 1/f noise and signal of the SQUIDs have been investigated. For the normal incident ion beam etched steps, a hard layer of re-deposited material is found to form on the side-walls of the steps standing up to e.g. a few hundred nm height above the step-edge for 300 nm deep steps on $LaAlO_3$. This is found to be much less for the steps on $SrTiO_3$ substrates. The re-deposited layer is found to strongly reduce the yield of the SQUIDs on $LaAlO_3$ substrates and drastically increase the 1/f noise for the working devices. The SQUID signal on $SrTiO_3$ substrates is found to be less sensitive to the re-deposited material at the edge of the steps. SQUIDs made on $LaAlO_3$ substrate with steps etched using different angled incident ion beam, have shown a higher reproducibility and lower 1/f noise behavior avoiding re-deposition of the material at the steps. The detailed effects of the step etching parameters on the noise and signal of the devices are presented in this work.

II10.17
MICROWAVE AND DC FLUXON DYNAMICS IN BICRYSTAL YBCO GRAIN-BOUNDARY JOSEPHSON JUNCTIONS.
H. Xin,[1,2,3] D.E. Oates,[1,2] G. Dresselhaus,[1,3] M.S. Dresselhaus[1]; [1]Massachusetts Inst of Tech, Cambridge, MA, [2]Lincoln Laboratory, Lexington, MA, [3]AFRL, Hanscom AFB, Bedford, MA.

Measurements of the power-dependent and dc-magnetic-field-dependent microwave (rf)impedance $Z_S(H_{\rm rf}, H_{\rm dc})$ of a YBCO suspended microstrip resonator with an engineered bicrystal grain-boundary Josephson junction across the center are presented. By measuring the fundamental and first overtone modes of the resonator, effects of the junction can be separated from those of the background film, and we can infer the microwave impedance $Z(H_{\rm rf}, H_{\rm dc})$ of the junction. The suspended microstrip geometry has no superconducting ground plane so that small calibrated magnetic fields can be applied to the junction. Both rf power dependence and dc magnetic field dependence of Z were studied at frequencies 4 to 8 GHz for temperatures ranging from 5 to 75 K. The second and third harmonics generated by the grain boundary junctions were also measured as functions of rf power and external dc magnetic field. The applied dc magnetic field ranged from 0 to 1000 Oe. The measurements show that the dc magnetic field reduces the rf critical current of the junction. Additionally, as the dc magnetic field was scanned in fine steps at fields of the order of 1 to 10 Oe, with constant rf input power (constant $H_{\rm rf}$), changes of the microwave impedance by greater than a factor of two were observed. This behavior is consistent with single dc fluxons entering and interacting with the rf field in the junctions. Experimental results will be compared with various model predictions. The results will be related to the origins of the nonlinear microwave impedance of epitaxial thin films. This work is supported by Air Force Office of Scientific Research, account number F49620-98-1-0021.

II10.18
JOSEPHSON PHENOMENOLOGY AND MICROSTRUCTURE IN YBCO ARTIFICIAL GRAIN BOUNDARIES CHARACTERIZED BY MISALIGNMENT OF THE C-AXES. F. Tafuri, INFM, Dip. Ingegneria dell'Informazione, Seconda Universita di Napoli, Aversa (CE), ITALY; F. Miletto Granozio, F. Carillo, F. Lombardi, U. Scotti Di Uccio, INFM-Dip. Scienze Fisiche dell'Universit di Napoli Federico II, Napoli, ITALY; K. Verbist, G. Van Tendeloo, EMAT, University of Antwerp (RUCA), Antwerp, BELGIUM.

Several different kinds of YBCO grain boundary Josephson junctions, all characterized by a 45° misalignment of the c-axes have been obtained by employing a recently implemented biepitaxial technique. Different kinds of boundaries are obtained by using either MgO or CeO_2 as seed layers. Junctions based on these grain boundaries exhibit good Josephson properties, which could be fruitfully used for applications. High values of the IcRn product and a Fraunhofer-like dependence of critical current on the magnetic field, differently from traditional biepitaxial junctions, have been obtained by using an MgO seed layer. The correlation between transport and structural properties has been investigated by high resolution electron microscopy, which was also performed on previously measured junctions, and by X-ray Diffraction. The presence of atomically clean basal plane faced tilt boundaries, among other types of interfaces, has been shown. The possibility of selecting these kind of boundaries by controlling film growth, and their possible advantages in terms of reproducibility and uniformity of the junction properties, are presented. The possibility of employing these junctions to explore the symmetry of the order parameter is also discussed.

II10.19
FABRICATION AND PROPERTIES OF YBCO COATED CONDUCTOR ON METALLIC TAPE SUBSTRATE. Guansen Yuan, Jian Yang, Dongqi Shi, Superconducting Materials Research Center, General Research Institute for Nonferrous Metals, Beijing, CHINA.

The YBCO coated conductor as a second generation of high-Tc superconducting tape has widely attracted people's attention. We have deposited YBCO superconducting film with CeO_2 and YSZ buffer layers on rolling-textured Ni substrates by magnetron sputtering system. The metal tape substrates were formed by progressive rolling of high pure nickel at room temperature and recrystallized by high temperature annealing. After well-polished nickel substrates, the CeO_2 buffer layer was deposited on the cube-textured Ni surface at temperatures in the range 200°C to 400°C with $Ar : H_2 = 9 : 1$ sputtering atmosphere using RF magnetron sputtering, and then the second buffer layer was deposited at 700°C to 800°C in $Ar : O_2 = 2 : 1$. Finally the YBCO was grown on the buffers using an inverted cylindrical DC magnetron sputtering at the same condition as the YSZ deposit. X-ray diffraction shows that the buffer layers were pure c-axis orientation, and 11° and 15° FWHM in-plane texture from Φ-scans for CeO_2 and YSZ respectively. AES analyses indicate that the buffer layers effectively prevented oxidation and interdiffusion of the Ni surface. The YBCO film was grown epitaxially on buffer layers and critical current density reaches $6x10^5$ A/cm^2 at 77K in zero field.

II10.20
MAGNETIC FIELD SCANNER OF HIGH-T_c SUPERCONDUCTORS. H.R. Kerchner, and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

A mutual inductor is used to scan high-T_c superconductive tapes to show spatially varying critical current densities. A superconductive coated conductor placed between coaxially aligned primary and secondary windings shields the secondary from a small ac magnetic field of current supplied to the primary. No secondary signal is induced until the primary current and the induced magnetic field, enhanced by a ferrite core, induces critical current density in the superconductive material. Similarly multi-filamentary tape reduces the mutual inductive coupling roughly to the volume fraction of the matrix. By varying the primary ac current we deduce both the magnitude of the critical current density and the carrier density of the superconductor. Such scans at liquid-nitrogen temperature, T=77 K, have been carried out for multi-filamentary BSCCO tape and for YBCO film grown on a rolled, annealed, biaxially textured substrate (RABiTS).

II10.21
FABRICATION OF COATED YBCO SUPERCONDUCTOR ON METAL SUBSTRATES BY METAL ORGANIC CHEMICAL VAPOR DEPOSITION. V. Selvamanickam, G. Galinski, C. Trautwein, G. Carota, J. DeFrank and P. Haldar, Intermagnetics General Corporation, Latham, NY; U. Balachandran, and M. Chudzik, Argonne National Lab, Argonne, IL; P. Arendt, S. Foltyn, B. Newnam and D.E. Peterson, Los Alamos National Lab, Los Alamos, NM.

Intermagnetics is developing a wide range of HTS conductors for electric power applications such as Transformers, Fault Current Limiters, Transmission Cables and Generators. In addition to manufacturing kilometer-lengths of Bi-2223 Conductor for these applications, Intermagnetics has been developing Coated YBCO Superonductor as a high performance, low cost alternative HTS conductor. A Metal Organic Chemical Vapor Deposition (MOCVD) process is being developed at Intermagnetics to fabricate Coated YBCO Superconductor using metal substrates. MOCVD is a viable alternative to Physical Vapor Deposition techniques as an industrial process for large-scale YBCO conductor manufacturing. Nickel alloy substrates with a biaxially-textured buffer layer of zirconia were used for YBCO deposition. The biaxially-textured buffer layers were fabricated at Argonne and Los Alamos National Labs by Ion Beam Assisted Deposition (IBAD). The in-plane texture of the IBAD buffer layers used for YBCO deposition ranged from 15 to 25 degrees FWHM. MOCVD reactor design, precursor delivery scheme and process conditions were optimized to deposit epitaxial YBCO films with high Transition Temperature (T_c) and high Critical Current Densities (J_c). YBCO films with in-plane texture less than 4 degrees FWHM, T_c of 90 K and J_c greater than 1 MA/sq.cm. at 77 K have been fabricated by MOCVD using the IBAD substrates. Details of texture, composition, film coverage, transition temperature and current density of the YBCO films will be discussed in the presentation.

II10.22
EPITAXIAL GROWTH OF CONDUCTIVE BUFFER LAYERS FOR THE DEVELOPMENT OF COATED CONDUCTORS. Tolga Aytug, Judy Z. Wu, Univ of Kansas, Dept. of Physics and Astronomy, Lawrence, KS; Claudia Cantoni, Darren T. Verebelyi, David P. Norton, David K. Christen, Oak Ridge National Laboratory, Solid State Div., Oak Ridge, TN; Amit Goyal, Eliot D. Specht, Oak Ridge National Laboratory, Metals and Ceramics Div., Oak Ridge, TN; Mariappan Paranthaman, Oak Ridge National Laboratory, Chemical and Analytical Sciences Div., Oak Ridge, TN.

Epitaxial buffer layers of electrically conductive TiN, ZrN and $LaNiO_3$ have been deposited on biaxially textured nickel tapes by sputter deposition. Epitaxial deposition of TiN and ZrN films were achieved reactively by dc-magnetron sputtering techniques at substrate temperatures ranging between 600-650°C, at rates of 3Å/sec. For the growth of $LaNiO_3$ films, rf-magnetron sputtering techniques were employed. While the deposition temperatures of 400-500°C yield epitaxial films, higher temperatures lead to both formation of NiO and decomposition of $LaNiO^3$ phase. From the X-ray θ-2θ, ω, ϕ-scans, and pole figure analyses, all the films show good in-plane and out-of-plane orientations. In addition, smooth, dense and continuos surface morphologies were observed by SEM for all the films deposited on biaxially textured nickel substrates. We also describe the development of new buffer layer configurations directed towards the implementation of high temperature superconducting coated conductors. The relevance of the conductive films for this and other potential applications will be discussed.

II10.23
THE MICROSTRUCTURE OF Y123 COATED CONDUCTORS WITH UNDERLYING CERIA AND IBAD YSZ BUFFER LAYERS. Terry Holesinger, Harriet Kung, Steve Foltyn, Paul Arendt, Quanxi Jia, Eric Peterson, John Bingert, Jim Smith, Robert Dickerson, Paul Dowden, Ron DePaula, James Groves and J. Yates Coulter.

The microstructure of Y123 thick films on Inconel 625 metallic substrates with intervening buffer layers of ion-beam deposited, yttria-stabilized zirconia (IBAD YSZ) and ceria was examined by transmission and scanning transmission electron microscopy (TEM and STEM). The best, one-meter Y-123 coated conductor had a critical current (Ic) of 122 A and critical current density (Jc) of 0.87 MA/cm^2 at 75 K and self-field. Several segments of these tapes were found to have Ic values in excess of 200 A. All layers showed good adhesion to the underlying layer or substrate. A proctective chromium oxide layer was found to form during processing between the substrate and the IBAD YSZ layer. Chemical reactions between Y-123 and the ceria buffer layer resulted in the random formation of $BaCeO_3$ and $YCuO_2$. The Y-123 grains were found to contain a large number of dislocations, stacking faults, and coherent precipitates of other phases.

II10.24
OXIDE BUFFER LAYERS ON TEXTURED-Ni SUBSTRATES BY SOL-GEL PROCESS FOR HIGH CURRENT YBCO CONDUCTORS. T.G. Chirayil, M. Paranthaman, D.B. Beach, J.S. Morrell, C.M. David, A. Goyal, D.F. Lee, D.M. Kroeger, R. Feenstra, D.T. Verebelyi and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

The RABiTS (Rolling-Assisted Biaxially Textured Substrates) process is one of the leading techniques used to develop high J_c superconducting tapes for high temperature, high field applications. The process involves the epitaxial growth of buffer layers on textured metal substrates followed by the deposition of the in-plane aligned YBCO film. We have developed a cost-effective and easily scalable solution technique for the epitaxial growth of rare-earth oxide buffer layers on textured Ni (100) substrates. The precursor solution for the oxide buffer layers was prepared by an all alkoxide sol-gel route and was deposited on the substrates by spin coating or dip coating methods. Under optimum processing conditions, crack free dense films were obtained. The oxide buffer layers exhibited a strong c-axis orientation on the Ni (100) substrate and the phi and omega scans indicated good in plane and out of plane orientations. The X-ray (111) pole figure showed a single cube epitaxy. High J_c YBCO films of over 1 MA/cm^2 at 77 K and zero field were grown on sol-gel buffered Ni substrates. This research was sponsored by the U.S. Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences and Office of Energy Efficiency and Renewable Energy, Office of Power Technology-Superconductivity Program. This research was partly supported by Oak Ridge Institute for Science and Education. Oak Ridge National Laboratory is managed by Lockheed Martin Energy Research Corporation for the U.S. Department of Energy under contract #DE-AC05-96OR22464.

II10.25
COMPARISON OF MICROSTRUCTURE IN YBCO FILMS DEPOSITED ON ALTERNATIVE OXIDE BUFFER LAYERS ON RABiTS. Chau-Yun Yang, S.E. Babcock, Department of Materials Science and Engineering and Applied Superconductivity Center, University of Wisconsin, Madison, WI; A. Goyal, F.A. List, J.E. Mathis, C. Park, R. Feenstra, M. Paranthaman, D.F. Lee, D.P. Norton and D.M. Kroeger, Oak Ridge National Lab, Oak Ridge, TN.

The microstructure of pulsed laser deposited YBCO films grown on three different buffer layer materials, YSZ, CeO2, and Yb2O3, was analyzed with a view toward identifying current limiting defects and understanding the role of the buffer layer material in YBCO coated conductors. YBCO films deposited directly onto YSZ have an island microstructure composed of sub-micron sized grains separated by very low angle grain boundaries. Films over one micron thick generally contain columnar pores spaced 1-2 microns apart that meander in the (001) plane. The YBCO film deposited on CeO2 cap layers on YSZ do not show the same island microstructure, rather a single YBCO grain that contains twin boundaries on both sets of {110} planes extend over the entire 10 to 20 microns observable area of TEM specimens. Columnar pores with a distinct, crystallographically oriented rectangular cross-section, dimensions of the order of 1 micron by 0.1 micron and a one-micron spacing are observed. Perhaps of more consequence, the YBCO appears not to grow over the grain boundaries in the substrate, leaving a contiguous network of ~0.1 micron gaps in the superconductor. The results is a films with a low Jc value of just ~0.2 MA/cm2. A high Jc (1.7 MA/cm2) YBCO film grown on a Yb2O3 buffer layer has an island microstructure like that of YBCO on YSZ. These results suggest that microstructural details of length scale ranging from sub-micron to many tens of microns depend on the buffer layer. This work is supported by US-DOE through ORNL. The NSF-MRSEC at the University of Wisconsin (UW) provides partial support for the UW electron microscopy facilities.

II10.26
FABRICATION OF Y-SYSTEM COATED CONDUCTOR ON METAL SUBSTRATE BY LPE METHOD. Hobara Natsuro, Kazuomi Kakimoto, Yuichi Nakamura, Teruo Izumi, Yuh Shiohara, Superconductivity Research Laboratory (SRL), International Superconductivity Technology Center (ISTEC), Tokyo, JAPAN, Koso Fujino, Kazuya Ohmatsu, Sumitomo Electric Industries, LTD, Osaka, JAPAN.

In application of the long-length RE-system coated conductor, it is important to obtain three dimensional crystal orientation, high critical current, those homogenity and high production rate. The LPE process is advantageous to fabricate thick film at high growth rate with high superconducting properties as compared with the vapor process. In order to grow superconducting film on metal substrates by LPE, it has to be found out to prevent the reaction between the meltal substrate and the oxide melt. In this paper, the reactivity between the metallic substrate, the buffer layers and the oxide melt was investigated. A hastelloy was used as the metallic substrate and MgO was doposited on this substrate as the buffer layer by the laser ablation method. These substrates were dipped at 1273K into the melt. Two different kinds of melt were prepared. One had the composition of Y:Ba:Cu=0.6:37.3:62.1 and the other was basically the same composition but saturated with MgO. The cross section of the dipped samples was observed by the optical microscopy. In the results, the substrate was dissolved completely in the case of the dipping into the non-saturated melt. On the other hand, the dissolution was scarcely observed in the case of the dipping into the melt saturated with MgO. Additionally, the YBa2(Cu,Mg)3Oy layer was grown using this technique without reaction between melt and matel substrate. Futhermore, the superconducting YBa2Cu3Oy phase which reveals Tc of 92K was successflly grown on the substituted layer by LPE method. This work was supported by New Energy and Industrial Technology Development Organization (NEDO).

II10.27
INVESTIGATION OF EPITAXIAL OXIDE FILM GROWTH ON BIAXIALLY TEXTURED METALS USING REFLECTION HIGH ENERGY ELECTRON DIFFRACTION. Chan Park, David P. Norton, John D. Budai, Amit Goyal, David K. Christen, Darren Verebelyi, Dominic Lee and M. Paranthaman, Oak Ridge National Laboratory, Oak Ridge, TN.

The epitaxial growth of high temperature superconducting films on so-called Rolling Assisted Biaxially Textured Substrates (RABiTS^{TM}) represents one approach to the formation of a highly aligned deposited conductor possessing a high critical current. A key component to this approach is the formation of an epitaxial oxide buffer layer architecture on the biaxially-textured metal surface that is mechanically robust and chemically inert with respect to both the substrate and the superconducting film. In this talk, we will report on the nucleation, epitaxy, and properties of various oxide buffer layers grown on (001) Ni and Ni-Cr tapes using pulsed-laser deposition. Reflection high-energy electron diffraction is used to study the nucleation and epitaxy of various oxide materials on these biaxially textured substrates. These RHEED results are directly correlated with the microstructure and transport properties of the oxide films. We will also report on recent progress in developing a single layer buffer architecture suitable for the RABiTS^{TM} approach. This research was sponsored by the Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corp., for the U.S. Department of Energy, under contract DE-AC05-96OR22464.

II10.28
GROWTH AND CHARACTERIZATION OF IBAD MGO BUFFER LAYERS ON METALLIC SUBSTRATES FOR HIGH PERFOMANCE YBCO SUPERCONDUCTING FILMS. James R. Groves, Paul N. Arendt, Q.X. Jia, Stephen R. Foltyn, Raymond F. DePaula, Terry G. Holesinger, Eric J. Peterson, Philip C. Yashar, Harriet Kung, Materials Science Division, Los Alamos National Laboratory; Michael R. Fitzsimmons, Los Alamos Neutron Science Center, Los Alamos National Laboratory, Los Alamos, NM.

We describe our progress on implementing magnesium oxide (MgO) as a textured buffer layer for subsequent heteroepitaxial deposition of yttrium barium copper oxide (YBCO) superconductors on technically useful metallic substrates. Ion-beam assisted deposition (IBAD) was used to form bi-axially textured MgO template films. An initial layer of amorphous Si_3N_4, the IBAD MgO film, an e-beam deposited MgO film, a pulsed-laser deposited (PLD) $SrTiO_3$ buffer layer, and the final PLD YBCO film constitute the system architecture. X-ray diffraction phi scans for these films resulted in full width half-maximum (FWHM) values of 7 degrees for the MgO template and 5 degrees for the best-textured PLD YBCO film. Our best transport critical current

density (75K, self-field) for these YBCO films was 0.78 MA/cm^2 for a thickness of 0.8 micrometer. We also present data on the quantification of the 10 nm thick IBAD MgO layer with in-situ reflected high-energy electron diffraction (RHEED) and ex-situ x-ray grazing incidence diffraction (GID).

II10.29
DEVELOPMENT AND USE OF CUBE TEXTURED NON-MAGNETIC NI-ALLOY TAPES AS SUBSTRATES FOR YBCO FILM GROWTH. Norman Reger, Laura Fernandez, Bernd de Boer, Jörg Eickemeyer, Bernhard Holzapfel, Ludwig Schultz, Institute for Solid State and Materials Research, Dresden, GERMANY.

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

II10.30
TEM ANALYSIS OF SILVER METAL CONTACTS ON YBCO HIGH Tc SUPERCONDUCTING FILMS. Ron Anderson, Mark Hudson, IBM Analytical Services, Hopewell Jct., NY; Claire Pettiette-Hall, John Burch, TRW Space and Electronics Group, Redondo Beach, CA.

There are very few references to TEM analytical studies of metal contacts to high temperature superconducting (HTSC) materials because most metals do not adhere well to these complex oxides and attempts to prepare cross section TEM specimens fail because of delamination problems. Conventional TEM preparation methods, where the cross section specimen is mechanically thinned to 30 to 50 microns in thickness and then ion milled to electron transparency, are difficult to prepare because: HTSC materials, in our case YBCO, may not be mechanically polished in water, and even very short ion milling steps cause severe radiation damage to the HTSC and considerable thickness variations in the final cross section due to differential milling rates of the various layers. The use of a focused ion beam (FIB) tool is problematical because of electrostatic discharge problems and severe radiation and implantation effects from the 30 to 50 KeV ion beam. We have developed TEM specimen preparation protocols for preparing Ag contacts on YBCO films that are free of water etching artifacts and can be performed with either no, or at most one or two minutes, of ion mill thinning. These methods are based on the tripod polisher tool invented in the IBM Analytical Services facility. Mechanical tripod polishing is performed in propylene glycol to a thickness of less than 0.5 micron. Ion milling, when needed, is performed at a very low angle of incidence directed from the substrate to the film stack only for no more than one or two minutes. The entire process was abetted greatly by the design and implementation of a special TEM device macro on the HTSC chip. The method yields large-area, artifact-free specimens with no differential thinning of the silver metal, YBCO HTSC, the strontium titanate insulator, or the chip substrate. The specimens are thin enough for atomic resolution TEM analysis over several hundred thousand square microns. Numerous examples will be shown with emphasis on the Ag to YBCO interface structure.

II10.31
REEL-TO-REEL CONTINUOUS DEPOSITION OF OXIDE BUFFER LAYERS ON BIAXIALLY TEXTURED Ni TAPES BY RF SPUTTERING. F.A. List, D.F. Lee, D.M. Kroeger, X. Cui, A. Goyal, M. Paranthaman and P.M. Martin, Oak Ridge National Laboratory, Oak Ridge, TN.

A reel-to-reel, RF sputtering system has been developed to continuously deposit epitaxial oxide buffer layers on meter long lengths of biaxially textured Ni substrates. The deposition system consists of two interconnected sputtering chambers for YSZ and CeO^2 buffer layer deposition. The most commonly used buffer layer architecture consits of a CeO^2 layer deposited by electron-beam evaporation or sputtering, followed by sputtering of YSZ and CeO^2 layers. X-ray diffraction measurements suggest that delamination of YSZ deposited by RF sputtering under moving tape condition is associated with loss of oxygen in the initial e-beam or sputtering deposited CeO^2 film. By supplying sufficient water vapor during the deposition process, length of delamination-free YSZ and CeO^2 films can be grown epitaxially on previously e-beam or sputtering coated CeO^2-buffered Ni tapes under moving tape condition. The microstructures of these buffer layers will be presented. We will also report the results of YBCO films deposited on short segments of these buffered substrates. This work was conducted in part under a CRADA with 3M/Southwire/LANL funded by the U.S. Department of Energy, the Office of Energy Efficiency and Renewable Energy, and the Office of Energy Research. Oak Ridge National Laboratory is managed by Lockheed Martin Energy Research Corporation for the U.S. Department of Energy under contract # DE-AC05-96OR22464.

II10.32
REEL-TO-REEL DEPOSITIOIN OF STOICHIOMETRIC Y-BAF_2-Cu PRECUSOR FILMS ON EPITAXIALLY OXIDE BUFFERED NI TAPES BY ELECTRON BEAM EVAPORATION. X. Cui, F.A. List, D M. Kroeger, D.F. Lee, A. Goyal, M. Paranthaman, E.D. Specht and P.M. Martin, Oak Ridge National Laboratory, Oak Ridge, TN.

One of the steps to grow a high current density YBCO film on biaxially textured, oxide buffered, Ni tape (RABiTS) is to deposit high quality Y-BaF_2-Cu precursor film. This film should be stoichiometric in cation composition (i.e., Y:Ba:Cu=1:2:3) for the entire precursor deposition process. Rutherford backscattering spectroscopy studies indicate that partial pressure of water vapor during deposition has a significant effect on cation deposition rates and the oxygen content in the precursor film. Oxygen content of the precursor is increased and its stability upon exposure to air is improved by the introduction of water vapor during precursor deposition. High quality precursor films with a thickness of 300 nm were deposited on lengths of RABiTS in a reel-to-reel, electron beam evaporation system. Properties of post-annealed YBCO films will also be presented. This work was conducted in part under a CRADA with 3M/Southwire/LANL funded by the U.S. Department of Energy, the Office of Energy Efficiency and Renewable Energy, and the Office of Energy Research. Oak Ridge National Laboratory is managed by Lockheed Martin Energy Research Corporation for the U.S. Department of Energy under contract # DE-AC05-96OR22464.

II10.33
BIAXIALLY TEXTURED $LaAlO_3$ BUFFER LAYERS ON TEXTURED Ni SUBSTRATES FOR HIGH-TEMPERATURE SUPERCONDUCTING TAPES. C.M. Carlson[1], P.A. Parilla[2], D.S. Ginley[2], R.N. Battacharya[2], R.D. Blaugher[2], Z.F. Ren[3], A. Goyal[4], M. Paranthaman[4], D.M. Kroeger[4], D.K. Christen[4], M. Tomsic[5]; [1]University of Colorado, Boulder, CO, [2]National Renewable Energy Laboratory, Golden, CO, [3]Boston College, Boston, MA, [4]Oak Ridge National Laboratory, Oak Ridge, TN, [5]Euro-Plastronic, Inc., Tipp City, OH.

One of the challenges in making commercially useful coated conductors is the fabrication of a biaxially textured buffer layer that serves as the structural template for subsequent superconductor growth. This biaxial texture is necessary to maximize both the critical current and magnetic field performance of the superconductor. $LaAlO_3$ (LAO) single crystal substrates have previously been shown to have several advantages for the growth of Tl-based superconducting films especially with regard to proper phase formation. LAO single crystals have also supported high-quality films of $YBa_2Cu_3O_7$ (YBCO). We will describe the development of an epitaxial tri-layer buffer scheme consisting of LAO/$LaNiO_3$/CeO_2 deposited in-situ on biaxially textured Ni (100) substrates using pulsed laser deposition (PLD). The full-width-at-half-maximum of the (110) LAO phi scan peak is $\sim$ 10-12°. The use of $LaNiO_3$ (LNO) as the middle layer overcomes problems with previous tri-layer buffer configurations. Specifically, LNO has both a more consistent growth orientation and a lower deposition temperature on CeO_2. However, the proper phase formation of LNO requires the presence of O_2 during deposition. This can cause problems in the surface morphology due to the reaction of Ni with O_2, if the PLD parameters are not properly controlled. Issues concerning both the crystal orientation and surface morphology will be discussed. We will also present the results of superconducting films grown on the LAO/LNO/CeO_2/Ni substrates.

II10.34
EFFECT OF EXTREME SUBSTRATE SURFACE ROUGHNESS ON THE STRUCTURAL AND SUPERCONDUCTING PROPERTIES OF YBCO FILMS. Q.X. Jia, S.R. Foltyn, P.N. Arendt, H. Kung, J.F. Smith, R.F. DePaula, Y. Fan, J.R. Groves, M. Ma and P.C. Dowden, Superconductivity Technology Center, MS, Los Alamos National Laboratory, Los Alamos, NM.

Substrate finishing plays a big role in determining the superconducting properties of YBCO films that are used both for electronic devices and second generation coated conductors. We have

investigated the effect of extreme substrate surface roughness on the structural and superconducting properties of YBCO films. Results are compared for films deposited on highly polished and unpolished strontium titanate substrates. The YBCO films on unpolished substrates show reduced critical transition temperature and current density. The crystallographic misalignment, both out-of-plane and in-plane, for the films on unpolished substrates increases compared to the films on polished substrates. Scanning and transmission electron microscopies show poor connectivity in the films on extremely rough substrates.

II10.35
EVALUATION OF NICKEL BASE ALLOYS AS METALLIC SUBSTRATES FOR GENERATION II TEXTURED YBCO COATED CONDUCTORS. Rama M. Nekkanti, Lyle B. Brunke, Venkat Seetharaman, UES, Inc., Dayton, OH; John A. McDaniel, Iman Maartense, Gerry Landis, Dave Dempsey, University of Dayton Research Institute, Dayton, OH; Gregory K. Kozlowski, Wright State University, Dayton, OH; Dave Tomich, Rand Biggers, Timothy Peterson, Materials and Manufacturing Directorate, Paul N. Barnes and Charles E. Oberly, Propulsion Directorate, Air Force Research Laboratories, Wright-Patterson Air Force Base, OH.

High current densities ($\sim 10^6$ A/cm^2) were recently demonstrated in short specimens by epitaxial growth of YBCO on biaxially textured polycrystalline nickel substrates. The process has the potential for long-length processing of the conductors and is being pursued for practical applications. Unfortunately, the low strength of recrystallized nickel requires delicate handling and its low electrical resistivity and high magnetic permeability (leading to eddy current losses) pose a problem for AC applications. There is a definite need for substrate materials characterized by high strength, good oxidation resistance, high electrical resistivity and low magnetic permeability. The present work is focused on binary nickel base alloys as alternate metallic substrates. High purity Ni-Cr binary alloys were melted and hot/cold worked to obtain thin sheets/strips. Results to date on the development of texture by thermo-mechanical processing of these alloys will be presented and compared with those from nickel.

II10.36
FABRICATION OF 3-m LONG Y-123 COATED CONDUCTORS BY IBAD METHOD. Yasuhiro Iijima, Mariko Kimura, Takashi Saitoh, Fujikura Ltd., Material Technology Lab., Tokyo, JAPAN; Kaoru Takeda, Super-GM, Osaka, JAPAN.

$YBa_2Cu_3O_{7-x}$ (Y-123) conductors is a hopeful candidate for practical use of superconducting wires in liquid nitrogen temperature. In order to derive the intrinsic pinning properties of Y-123, biaxially aligned structure is so far the only way, which can avoid its severe weaklink problems. Ion-beam-assisted deposition (IBAD) method offers an ideal tape-shaped substrate for Y-123 conductors which have both biaxially aligned surface structure and sufficient mechanical strength and flexibility. YSZ films of 0.7-1.0 mm thick were continuously deposited by IBAD on Ni-based alloy tapes with lengths of 1.2 -5.0 m, and tape shifting speeds of 2.5 - 25 cm/h. The YSZ films were uniformly coated with the in-plane mosaic spread of 17-19 degrees. Y-123 films were deposited by pulsed laser deposition (PLD) on the YSZ template layers with tape shifting speed of 1.0 m/h. The minimum in-plane mosaic spread for the Y-123 films was 9.8 degrees. I_c of 19.7 A and J_c of $2.3x10^5$ A/cm^2 were obtained at 77K, 0T, in a sample with 0.85 mm thick at the length of 1.9 m. In order to avoid slight interdiffusion and lattice mismatch between Y-123 and YSZ, thin Y_2O_3 films were deposited on several YSZ templates by PLD. I_c of 44.9 A and J_c of $3.8x10^5$ A/cm^2 were obtained at the length of 0.9 m, in a sample which had a 1.2 mm thick Y-123 film with a 0.1 mm thick Y_2O_3 layer. The longitudinal uniformity of J_c values was clearly improved by the intercalation of the Y_2O_3 layer. The trials for further length are in progress and the scheme of practical scale-up for both YSZ and Y-123 films would be discussed. This work was carried out as a part of R&D on superconducting technology for electric power apparatuses under the New Sunshine Project of AISI, MITI, being consigned by NEDO.

II10.37
CRITICAL CURRENT DENSITY MEASUREMENTS OF ONE METER LONG, HIGH CURRENT, YBCO COATED CONDUCTOR TAPES. Raymond F. DePaula, J. Yates Coulter, Michael J. Demkowicz, Eric J. Peterson and Fred M. Mueller, Los Alamos National Laboratory Superconductivity Technology Center, Los Alamos, NM.

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

II10.38
TiN BUFFER LAYERS ON TEXTURED Ni FOR YBCO SUPERCONDUCTING TAPE. I.W. Kim, S. Sambasivan and S.A. Barnett, Applied Thin Films, Inc.; A. Goyal, Metals and Ceramic Division, Oak Ridge National Laboratory; C.E. Oberly, C. Varanasi, P. Barnes, R. Biggers, Air Force Research Laboratory, Dayton, OH.

Yttria-stabilized Zirconia and Ceria have been widely used as epitaxial buffer layers between roll-textured Ni tapes and the YBCO layer in superconducting tapes. In this talk, we describe results on an alternate buffer layer, TiN. TiN has the advantages of high electrical conductivity, potentially allowing shunting of current into the Ni tape, better mechanical toughness than oxides, and relative ease of high-rate deposition by reactive sputtering. While nitride growth avoids any difficulty with NiO formation at the substrate, there is a potential problem with oxidation of the nitride during YBCO growth. Epitaxial TiN films with thickness ~300nm were grown on Ni RABiTS by reactive magnetron sputtering at ~ 600-650°C. X-ray diffraction (XRD) phi-scans of the TiN (111) reflection showed cube-on-cube epitaxy, despite large lattice mismatch (18.5%). Typical full width half maximum (FWHM) values were 7°, less than that of the textured Ni substrate, ~ 8°. Films were also grown on MgO substrates, which has an excellent lattice match with TiN, for initial studies of YBCO growth and superconducting properties. Pulsed-laser deposition was used to grow a thin (<<100 nm thick) layer of ceria on the TiN-coated MgO, followed by an ~300nm thick YBCO layer. The XRD phi scan showed a YBCO (226) reflection with a FWHM of < 8°, and XRD rocking curve showed FWHM of <1° on (005) reflection, indicating excellent in-plane and out-of-plane texture. A superconducting critical transition temperature (T_c) of 89 K was measured by AC susceptibility. A critical current density (J_c) of $6x10^5$ A/cm^2 was reproducibly obtained at 77 K by whole body transport current measurement in self field using a 1mv/cm criteria.

II10.39
THE ROLE OF GRAIN BOUNDARY PLANE AND FACETING IN THE CURRENT CARRYING CAPABILITY OF HIGH-ANGLE GRAIN BOUNDARIES IN YBCO. M. Mironova, S. Stolbov, and K. Salama, Univ of Houston, Texas Center for Superconductivity, Houston, TX.

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

II10.40
SUPERCONDUCTING PROPERTIES OF [001] TILT YBCO THICK FILM GRAIN BOUNDARIES ON $SrTiO_3$ AND TEXTURED Ni-SUBSTRATE USING THE BaF_2 EX-SITU POST REACTION PROCESS. Qiang Li, V.F. Solovyov, H.J. Wiesmann, Y. Zhu and M. Suenaga, Department of Applied Science, Brookhaven National Laboratory, Upton, NY.

We present a study of superconducting properties of [001] tilt YBCO thick film grain boundaries on $SrTiO_3$ bicrystal substrates and textured Ni substrates. The YBCO films with thickness ranging from 0.5 to 5 micrometer were prepared using BaF_2 ex-situ post reaction process of the precursor by high rate e-beam deposition. The misorientation angles of the grain boundaries ranged from 0° to 24°. 2-5 micrometer wide strips containing a single [001] tilt grain

boundary can be isolated by either photolithographic or laser patterning. An electric configuration, with up to 5 voltage pairs located along the grain boundaries, was used to measure the resistance and the voltage-current (V-I) characteristics of the grain boundary at various section. The microstructure of the grain boundaries was examined using advanced TEM. We shall discuss the relation between the local microstructure and transport properties of these grain boundaries at micrometer length scale. *This work was supported by the U. S. Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under contract No. DE-AC02-98CH10886.

II10.41
SUPERCONDUCTING YBCO THIN FILM ON MULTI-CRYSTALLINE Ag FILM EVAPORATED ON MgO SUBSTRATE. Jacob Azoulay, Armen Verdyan, Igor Lapsker, Center for Technological Education Holon affiliated with Tel-Aviv University, Dept of Science, Holon, ISRAEL.

Superconducting $YBa_2Cu_3O_{7-\delta}$ films were grown by resistive evaporation on multicrystalline silver film which was evaporated on MgO substrate. A simple inexpensive vacuum system equipped with resistively heated boat was used for the whole process. Silver film was first evaporated on MgO substrate kept at 400°C during the evaporation after which with no further annealing a precursor mixture of yttrium small grains and Cu and BaF2 in powder form weighed in the atomic proportion to yield stoichiometric $YBa_2Cu_3O_7$ was evaporated.
The films thus obtained were annealed at 740°C under low oxygen partial pressure of about 1Pa for 30 minutes to form the superconducting phase, X-ray diffraction and scanning electron microscopy techniques were used for texture and surface analysis. Electrical properties were determined using a standard dc four-probe for electrical measurements.
The physical and electrical properties of the YBCO films are discussed in light of the fact that X-ray diffraction measurements done on the silver film have revealed a multicrystalline structure.

II10.42
FABRICATION OF DIELECTRIC/NBCO MULTI-LAYER STRUCTURES. Michitomo Iiyama, Osami Horibe, Yasuo Oshikubo, Youichi Enomoto, ISTEC, Superconductivity Research Laboratory, Chiba, JAPAN.

NdBaCuO(NBCO) has outstanding characteristics among the 90K class superconductors. Particularly NBCO films have smooth and particle free surfaces, suitable for multi-layer structures. Therefore ramp-edge type Josephson junctions utilizing NBCO films have been extensively studied. Superconducting circuits require large area NBCO films with uniform characteristics to accomplish their appropriate operation. NBCO films have been deposited by either sputtering or pulsed laser deposition methods. We already reported that the deviation of the critical temperature was suppressed less than 5% over 4-cm-diam. area by the 90ÅKoff-axis RF sputtering method. The 2-in.-diam. PBN/PG/PBN heater with flat heating head was revealed to be effective to minimize the radial distribution of the specimen temperature mentioned above. Multi-layer structures with NBCO layers, either device structure or multi-layer inter-connection, can also be deposited sequentially by this sputtering apparatus attaching multiple targets. In this work, we report on the adjustment of fabrication conditions for dielectric/NBCO multi-layer structures. We utilized either LSAT or CeO as dielectric material. We would also like to describe about the in situ process to suppress the deviation of the critical current of NBCO layers, effective to enlarge the operation margin of the superconducting circuits. This work was supported by New Energy and Industrial Technology Development Organization.

II10.43
COMPETITION BETWEEN A-AXIS AND C-AXIS GROWTH IN SUPERCONDUCTING REBCO THIN FILMS : A NEW MODEL. F. Miletto, M. Salluzzo, U. Scotti di Uccio, INFM, Dipartimento di Scienze Fisiche, Universita di Napoli Federico II, Napoli, ITALY; I. Maggio-Aprile, y. Fischer Departement de Physique de la Matiere Condensee, Universite de Geneve, Geneve, SWITZERLAND.

The competition between a-axis and c-axis growth in $REBa_2Cu_3O_{7-x}$ (RE = rare earth or Y) superconducting thin films has been analysed. Stoichiometric and Nd-rich NdBaCuO thin films have been deposited by DC sputtering. The relative a/c-axis content has been estimated by X-ray diffraction and plotted as a function of deposition temperature. Comparison of obtained results with the experimental data available for other compounds yields clear evidence that a universal behaviour, valid for $REBa_2Cu_3O_{7-x}$ oxides, is obtained as a function of a normalised deposition temperature. A model for the nucleation of supercritical clusters from a multielemental adsorbed phase, allowing to face the problem of phase competition in complex compound, is presented. As a particular case, the competition between different phases of a single compound is analysed as a function of supersaturation. A general behaviour for the orientation dependence as a function of a normalised deposition temperature is thus obtained. It is shown that our experimental results about the relative a/c axis content fall on the theoretical curve. The same procedure is also successfully applied to all the data derived from the literature regarding the relative a/c axis content of other $REBa_2Cu_3O_{7-x}$ oxides.

II10.44
STRAIN, OXYGENATION AND DOPING EFFECTS ON SUPERCONDUCTIVITY OF $La_{2-x}Sr_xCuO_{4+\delta}$ THIN FILMS. Weidong Si and Xiaoxing Xi, Department of Physics, The Pennsylvania State University, University Park, PA.

We have measured normal-state and superconducting properties of $La_{2-x}Sr_xCuO_{4+\delta}$ thin films as a function of Sr content x, lattice strain and oxygen content. Strain was controlled by depositing $SrLaAlO_4$ buffer layer of different thickness on $SrTiO_3$, $SrLaGaO_4$ and $SrLaAlO_4$ substrates. An ozone/molecular oxygen mixture was used during cooling to achieve better oxygen uptake. We found that both full oxygenation and compressive in-plane strain are critical for the properties of the $La_{2-x}Sr_xCuO_{4+\delta}$ thin films, and perhaps the compressive strain makes the oxygenation much easier. Also, we found samples with x¡0.05 is superconducting while the bulk counterpart is insulating.

II10.45
DEPOSITION RATE MONITOR FOR LARGE-AREA PULSED LASER DEPOSITION. J.A. Greer, M.D. Tabat and D.B. Fenner, Epion Corporation, Billerica, MA; C. Lu, E. Augustyniak, Intelligent Sensor Technology, Mountain View, CA.

Applications using High Temperature Superconducting (HTS) thin films are expected to reach maturity very early in the next century. Such applications will include low-loss narrow band microwave filters for cellular phone repeater stations, microwave delay lines, and SQUIDS. Furthermore, YBCO films deposited onto metal tapes have applications in high-field supercondcuting magnets and motors. To date, the devices requiring the highest quality YBCO materials are still fabricated using Pulsed Laser Deposition (PLD). PLD has been ignored for production-oriented HTS applications as it was felt early on that industrial scale-up would not be possible for this process. Deposition equipment exists today that can handle up to 5-inch diameter substrates (or three 2-inch substrates, simultaneously). Furthermore, production PLD systems that can handle sixteen 3-inch, or twenty-four 2-inch diameter substrates are currently under development. Prototype reel-to-reel PLD equipment is also under development for metal tape applications as it is felt that the PLD plume shape and other unique features are ideal for this purpose. However, several other deposition techniques also work well, and co-evaporation is actively being pursued. PLD will readily compete with co-evaporation in terms of film quality, uniformity, and deposition rate. In order to make PLD a more mainstream production tool, improved in-situ deposition rate monitors will certainly be required for this process. We have developed a rate monitor for PLD based on Atomic Absorption (AA). A proto-type AA rate monitor for copper-vapor sensing has been built and incorporated onto a 5-inch PLD system. The design of the AA monitor and its performance characteristics will be presented along with associated YBCO film qualities.

II10.46
SUPERCONDUCTING PROPERTIES OF $YBa_2Cu_3O_7$ THIN FILMS ON $La_{0.7}Ca_{0.3}MnO_3$ FERROMAGNETIC TEMPLATE LAYERS. S. Freisem, J. Aarts, P.H. Kes, Kamerlingh Onnes Laboratory, University Leiden, THE NETHERLANDS; T. Nojima, Center for Low Temperature Science, Tohoku University, JAPAN; H.W. Zandbergen, Material Science Department, Delft University of Technology, THE NETHERLANDS.

In view of the interest in spin injection from a ferromagnet into a superconductor, we have investigated the superconducting properties of bilayers of $YBa_2Cu_3O_7$ / $La_{0.7}Ca_{0.3}MnO_3$ (YBCO/LCMO) grown by sputter deposition on $SrTiO_3$ (STO) substrates. The LCMO bottom film grows very smooth, leading to a high quality interface with the YBCO on top. For YBCO films in a thickness range of 12 nm to 48 nm, the superconducting transition temperature lies in the range 87.5 K - 89.5 K, with no discernible influence of the ferromagnet. However, critical currents in fields up to 8 T proved much lower for the YBCO / LCMO bilayers than for single layers of YBCO on STO, accompanied with lower vortex glass temperatures. A possible explanation is that the good lattice match of the pseudo-cubic lattice parameter of LCMO (0.386 nm) with the a-, and b-axes of YBCO (0.382 nm and 0.389 nm) leads to less twinning of the YBCO layer than when grown on STO (lattice parameter 0.391 nm).

II10.47
CONTROL OF THE IN-PLANE EPITAXY OF YBCO FILMS DEPOSITED ON (100) MgO FOR MICROWAVE APPLICATIONS. Kyoko Kawagishi, Kazunori Komori, Masao Fukutomi, Kazumasa Togano, National Research Inst for Metals, 1st Research Group, Ibaraki, JAPAN.

Recently, technological progress in the deposition and characterization of high-T_c superconducting (HTS) films has enabled the design and fabrication of various high-T_c microwave devices. For the HTS film deposition for microwave applications, MgO is widely used as substrate material because of its low dielectric constant, low cost and availability in large sizes. However, $YBa_2Cu_3O_y$ (YBCO) deposition on MgO often causes mixtures of 0° and 45° in-plane rotations, resulting in degradation of electrical properties, such as critical current density (J_c) and microwave surface resistance (R_s). In order to overcome this problem, we used yttria-stabilized zirconia (YSZ) and CeO_2 buffer layers between MgO and YBCO to control the in-plane epitaxy of YBCO films. Buffer layers and YBCO films were deposited on MgO (100) using off-axis rf magnetron sputtering and pulsed laser deposition. XRD (θ, ω and ϕ scans) results revealed an excellent in-plane epitaxy with no evidence of misoriented grains in a YBCO/CeO_2/YSZ/MgO structure. The dielectric rod resonator method was used for R_s measurements of HTS films obtained. Various other methods such as ac susceptibility, scanning electron microscopy, atomic force microscopy and inductively coupled plasma spectrometry have been used to analyze the film properties. An attempt was made to correlate these film properties with the measured Rs values. The possibility of new buffer layer structures will also be discussed.

II10.48
ELECTROSTATIC MODULATION OF SUPERCONDUCTIVITY IN EPITAXIAL $Pb(Zr_xTi_{1-x})O_3/GdBa_2Cu_3O_{7-y}$ HETERO-STRUCTURES. C.H. Ahn, S. Gariglio, P. Paruch, T. Tybell, L. Antognazza, J.-M. Triscone, DPMC University of Geneva, Geneva, SWITZERLAND.

Using the polarization field of the ferroelectric oxide $Pb(Zr_xTi_{1-x})O_3$ in epitaxial $Pb(Zr_xTi_{1-x})O_3/GdBa_2Cu_3O_{7-y}$ heterostructures, we have electrostatically modulated superconductivity in the high temperature superconductor $GdBa_2Cu_3O_{7-y}$, shifting the doping level without introducing chemical or structural disorder. For slightly underdoped samples, a change in the normal state resistivity of 50 percent and a uniform shift of the superconducting transition of 7 K were observed. In more underdoped samples, an insulating state was induced. This approach allows one to switch between superconducting and insulating behavior in a reversible and nonvolatile fashion. C.H. Ahn, S. Gariglio, P. Paruch, T. Tybell, L. Antognazza, J.-M. Triscone, Science 284, 1152 (1999).

II10.49
THE EFFECT OF SUBSTRATE ON THE GROWTH UP SUPERCONDUCTING THIN FILMS. Cláudio Luiz Carvalho, Universidade Estadual Paulista, Dept of Physics and Chemistry, Ilha Solteira, SP, BRAZIL; José Arana Varela, Un Estadual Paulista, Dept of Chemistry, Araraquara, SP, BRAZIL; Paulo N.L. Filho, Universidade Federal de São Carlos, SP, BRAZIL.

Differents techniques have been used to obtain thin superconducting films as molecular beam epitaxy, magnetron sputtering, etc, but all of them are expensive and the substrates play an important rule in the growth of the films. In this work, we propose to obtain large area films as cheap as possible with favorable orientation of the $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ system. Based on this aim we have used a dip-coating technique to deposite thin film on polycrystalline substrates like silver (Ag). A polymeric precursor solution obtained by Pechini method has been used to obtain the thin film after rheology characterization. Multiple layer were applied on substrate before to heat treated in a cilindrical furnace at 810°C during 1 hour in ambient atmosphere. X-ray diffraction pattern shown high oriented films deposited on Ag substrate and the presence of the 2212 and 2223 phases that are in agreement with electric and magnetic characterization. Both technique shown transition temperature around 85K and 110K that correspond to 2212 and 2223 phases, respectively.

II10.50
A STAGNATION-FLOW REACTOR FOR MOCVD OF YBCO WITH INTEGRATED SENSING AND CONTROL. D.G. Goodwin, H.A. Atwater, D.A. Boyd, M.A. Gallivan, R.M. Murray, A.B. Tripathi, Division of Engineering and Applied Science, California Institute of Technology, Pasadena, CA; R.J. Kee, L. Raja, Engineering Division, Colorado School of Mines, Golden, CO.

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

SESSION II11: Bi-, Tl, AND Hg-CONTAINING SUPERCONDUCTORS: PROCESSING AND PROPERTIES

Chair: Lawrence P. Cook
Friday Morning, December 3, 1999
Room 200 (H)

8:30 AM *II11.1
EVOLUTION OF PHASES AND MICROSTRUCTURE OF BPSCCO FILAMENTS DURING HEATING AND COOLING OF 2223-BPSCCO TAPES. Klaus Fischer, Torsten Fahr, Institut fuer Festkoerper- und Werkstofforschung, Dresden, GERMANY; Peter Majewski, Max-Planck-Institut fuer Metallforschung, Stuttgart, GERMANY.

In the recent past the high-temperature reactions of the precursor in BPSCCO/Ag tapes in air were intensively studied. We have investigated the precursor conversion in an atmosphere of a reduced oxygen content ($N_2/8\%O_2$). The investigations were performed in situ using high temperature XRD on tapes after removal of the Ag cladding from one side of the samples as well as ex situ by XRD and SEM/EDX measurements on tapes after annealing at different temperatures and subsequent rapid cooling. During heating up the Pb-rich phases such as $(Pb, Bi)_4(Sr,Ca)_5CuO_x$ could not longer be detected above $790 group$° C. This temperature is slightly lower than that published for the precursor conversion inside of BPSCCO/Ag tapes heated up in air but it is substantial lower as expected from phase diagram studies in the Bi-Pb-Sr-Ca-Cu-O system. It was found that the evolution of the degree of texture of the 2212 phase observed during the heating up process depends on the rolling parameters used for the preparation of the samples by flat rolling of round wires to tapes. The maximum texture degree (Lotgering factor F) of the 2212 as well as of the 2223 phase observed during the conversion of 2212 in to 2223 depends on the reaction temperature. Since the temperature dependence of F is nearly the same for both phases at least at temperatures above $820 group$° C, it is concluded that the texture of the growing 2223 phase is determined by that of the decomposing 2212 phase. Our assumption, that this texture correlation is caused by the heterogeneous nucleation of the 2223 phase at 2212 grain boundaries, could be supported by a kinetic analysis of the 2223 phase formation. Finally, we studied the phase composition and its influence on the critical current density of tapes cooled down with different cooling rates after the reaction annealing.

9:00 AM II11.2
LOCAL OXYGEN AND POINT DEFECTS PROBING IN HG-1201 HIGH-Tc SUPERCONDUCTORS. J.G. Correia, CERN, Div. EP, Geneva, SWITZERLAND; J.P. Araujo, Porto University, Dept of Physics, Porto, PORTUGAL; S.M. Loureiro, Princeton University, Dept of Chemics, Princeton, NJ; P. Toulemonde, S. Le Floch, P. Bordet, J.J. Capponi, CNRS, Laboratoire de Cristallographie, Grenoble, FRANCE; W. Troeger, B. Ctortecka, T. Butz, Leipzig University, Dept of Physics, Leipzig, GERMANY; H. Haas, Hahn-Meitner-Institut, Dept of Physics, Berlin, GERMANY; R. Gatt, Wisconsin University, Dept of Physics, Madison, WI; J.G. Marques, Nuclear and Technologic Institute, Dept of Physics, Sacavem, PORTUGAL; J.C.Soares, Lisbon University, Nuclear Physics Centre, Lisbon, PORTUGAL.

The family of mercury-based copper oxides, $Hg_1Ba_2Ca_{n-1}Cu_nO_{2n+2+\delta}$, crystallise with a tetragonal lattice and at 30 GPa a high critical temperature of 164 K can be achieved in the Hg-1223 (n=3) compound. These facts create the expectation that precise measurements of the structural properties would allow a better understanding of the charge transfer mechanisms that generate carriers in the superconducting CuO_2 planes. However, the Hg planes are particularly disordered and it is not understood how Hg interacts with point defects like the non-stoichiometric oxygen that regulates T_C, O_δ, and with impurities such as CO_3^{2-}. Many studies using crystallographic techniques as well more local studies such as EXAFS suggested the existence of local distortions in these compounds. So far, it is not known if such effects are linked to the superconductivity mechanisms or are simply due to crystal-chemical effects. In this work the Perturbed Angular Correlation (PAC) local technique was applied to measure the electric field gradients (EFG) at Hg sites in the Hg-1201 (n=1) high-T_C superconductor. High quality pellets were implanted at ISOLDE/CERN with very low doses of the ^{199m}Hg radioactive isotope. The PAC experiments were performed during annealing at 490 K, under Ar or O_2 flow, to change the O_δ doping

concentration. The results show that the EFG parameters are highly sensitive to the O_δ content, that is clearly distinguished from other point-like oxygen-defect complexes. First principle calculations (FLAPW) of the EFGs were performed in undoped and oxygen doped Hg1201 that show an excellent agreement with the experimental results. The local behaviour of Hg atoms in highly oxygen doped Hg1201 was further studied at low temperatures. Below 100 K the asymmetry parameter of the EFG becomes non-zero showing that the charge distribution near the O(2)-Hg-O(2) apical chain becomes non-axially symmetric at low temperature in these samples.

9:15 AM II11.3
CONTROLLING TEXTURE IN Tl-1223/Ag COMPOSITES.
Jo Moore, Tom Gladstone, Angus Wilkinson, Chris Grovenor, Dept. of Materials, University of Oxford, Oxford, UNITED KINGDOM.

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

9:30 AM II11.4
SYNTHESIS OF $Hg_xBa_2CuO_{4+\delta}$ UNDER CONTROLLED OXYGEN AND MERCURY PARTIAL PRESSURES. Emmanuel G. Van-Dyck, Wesleyan Univ, Dept of Physics, Middletown, CT; John L. Wagner, Univ of North Dakota, Dept of Physics, Grand Forks, ND.

We have investigated the Structure, defect concentration, and superconducting properties of $Hg_xBa_2CuO_{4+\delta}$ ($0.7 \leq x \leq 1.1$) by resistivity, A.C.Susceptibility, X-ray and neutron diffraction measurements. With the possibility of more than one defect in $Hg_xBa_2CuO_{4+\delta}$, the role of individual defects on the stability and superconducting properties is obscured by the variations and correlations of different defects. To isolate the effect of a single defect, other defect must be held constant. Numerous studies have succeeded in correlating T_c with the variable oxygen defect, δ, through low temperature annealings. However, little is known concerning the variability of additional defects, specifically ones associated with disorder on the Hg-site. In this work, we demonstrates variations in defects associated with Hg stoichiometry. As the disorder of the Hg-site increases, T_c is found to decrease. Attempts to minimize or eliminate this metal-ion dfect resulted in a decrease in stability of this compound. These results indicate that defect associated with Hg-stoichiometry is required to form and stabilize the compound with the effect of suppression in T_c.

9:45 AM II11.5
POROSITY AND CONNECTIVITY IN HIGH Jc Ag-CLAD (Bi,Pb)2Sr2Ca2Cu3Ox TAPES. J. Jiang, X.Y. Cai, D. Apodaca, L. Schwarzkopf, A. Polyanskii, D.M. Feldmann and D.C. Larbalestier, Applied Superconductivity Center, University of Wisconsin, Madison, WI; R.D. Parrella, Q. Li, M.W. Rupich, G.N. Riley Jr., American Superconductor Corporation, Westborough, MA; J.A. Parrell, K.R. Marken, S. Hong Oxford Superconducting Technology, Carteret, NJ.

The porosity and its effects on the connectivity of several series of critical current density Jc optimized Ag-clad multifilament (Bi,Pb) 2Sr2Ca2Cu3Ox (Bi-2223) tapes throughout important steps in their thermomechanical treatment was investigated by mass density measurement, microstructural observation, and extensive superconducting property characterization. The filament mass density, measured by digital image analysis of transverse cross sections of the silver and filament areas, could attain relative values normalized to full 2223 phase density of 85-90% before heat treatment (HT), declining to ~70% after the first HT, rising again to about 90% after intermediate rolling (IR), then declining again to values of 70-85% after final HT. Consistent with these large porosity and crack fractions, the limiting low temperature moment of samples measured by SQUID magnetometry was almost constant, irrespective of process step, indicating a high degree of porosity throughout the process. After the IR step, extensive cracks were observed by magneto optical imaging, and these cracks did not heal properly through subsequent HT, showing that the densification during the IR step is obtained at the expense of cracking. Our studies, made on a variety of high Jc tapes (Jc(0T,77K) values from ~20-60 kA/cm2), suggest that the performance of 2223 tapes could be significantly improved by raising the mass density if this can be done without simultaneously introducing detrimental cracks.

10:30 AM *II11.6
POLYCRYSTALLINE HTC MATERIALS WITH HIGH CURRENT CARRYING CAPACITY. L.J. Gauckler, D. Schneider, S. Koebel, ETH Zürich, Department Materials, Nonmetallic Materials, Zurich, SWITZERLAND.

Bi 2212 and Bi-2223 are the only HTC materials that show sufficient power carrying capacities for industrial applications. In Bi-2212/Ag conductors the growth of the HTS phase can be controlled more easily than in Bi-2223/Ag conductors since the phase development of Bi-2212 is well understood [1,2]. Bi-2212 tape conductors and bulk materials today are used in current limiters and current leads. Outstanding issues are the limitations of critical currents in polycrystals and the effects of surface barriers in single crystals. Oriented Bi-2212 thick films exhibit much higher critical currents than expected if transport is limited by j_c of the a-b plane and if it would be obstructed by large angle grain boundaries. Therefore macroscopic transport is thought to occur differently in Bi-2212 than in the Bi-2223 and Y-123 [3]. This had led to several models for the current transport in Bi-2212 polycrystals [4,5]. We give an overview on the processing/property relations of Bi-2212 thick film and bulk materials on Ag and MgO substrates and present results from Magnetic Force Microscope (MFM) measurements[6] of oriented Bi-2212 thick films and single crystals in the temperature range 40 70 K. The current paths in Bi-2212 differ from Y-123 and Bi-2223 by the absence of weak-links [7,8]. Magnetically measured j_c in Bi-2212 were comparable to direct transport currents in polycrystalline material. Therefor this material shows no weak link behavior. On the edges of Bi-2212 single crystals, our MFM measurements showed the current to be highly confined in a layer close to the grain boundaries parallel to the c-axis. In polycrstalline material their percolating network allows appreciable current densities (>3000 A/cm^2, 77 K, 0 T) even in non-textured bulk Bi-2212. The MFM method makes it possible to localize such type of current distributions with a precision almost on the 10-100 nm scale.

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11:00 AM *II11.7
FLUX-PINNING ENHANCEMENT OF SOLID-SOLUTION $Bi_2Sr_2CaCu_2O_{8+z}$ HIGH T_c SUPERCONDUCTING TAPES UTILIZING A-M-O (A=Bi-Sr-Ca-Cu, M=Al,Au) and CARBON NANOTUBE DEFECTS T. Haugan, W. Wong-Ng, L.P. Cook, H.J. Brown, L. Swartzendruber, National Institute of Standards and Technology, Materials Science and Engineering Laboratory, Gaithersburg, MD; D.T. Shaw, J. Ye, State Univ. of New York at Buffalo, Dept. of Electrical Engineering, Amherst, NY.

Efforts to improve the magnetic flux-pinning properties of solid-solution $Bi_{2+x}Sr_{2-x-y}Ca_{1+y}Cu_2O_{2+z}$/Ag (2212/Ag) tape conductors utilizing A-M-O (A=Bi-Sr-Ca-Cu, M=Al,Au) and carbon nanotube solid-state defects will be presented. A goal of this work was to produce a high density (~10^{11} cm^2, ~20 vol%) of nanometer sized defects chemically compatible with 2212 melt-growth methods. Studies focused on determining precise chemical reactions of nanosized additions (Al_2O_3, Au, carbon nanotube), how to vary the Bi:Sr:Ca:Cu precursor composition to compensate for reactions and maintain single-phase domain 2212 processing, and possible effects on partial-melt (air) or isothermal-melt (Ar/7%O2) growth process parameters. The effect of different melt-growth parameters (melting from ~850°C to 900°C) and precursor compositions on film

properties (phase assemblages, orientations, and defect sizes) was examined with XRD and SEM/EDS. Reactions of nanophase Al_2O_3 with Bi:Sr:Ca:Cu:O melts show promise for producing chemically stable solid-solution $Sr_{3-x}Ca_xAl_2O_6$ defects, where the Sr:Ca ratio is close to the ratio of the precursor powder. Carbon nanotubes had large effect on phase assemblages observed, and Au addition caused a high degree of 2212 misorientation in the film. In general, addition of Al_2O_3 and carbon nanotube defects improved flux-pinning in the range 20 - 60 K, however worsened the 2212 c-axis orientation and reduced transport Jc. The problem of nanophase defect coarsening will be presented. Flux-pinning and critical current density (J_c) was studied in 0 - 5 T magnetic fields from 5 - 60 K using magnetic and transport methods.

11:30 AM II11.8
ABNORMAL ANNEALING EFFECT ON (Cu,Tl)-1223 AND (Cu,Tl)-1234 AND ACHIEVING $T_c > 130K$. K. Tanaka, Y. Tanaka, A. Iyo, N. Terada, M. Tokumoto, H. Ihara, Electrotechnical Laboratory, Tsukuba, JAPAN and CREST of JST; T. Tsukamoto, Science Univ of Tokyo, Dept of Applied Physics, Tokyo, JAPAN; M. Ariyama, Science Univ of Tokyo, Dept of Physics, Noda, JAPAN; K. Tokiwa, S. Miyashita,T. Watanabe, Science Univ of Tokyo, Dept of Applied Electronics, Noda, JAPAN and CREST of JST.

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

11:45 AM II11.9
LARGE AREA Bi-2212 THICK FILMS ON CERAMIC SUBSTRATES. Stefan Köbel, D. Schneider, L. Fall, P. Sütterlin, L.J. Gauckler, Swiss Federal Institute of Technology Zürich, Dept of Materials, Zürich, SWITZERLAND.

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