# SYMPOSIUM E

## Materials for High-Temperature Superconductor Technologies

November 26 – 29, 2001

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* Invited paper

SESSION E1: NEW SUPERCONDUCTORS I - $MgB_2$
Chairs: David K. Christen and
Judith L. MacManus-Driscoll
Monday Morning, November 26, 2001
Room 200 (Hynes)

**9:00 AM *E1.1**
POTENTIAL ELECTRIC POWER APPLICATIONS FOR MAGNESIUM DIBORIDE. Paul Grant, EPRI, Palo Alto, CA.

The newly discovered superconductor, $MgB_2$, has significant potential for a number of electric power applications, even though its critical temperature, $T_C$, is "only" 39 K. In recent months, there has been rapid improvement in its critical state parameters, $J_C$ and H*, properties crucial to deployment in power devices, which now rival NbTi at 4.2 K, and equal or surpass many of the high temperature superconducting copper oxide perovskites in the 20 - 25 K range. Moreover, substantial progress has been achieved in realizing wire embodiments that appear economically scalable to commercial production. In this talk, we will review several opportunities to exploit these developments for transformer, motor and electric cable applications, and discuss a visionary power delivery system centered on an $MgB_2$-based dc cable cooled by gaseous or liquid hydrogen supplying both electrical and chemical energy to the end user.

**9:30 AM *E1.2**
IRON-CLAD $MgB_2$ SUPERCONDUCTOR WIRES. S. Jin, H. Mavoori, C. Bower and R.B. van Dover, Agere Systems/Lucent Technologies, Bell Labs., Murray Hill, NJ.

The recent discovery of the 39K superconductivity in magnesium diboride presents a new possibility for significant bulk applications, especially in view of the weak-link-free characteristics of the grain boundaries and the high critical currents observed.[1-3] Technically useful bulk superconductors need to be fabricated in metal-clad wire configuration and must have high transport critical current density. The fabrication of bulk, stoichiometric $MgB_2$ wires having an acceptable density, 39K superconductivity, and high critical currents is a challenge because of the difficulties associated with the volatility and reactivity of magnesium and the brittleness of $MgB_2$. Metal-clad processing of $MgB_2$ is a convenient, practical method of overcoming these problems. Recent work[4] demonstrated the successful fabrication of iron-clad $MgB_2$ superconductor wires exhibiting desirable characteristics, with the transport $J_c$ as high as 85,000 A/cm$^2$ at 4.2K. In this talk, further progress and understanding of the effect of processing parameters, composite wire structures, grain boundary characteristics, and critical current bahavior of the $MgB_2$ wires will be described. Various approaches of microstructural modifications to induce coherence-length-scale defects and higher density of flux-pinning sites will also be discussed. 1. J. Nagamatsu, et al., Nature 410, 63 (2001). 2. D.C. Larbalestier, et al., Nature 410, 186 (2001). 3. D.K. Finnemore, et al., Phys. Rev. Lett. 86, 2420(2001). 4. S. Jin, et al., Nature 411, 563 (2001).

**10:00 AM E1.3**
HALL EFFECT IN SUPERCONDUCTING $MgB_2$ FILMS AND RELATED MATERIALS. R. Jin, M. Paranthaman, B.C. Sales, H.Y. Zhai, H.M. Christen, D.K. Christen, D. Mandrus, Oak Ridge National Laboratory, Oak Ridge, TN.

We have investigated the temperature and magnetic field dependence of the Hall coefficient of two well-characterized superconducting $MgB_2$ films ($T_{c0}$=38.0 K) in both the normal and superconducting states. Our results show that the normal-state Hall coefficient $R_H$ is positive and increases with decreasing temperature, independent of the applied magnetic field. Below $T_c$(H), $R_H$ decreases rapidly with temperature and changes sign before it reaches zero. The position and magnitude at which $R_H$ shows a minimum depends on the applied field. Quantitative analysis of our data indicates that the Hall response of $MgB_2$ behaves very similarly to that of high-$T_c$ cuprates: $1/R_H \propto T$ and $\cot\theta_H \propto T^2$ in the normal state, and a sign reversal of $R_H$ in the mixed state. This suggests that the B-B layers in $MgB_2$, like the Cu-O planes in high-$T_c$ cuprates, play an important role in the electrical transport properties. Results obtained from related materials will also be presented.

**10:45 AM E1.4**
DIRECT OBSERVATION OF NM-SCALE Mg- AND B-OXIDE PHASES AT GRAIN BOUNDARIES IN $MgB_2$. R.F. Klie, J.C. Idrobo and N.D. Browning, Department of Physics, University of Illinois at Chicago, Chicago, IL; K.A. Regan, N.S. Rogado and R.J. Cava, Department of Chemistry and Princeton Materials Institute, Princeton University, Princeton, NJ.

The discovery of superconductivity in $MgB_2$, with a record breaking transition-temperature ($T_c = 39K$) for "conventional" superconductors, has sparked an enormous research effort to understand its structure-property relationships. One aspect of $MgB_2$ that has particular technological importance is that the grain boundaries in the polycrystalline materials studied so far do not appear to significantly decrease the overall critical current ($J_c$). This is in marked contrast to the cuprate high-$T_c$ superconductors, where high-angle grain boundaries universally act as weak links and dramatically reduce the inter-granular critical currents, and offers the promise of a vast number of new industrial applications. A critical first step in understanding the mechanism by which these boundaries can support a high critical current is a determination of the structure and composition of both the grains and the grain boundaries. Here we report a series of direct atomic resolution studies of the grains and the grain boundaries in a polycrystalline $MgB_2$ sample by the combination of Z-contrast imaging and electron energy loss spectroscopy in the UIC JEOL 2010F. Specifically we find that there is no oxygen within the bulk of the grains (within the few percent detection limits of our techniques) but significant oxygen enrichment at the grain boundaries. Furthermore, the boundaries are found to consist of two distinct boundary types: one containing $BO_x$ phases with a width of $<$4nm (i.e. smaller than the coherence length) and a second containing a $BO_x - MgO_y(B) - BO_z$ trilayer $\sim$10-15nm in width (i.e. larger than the coherence length). Such boundary features indicate that although $J_c$ is high overall, the structure-property relationships at grain boundaries in $MgB_2$ are a complex function of processing conditions, and that control of the oxygen content at grain boundaries will be essential for attaining optimal bulk critical currents.

**11:00 AM *E1.5**
SUPERCONDUCTIVITY IN $MgCNi_3$ - UNCONVENTIONAL OR ORDINARY? R.J. Cava, T. He, M.A. Hayward, M.K. Haas, K.A. Regan, Dept. of Chemistry, Princeton University, Princeton, NJ; Q. Huang, NIST Dept of Neutron Research, Gaithersburg, MD, and Dept. of Materials and Nuclear Engineering, University of Maryland, MD; A.P. Ramirez, Condensed Matter and Thermal Physics, Los Alamos Nat. Lab., Los Alamos, NM.

The discovery of superconductivity in $MgB_2$ has stimulated a great deal of research into characterization of its physical properties. Soon after that material was discovered, we discovered superconductivity at 8 K in the non-oxide perovskite $MgCNi_3$. The low superconducting transition temperature makes it inappropriate for consideration for real applications, and the relatively low $T_c$ suggests at first sight that is may not be a very interesting material. However, the very large proportion of Ni in the compound suggests that magnetic interactions may play a role in the superconductivity. Its structural and chemical relation to the superconducting quaternary nickel borocarbides is also of interest. Some very interesting proposals have been made by theorists concerning what may be going on in this material. Here we will report our latest results designed to probe the nature of the superconductivity in $MgCNi_3$ by a variety of chemical substitutions and microscopic characterization methods.

**11:30 AM E1.6**
MICROSTRUCTURE AND PHASE DEVELOPMENT IN $MgB_2$. D.J. Miller, D.G. Hinks, Materials Science Division, Argonne National Laboratory, Argonne, IL.

We have studied the microstructure and phase development in $MgB_2$ bulk ceramics. Samples were prepared by high-pressure annealing of dense boron pellets in the presence of Mg. The sequence of phase development was studied by controlling the time of annealing. In these samples, porous particle boundaries that limit conduction area and a network of amorphous grain boundaries are commonly observed. Amorphous grain boundaries have been reported in samples prepared by other means and an evaluation of the growth process suggests these regions act as a reservoir for impurities. The progression of phases follows that predicted by the phase diagram, but equilibrium throughout an entire pellet is difficult to achieve. These results will be presented together with possible alternative routes for processing. This work was supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under contract #W-31-109-ENG-38.

**11:45 AM E1.7**
ANISOTROPY GRAIN MORPHOLOGY AND CRYSTALLOGRAPHIC TEXTURE IN POLYCRYSTALLINE $MgB_2$ SUPERCONDUCTORS. X. Song[a], S.E. Babcock[a], J. Jiang[a], L.D. Cooley[a], X.Y. Cai[a], A. Gurevich[a], A.A. Polyanskii[a], S. Patnaik[a], C.B. Eom[a], E.E. Hellstrom[a] and D.C. Larbalestier[a], R. Cava[b], S.L. Bud'-ko[c], P.C. Canfield[c], and D.K. Finnemore[c]; [a]Applied Superconductivity Center & Department of Materials Science and Engineering University of Wisconsin, Madison, WI; [b]Department of Chemistry, Princeton University, Princeton, NJ; [c]Department of Physics and Astronomy, Iowa State University, Ames, IA.

The microstructure of electromagnetically characterized bulk-scale sintered $MgB_2$ pellet, W-cored-$MgB_2$ filament and $MgB_2$ thin film,

was studied using transmission electron imaging and diffraction techniques with a view toward understanding the effect of materials microstructure on the electromagnetic properties of different samples. Anisotropic thick plate shaped crystals with an aspect ratio of order three and large faces parallel to (0001) planes of the layered structure were clearly observed in all three forms. Grains of intermediate size ($\sim 0.1mm \times 0.3mm$) were found in the best electromagnetically connected regions of the sintered pellet. These grains were strongly facetted, but showed little evidence of crystallographic texture. A tendency for parallel alignment of the [0001] axes of the considerably larger grains ($\sim 0.25\mu m \times 1\mu m$) of the filament was observed near the W core, but qualitative degradation of this texture with distance from the core was apparent in dense $MgB_2$ filament. The very small grains ($\sim$10 nm) of the thin film possessed a rather well defined fiber texture with [0001] parallel to the film normal, but no preferred orientation in the plane of the film. The observed grain morphology anisotropy and the crystallography textures in each material were all consistent with the anisotropic superconducting properties of the textured $MgB_2$ film. Critical current densities were 1-2 orders of magnitude higher in the thin film than the filament and the bulk samples. In contrast to the cuprate-based high Tc superconductors, all morphological data is consistent with conclusion that grain boundaries pose little barrier to current flow in the superconducting state and may even be flux pinning centers.

SESSION E2: NEW SUPERCONDUCTORS II - $MgB_2$
Chairs: Paul M. Grant and Sungho Jin
Monday Afternoon, November 26, 2001
Room 200 (Hynes)

**1:30 PM *E2.1**
SUPERCONDUCTIVE PROPERTIES OF HIGH-$J_c$ $MgB_2$ COATINGS. D.K. Christen, C. Cantoni, J.R. Thompson, M. Paranthaman, H.R. Kerchner, H.M. Christen, H.-Y. Zhai, C.W. White, Oak Ridge National Laboratory, Oak Ridge, TN; D.J. Miller, Argonne National Laboratory, Argonne, IL.

We have fabricated 0.5 $\mu$m thick, fine-grained polycrystalline coatings of $MgB_2$ on various substrate surfaces. Comparative studies of the magnetic persistent currents and electrical transport properties reveal excellent agreement over a wide range of temperature and magnetic field. This result is contrary to similar comparisons on high-temperature cuprates, where disparities arise from the effects of large flux creep and the diverse electric field regimes probed by the two techniques. The $MgB_2$ films exhibit extremely sharp voltage-current relations away from the irreversibility line, in qualitative agreement with observed low rates of magnetic flux creep. A quantitative assessment, using the relation $\mathrm{d}\ln J/\mathrm{d}\ln t = 1/(1-n)$, implies more complex behavior, with the power-law exponent, $n$, of the response $E \propto J^n$, dependent on the dissipation regime. The as-grown films exhibit large critical current densities, although the high-field properties appear limited by the intra-grain flux pinning as evidenced by improvements after particle irradiations. Measured levels of the irreversibility line are approximately encompassed by the anticipated range of the $H_{c2}$ anisotropy in the polycrystalline materials, confirming the strong flux pinning. Potential suitability of $MgB_2$ coatings for conductor applications will be discussed. Research sponsored by the U.S. Department of Energy under contract DE-AC05-00OR22725 with the Oak Ridge National Laboratory, managed by UT-Battelle, LLC.

**2:00 PM *E2.2**
MATERIAL ISSUES AND CHALLENGES IN $MgB_2$ THIN FILMS FOR BOTH WIRE AND ELECTRONIC DEVICE APPLICATIONS. C.B. Eom, M.K. Lee, J.H. Choi, S.D. Bu, L.J. Belenky, L.D. Cooley, X.Y. Cai, A. Gurevich, J. Jiang, M.T. Naus, S. Patnaik, A.A. Polyanskii, M. Rikel, A.A. Squitieri, X. Song, S. E. Babcock, E.E. Hellstrom, D.C. Larbalestier, University of Wisconsin-Madison, Madison, WI.

The discovery of superconductivity at 39 K in magnesium diboride offers the possibility of a new class of low-cost, high-performance superconducting materials for magnets and electronic applications. Epitaxial $MgB_2$ thin film is an ideal form for both understanding superconductivity and device fabrications because it offers an atomic scale control of the synthesis and structure. We have grown fiber textured c-axis oriented magnesium diboride thin films by pulsed laser deposition from a stoichiometric target. Our thin films have markedly improved irreversibility field, upper critical field and critical current densities that depend on resistivity and process conditions. $MgB_2$ thin films can exhibit a much steeper temperature dependence of $r\mathrm{H}^*(\mathrm{T})$ than is observed in bulk materials, yielding $r\mathrm{H}^*$ (4.2 K) above 14 T. In addition, very high critical current densities at 4.2K, 1 MA$\Sigma$ cm$^{-2}$ at 1 T and $10^5$ A/cm$^2$ at 10 T, are possible. These data demonstrate that $MgB_2$ has credible potential for high-field superconducting applications. We have also grown magnesium boride superconducting thin films by co-sputtering from elemental targets. The surface morphology, microstrcuture and superconducting properties are very sensitive to the processing conditions and the composition. We will discuss material issues and challenges in $MgB_2$ for both magnet and electronic device applications. D.C. Larbalestier et al., Nature, 410, 186 (2001) C.B. Eom et al., Nature, 411, 558 (2001) We thank Robert Cava (Princeton University) and Darrell Schlom (Pennsylvania State University) for collaborations in this work.

**2:30 PM E2.3**
IN-SITU DEPOSITION OF SUPERCONDUCTING $MgB_2$ FILMS. N.A. Stelmashenko, V.N. Tsaneva, M. Kambara, N. Hari Babu, D.A. Cardwell, M.G. Blamire, IRC in Superconductivity, University of Cambridge, Cambridge, UNITED KINGDOM.

The discovery of superconductivity in $MgB_2$ has been followed by many papers reporting attractive thin film properties. In most cases these have involved the deposition of precursor films followed by in-situ or ex-situ post annealing in a Mg-rich atmosphere. Although simple device structures have been fabricated from such films, it is desirable for a number of reasons that a heterostructure device technology be developed. Heterostructure growth is likely to require in-situ growth, preferably without post-annealing. To achieve this, low oxygen and high Mg background pressures are required in the vicinity of the sample. By using a novel heater geometry and a high density, Mg-rich bulk sintered target we have been able to grow superconducting $MgB_2$ films from Mg-rich targets at temperatures below 400C. This paper reports the deposition method, optical emission spectroscopy of the ablation plume, and structural and electrical characterisation of the films.

**2:45 PM E2.4**
SUPERCONDUCTING AND MICROSTRUCTURAL PROPERTIES OF $MgB_2$/Mg NANO-COMPOSITES. Qiang Li, G.D. Gu, Y. Zhu, and M. Suenaga, Brookhaven National Laboratory, Upton, NY.

A study is presented of superconducting and microstructural properties of bulk $MgB_2$/Mg composites. Advanced TEM investigation revealed that the composites are very dense, consisting of nano-sized $MgB_2$ and Mg grains (20-100 nm). Both magnetization and transport measurements were performed for superconducting property characterizations. We found that the composite carried over 1 MA/cm$^2$ critical current density at 5 K and self-field. Based on the remnant magnetization measurement of bulk sample, the composites appeared to be free of any weak-link region. We shall discuss the relation between the transport properties of this superconducting material with its microstructures in detail.
*This work was supported by the U.S. Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under contract No. DE-AC02-98CH10886.

**3:30 PM *E2.5**
THE SYNTHESIS AND PERFORMANCE OF $MgB_2$. R.L. Meng, X.H. Chen, D. Pham, J. Cmaidalka, B. Lorenz, J.K. Meen, Y.Y. Sun, Y.Y. Xue and C.W. Chu[a], Dept of Physics and Texas Center for Superconductivity, Univ of Houston, Houston, TX. [a]Also at Hong Kong Univ of Science and Technology, HONG KONG.

The relatively high transition temperature, the small anisotropy, and the negligible grain boundary effects on the supercurrent flow of the newly discovered $MgB_2$ suggest that the compound may hold great promise for both large- and small-current applications. Unfortunately, the density, microstructure, electrical connectivity, and surface morphology of the $MgB_2$ samples, which define the performance of the superconducting devices, remain beyond control. We have examined systematically the influence of various synthesis conditions and identified the crucial parameters affecting the above properties. For instance, microstrain and lattice parameters have been found to be effective measures of the quality of the samples. These and other results, together with their effects on the transport and magnetic properties of $MgB_2$, are to be presented and discussed.

**4:00 PM E2.6**
GROWTH OF STRONGLY BIAXIALLY ALIGNED $MgB_2$ THIN FILMS ON SAPPHIRE BY POST-ANNEALING OF AMORPHOUS PRECURSORS. A. Berenov, Z. Lockman, X. Qi, Dept. of Materials, Imperial College, London, UNITED KINGDOM; Y. Bugoslavsky, L.F. Cohen, Dept of Physics, Imperial College, London, UNITED KINGDOM; M.-H. Jo, N.A. Stelmashenko, V.N. Tsaneva, M. Kambara, N. Hari Babu, D.A. Cardwell, M.G. Blamire, IRC in Superconductivity, University of Cambridge, Cambridge, UNITED KINGDOM; and J.L. MacManus-Driscoll, Imperial College, London, UNITED KINGDOM.

$MgB_2$ thin films were cold-grown on sapphire substrates by pulsed

laser deposition (PLD), followed by post-annealing in mixed, reducing gas, Mg-rich, Zr gettered, environments ($pO_2$ $\sim 10^{-24}$atm.) at 750°C and 950°C. The films had $T_c$s in the range 29K to 34K, $J_c$s (20K, H=0) in the range 3x10$^4$A.cm$^{-2}$ to 3x10$^5$A.cm$^{-2}$, and irreversibility fields H* at 20K of 4 T to 6.2 T. An inverse correlation was found between $T_c$ and H*. The films had grain sizes of $\sim$0.1-1$\mu$m and a strong biaxial alignment was observed in the 950°C annealed film. (111) oriented MgO was also observed. Mg coating of films during crystallisation appeared to improve film $T_c$.

**4:15 PM E2.7**
SYNTHESIS, CHARACTERIZATION AND AGEING OF $MgB_2$.
A. Serquis, Y.T. Zhu, J.Y. Coulter, D.E. Peterson, N.O. Moreno, P.G. Pagliuso and F.M. Mueller, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

Since the discovery of superconductivity in $MgB_2$ at 39 K, the highest Tc observed for a non-copper-oxide bulk superconductor, considerable progress has been made in the understanding of the fundamental properties of this material. However, different sample preparation methods lead to materials with different superconducting properties. It is interesting to note that the local composition (both the atomic ratio B:Mg and the effect of possible O contamination) remains a topic of debate. It has been found that Tc of the in-situ prepared films is much lower than in bulk $MgB_2$ samples and it was also reported that Tc changes significantly with different starting compositions of Mg:B in bulk materials. We study the influence of sample preparation and defects in the superconducting properties of $MgB_2$ samples to obtain materials which may be suitable for applications. We have prepared polycrystalline samples using a nominal ratio Mg:B=1:1 (Mg excess) Mg and Mg:B=1:2 (stoichiometric composition). Samples were characterized by SEM, XRD and the magnetization properties were examined in a SQUID magnetometer. We also make comparisons with commercial powder. The presence of Mg vacancies is determined by Rietveld analysis. Most of the samples are near single phase with small amounts of Mg impurities as determined by XRD and exhibit sharp superconducting transitions with Tc $\sim$39 K in zero field cooling measurements. Our most striking observation is that samples can undergo to a degradation with time when exposed to ambient conditions. Although the Tc does not change with time, the superconducting transition becomes broader and the Meissner fraction decreases. This aging effect is much smaller for the samples prepared with excess Mg. These results will be discussed in terms of the presence of different kind of defects, i.e. Mg vacancies or the presence of strain-inducing MgO inside the $MgB_2$ grains.

**4:30 PM E2.8**
THERMODYNAMICS AND THIN FILM DEPOSITION OF $MgB_2$ SUPERCONDUCTORS.
X.X. Xi, X.H. Zeng, A. Sukiasyan, Y.F. Hu, E. Wertz, Qi Li, Department of Physics, The Pennsylvania State University, University Park, PA; J. Lettieri, D.G. Schlom, C.O. Brubaker, Zi-Kui Liu, Department of MS&E, The Pennsylvania State University, University Park, PA; W. Tian, H.P. Sun, X.Q. Pan, Department of MS&E, The University of Michigan, Ann Arbor, MI.

The recent discovery of superconductivity in $MgB_2$ at 39 K has generated great interest in their high-current, high-field applications and in superconducting microelectronics. Thermodynamics studies with the calculation of phase diagrams (CALPHAD) modeling technique show that due to the high volatility of Mg, $MgB_2$ is thermodynamically stable only under fairly high Mg overpressures for likely growth temperatures. This provides helpful insights into appropriate processing conditions for $MgB_2$ thin films, including the identification of the pressure-temperature region for adsorption-controlled growth. The initial $MgB_2$ thin films were made by pulsed laser deposition followed by in situ annealing. The cross-sectional transmission electron microscopy reveals a nanocrystalline mixture of textured MgO and $MgB_2$ with very small grain sizes. A zero-resistance transition temperature of 34 K and a zero-field critical current density of 1.3 10$^6$ A/cm$^2$ were obtained. Superconductivity was observed also in in situ deposited films without annealing. The qualities of these films are limited by the thermodynamic stability conditions, which favor deposition techniques that can maintain a high flux of Mg. One such technique is the metalorganic chemical vapor deposition. We have set up a MOCVD system for the deposition of $MgB_2$ thin films, and the latest results using this technique will be reported.

**4:45 PM E2.9**
MICROWAVE MEASUREMENTS ON SUPERCONDUCTING $MgB_2$.
N. Hakim, P.V. Parimi, C. Kusko, S. Sridhar, Physics Department, Northeastern University, Boston, MA.

Measurements of the $10GHz$ surface resistance, $Rs$, and penetration depth, $\lambda(T)$, of dense wires, polycrystalline samples, and thin films of $MgB_2$ are presented. Substantial improvements in $Rs$ are observed upon improving the sample density and reducing porosity. The microwave absorption results are compared with measurements on low $Tc$ ($Nb$) and high $Tc$ ($YBa_2Cu_3O_{6.95}$) superconductors. The results are also compared with BCS calculations for an s-wave superconductor, which has been claimed to be the order parameter from other measurements. The results are discussed in the context of the nature of the superconducting state, intra- and inter-granular contributions, and the prospect for microwave applications. Work supported by Office of Naval Research.

## SESSION E3: HIGH TEMPERATURE SUPERCONDUCTORS I
Chairs: Terry G. Holesinger and Kamel Salama
Tuesday Morning, November 27, 2001
Room 200 (Hynes)

**8:30 AM *E3.1**
BOND-LENGTH FLUCTUATIONS IN THE COPPER-OXIDE SUPERCONDUCTORS.
John B. Goodenough, Texas Materials Institute, The University of Texas at Austin, Austin, TX.

The phase diagram of $La_{2-x}Sr_xCuO_4$ is interpreted. From the virial theorem, it is argued that the cross-over from localized to itinerant electronic behavior in the range $0 < x < 0.3$ is characterized by fluctuations between two equilibrium Cu-O bond lengths. Cooperative local fluctuations give rise to one-hole correlation bags of 5 to 6 copper centers on the underdoped side, to strong-correlation fluctuations in an itinerant-electron matrix on the overdoped side. Spinodal phase segregation between an antiferromagnetic, insulating parent phase and the superconductive phase occurs in the underdoped compositions, between the superconductive phase and the metallic overdoped phase on the other side of the phase diagram. Ordering of the fluctuations into a travelling bipolaronic charge-density/spin-density wave of composition $x \approx 1/6$ yields heavy fermions of symmetry ($x^2 - y^2$) coexisting with light electrons; the high-temperature superconductive pairs are condensed out from the heavy fermions.

**9:00 AM *E3.2**
DEVELOPMENT OF BSCCO-BASED HIGH TEMPERATURE SUPERCONDUCTING TAPE PRODUCTS.
Alex Otto, Bill Carter, Ralph Mason, Ron Parrella, Eric Podtburg, Bart Riley, Jeff Schreiber, Yibing Huang, American Superconductor Corporation, Westboro, MA.

$Bi_2Sr_2Ca_2Cu_3O_{10-y}$ (BSCCO) multifilament superconducting tape engineering has provided key progress on attributes required for specific large-scale applications. Key attributes include mechanical robustness and strain tolerance, performance including transport current density and ac loss, and piece length supported by splicing technology. Long length production capability now yields an average engineering current density (77K, 1 $\mu$V/cm, self-field) exceeding 13.8 kA/cm$^2$, with equivalent critical currents in excess of 115 A, representing a very significant increase over the past 18 months. Short length development activity in support of long length production has demonstrated that significant BSCCO cost / performance improvements are possible and BSCCO's potential is still far from fully tapped. Key among these has been the experimental demonstration of critical current in excess of 170 A (77K, 1$\mu$V/cm, self-field) in tape samples with the same architecture and geometry as long length product. This paper will report on the properties and microstructures of experimental and production BSCCO tapes.

**9:30 AM E3.3**
IMPROVED CRITICAL CURRENT DENSITY OF Ag-CLAD Bi-2223 TAPES BY CONTROLLED PROCESSING.
J. Jiang, J.G. Chandler, X.Y. Cai, A.A. Polyanskii, E.E. Hellstrom and D.C. Larbalestier Applied Superconductivity Center, University of Wisconsin, Madison, WI.

The processing of long length Ag-clad (Bi,Pb)2Sr2Ca2Cu3Ox (Bi-2223) tapes is a complex, multi-parameter process which typically involves 2 heat treatments with one intermediate rolling. The key to improving the performance of the Bi-2223 tapes is to improve their electrical connectivity, which is compromised by weak flux pinning regions or by lower Tc intergrowths, by weak-linked grain boundaries, by insulating second phases, by cracks or by porosity, or by filament sausaging. A fundamental component of the problem of poor connectivity is the tendency of Bi-2223 tapes to swell as they convert from Bi-22212 to Bi-2223 and thus for the mass density to decline strongly. Our detailed density measurement on monocore Bi-2223 tapes are showing that the retrograde densification during the first heat treatment depends greatly on the precursor powder pretreatment and that some treatments produced rising density during the first heat treatment, thus resulting in higher critical current density in the final product. On the other hand, the effects of a post Bi-2223 formation anneal was systematically investigated in monocore Bi-2223 tapes by microstructural observation and superconducting property measurement. It was found that annealing at 790°C in 0.075 atm O2

partial pressure could obviously improve the critical current even though more Pb-rich phases were produced in this treatment.

**9:45 AM E3.4**
THE MICROSTRUCTURAL DEVELOPMENT OF THE COLONY STRUCTURE ALONG THE SILVER SHEATH IN Bi-2223 TAPES. T.G. Holesinger, J.F. Bingert, Los Alamos National Laboratory, Los Alamos, NM; R.D. Parrella, Y. Huang, and G.N. Riley Jr., American Superconductor, Westborough, MA.

Controlling phase development and optimizing grain-to-grain connectivity are the primary materials issues that affect the performance of Bi-2223 multifilamentary tapes. The current protocol for processing Bi-2223 involves the in situ formation of the primary phase from a suitable mixture of precursor phases. As such, the phase development during the first few minutes of heat treatment determines to a large extent the efficiency of primary phase development, competing secondary phase development, texture evolution, and grain-to-grain connectivity. Using analytical electron microscopy, detailed compositional and microstructural data were obtained from partially and fully processed Bi-2223 tapes. Defects in these tapes were either structural, compositional, or a combination of the two. They were introduced into the filament microstructure either intrinsically through the normal evolution of phases to form the Bi-2223 phase or extrinsically by various aspects of the thermo-mechanical processing. Relationships between the defect structures and transport properties will be discussed.

**10:30 AM *E3.5**
CRYSTAL GROWTH AND SUPERCONDUCTING PROPERTIES OF $Bi_2Sr_2Ca_2Cu_3O_y$ (Bi2223) SINGLE CRYSTALS. Keisuke Shimizu, Takehiko Okabe, Shigeru Horii, Kenji Otzschi, Jun-ichi Shimoyama and Kohji Kishio, Dept. of Superconductivity, Graduate School of Engineering, Univ. of Tokyo, Tokyo, JAPAN.

$Bi_2Sr_2Ca_2Cu_3O_y$ (Bi2223) is one of the most important materials for practical application of superconducting wires. However, its substantial properties have not been well understood mainly due to difficulty in the synthesis of large single crystals enough to perform precise studies. Therefore, we have attempted to grow Bi2223 single crystals by the floating zone method with an extremely slow growth rate of $\sim$0.05mm/h in order to clarify their basic properties, and successfully obtained large single crystals with the largest crystal size of approximately $3\times2\times0.1mm^3$. Through the X-ray diffraction analysis and DC susceptibility measurement, the grown crystals were confirmed to be the almost single phase of Bi2223 with very few intergrowth of Bi2212 phase. Their carrier doping state was systematically controlled in wide range from underdoping to overdoping by post annealing under various conditions, temperature and $P(O_2)$. $T_c$s of the crystals varied from 98K(underdoped) to 108K(overdoped) via 110K(optimally-doped). The second peak effect was observed in magnetization hysteresis loops taken around 30K under $H \parallel c$. The peak field, $H_pk$, monotonically increased from 300 to 1100Oe with an increase of carrier doping level. In the careful resistivity measurements, abrupt resistivity drop was observed under fields below $H_pk$, suggesting the first order phase transition of the flux line system. These characteristic behaviors are similar to those observed in the Bi2212 single crystals. Form above results, vortex phase diagram of Bi2223 single crystals with various carrier doping levels will be discussed including the irreversibility line and surface barrier effect from a viewpoint of electromagnetic anisotropy.

**11:00 AM E3.6**
EFFECT OF SECONDARY PHASES IN THE PRECURSOR POWDERS ON THE TRANSFORMATION TO THE (Bi,Pb)-2223 PHASE. Ravi Gundakaram, Sung-Chun Chang, Ru-Shi Liu, Department of Chemistry, National Taiwan University, Taipei, TAIWAN; Lee Woodall, Michael Gerards, Merck KGaA, Darmstadt, GERMANY.

The $(Bi,Pb)_2Sr_2Ca_2Cu_3O_y$ system (also known as (Bi,Pb)-2223) has shown promise for use in superconducting tapes and wires due to its high transition temperature and high critical current density. The tapes and wires are usually fabricated by the powder-in-tube method, after which they are subjected to thermal and mechanical treatments. Depending on the nature of the heat treatment, phase transformations occur in the powders and for practical applications, it is essential to understand the response of the powders to different processing conditions. In this work, we have synthesized a precursor powder of the nominal composition $Bi_{1.8}Pb_{0.33}Sr_{1.87}Ca_2Cu_3O_x$ by spray pyrolysis. The powder was then calcined under controlled conditions between 760 and 800° in an atmosphere between 0 and 21% $O_2$ for 2 to 24 hours with controlled heating and cooling, such that different amounts of Pb are incorporated into the majority phase. The resultant powders were then converted into the (Bi,Pb)-2223 phase with suitable heat treatments. Using powder X-ray diffraction and magnetization measurements, the volume fractions of the secondary phases in the precursors were estimated and the effect of the phases on the conversion to the (Bi,Pb)-2223 phase was studied. We show that the ratio of the intensities of the (020) and (115) reflections in the X-ray diffractograms can be used to tune the precursor powders for optimal conversion. While a lower volume fraction of the secondary phases such as CaO and CuO is desirable in the precursor powders, a higher volume fraction of $Ca_2PbO_4$ seems to help in the rapid conversion to the (Bi,Pb)-2223 phase.

**11:15 AM E3.7**
ELECTRICAL AND MAGNETIC STUDIES OF A COMMERCIAL NST Bi-2223 TAPE. Ph. Vanderbemden, J.F. Fagnard, V. Misson, D. Crate, A. Genon, D. Marguillier, A. Rulmont, R. Cloots and M. Ausloos, SUPRAS, Montefiore Electricity Institute, University of Liege, Liege, BELGIUM.

Several fine electrical and magnetic studies of samples cut from a commercial NST Bi-2223 tape were made. Results on AC and DC susceptibility, direct electrical transport and indirect measurements of the critical current density, crystallographic and microscopic characterizations are reported and analyzed for different samples and different geometries. The analysis emphasizes the role of weak links as well as field and current orientations. Several I,V contacts were soldered on various positions on the sample. Various resistivity measurements were performed for different sample and field geometries. The magnetic susceptibility was measured following the cycling method at fixed fields. The results are discussed in terms of the observed microstructure. A discussion of applications, involving field trapping and current transport, of such tapes is made based on the fundamental characteristics.

**11:30 AM E3.8**
AC SUSCEPTIBILITY OF THE SINTERED $Bi_{1.6}$ $Pb_{0.4}$ $Sr_2$ $Ca_{2-x}$ $M_X$ $Cu_3$ $O_\delta$ BULK HIGH-Tc SUPERCONDUCTORS. A.I. Malik, S.A. Halim, Z.A. Hassan, K. Khalid, Universiti Putra Malaysia, Dept of Physics, Serdang, MALAYSIA.

AC susceptibility measurements as a function of temperature, frequency and applied ac field amplitude were performed on two rectangular bar shaped samples of nominal composition of $Bi_{1.6}$ $Pb_{0.4}$ $Sr_2$ $Ca_{2-x}$ $M_X$ $Cu_3$ $O_\delta$ with M=$Pr$, $Gd$ and $x_{0.01}$, obtained by the usual solid state reaction route. The incorporation of the rare earth elements at low concentration stage results in an improve of grain connectivity for the sample doped with $Pr$. On the other end, the ac field amplitude and frequency dependence of the susceptibility curves discloses bulk pinning hystersis loss feature. The potential barrier height activation energy at the grain boundary extracted from the low temperature loss peaks reveal that a flux creep is governing the pinning mechanism inside the samples. Qualitative discussion in the framework of the critical state model is highlighted.

**11:45 AM E3.9**
REAL-TIME DEGRADATION STUDY OF $HgBa_2CaCu_2O_{6+\delta}$ SUPERCONDUCTING FILM USING SCANNING PROBE MICROSCOPY TECHNIQUES. R.S. Aga Jr., Y.Y. Xie and J.Z. Wu, Univ of Kansas, Dept of Physics and Astronomy, Lawrence, KS.

All high-temperature (high-$T_c$) superconductors are susceptible to moist environment. The mechanism behind this degradation process is not yet well understood or even described. We present our experimental results in observing the real time degradation process of $HgBa_2CaCu_2O_{6+\delta}$ (Hg-1212) high-$T_c$ superconducting film. We implemented several scanning probe microscopy (SPM) techniques to image different time evolving properties of the sample. In our experiment, we used a commercial atomic force microscope system and a home-built combined microwave/optical scanning near-field probe. The scanning probes capture data from the exposed samples atregular time interval. This enabled us to have a real time observation of surface topography, morphology and resistivity maps of the samples as they react with the humid environment (T$\sim$30°C, RH$\sim$60%). We use this information to describe the mechanism behind the degradation of Hg-1212. The duration of sample exposure was two days. In this time frame, a typical sample would acquire residual resistance of about 0.0008 $\Omega$·m at 100 K ($T_c$=120 K) and would exhibit a broader superconducting transition due mostly to the formation of impurities.

## SESSION E4: HIGH TEMPERATURE SUPERCONDUCTORS II
Chairs: Judy Z. Wu and Raghu N. Bhattacharya
Tuesday Afternoon, November 27, 2001
Room 200 (Hynes)

**1:30 PM *E4.1**
HIGH-TEMPERATURE AND HIGH-PRESSURE PROCESSING ROUTES OF Bi,Pb(2223)/Ag TAPES FOR HIGH CURRENT

APPLICATIONS. E. Giannini, R. Passerini, C. Beneduce, B. Seeber, E. Walker and R. Flüikiger, Department of Condensed Matter Physics (DPMC), University of Geneva, SWITZERLAND.

The necessity to improve the transport properties of PIT processed Bi,Pb(2223) Ag-sheathed tapes has driven us to search for alternative processing routes closer to equilibrium conditions. Equilibrium can be neared by favoring the formation of homogeneous and stable liquid and by preventing the stoichiometry to locally change during processing. A detailed investigation of the equilibrium between the Bi,Pb(2223) phase and the melt was carried out by in-situ high temperature neutron powder diffraction both on sintered bulk samples and Ag-sheathed tapes. Stability, decomposition and re-crystallization of Bi,Pb(2223) were systematically investigated, and the secondary phase behavior was studied. Bi,Pb(2223) melts incongruently to (Sr.Ca)2CuO3 and liquid, and no precipitation of other Bi-based phases was observed at high temperatures. Opportune conditions for a direct reformation of Bi,Pb(2223) from the melt were found and applied to the tape fabrication. High temperature zone melting (>1000°C) proved to be a suitable technique to promote the formation of dense Bi,Pb(2223) samples. High isostatic pressure revealed to be an effective remedy for preventing volatile elements from evaporating, thus keeping the stoichiometry constant during processing. High-Pressure thermodynamic studies have been conducted on the Bi-Pb-Sr-Ca-Cu-O-Ag system by means of high pressure differential thermal analysis. The consequences of using a high pressure were exploited for tape processing in a HIP furnace at 10 MPa: both mechanical and thermodynamic positive effects were observed. High temperature crystallization of Bi,Pb(2223) and high isostatic pressure can be combined to succeed in processing Ag-sheathed Bi,Pb(2223) tapes with higher density, improved grain connectivity, and thus better transport properties.

**2:00 PM *E4.2**
IMPROVING CURRENT-CARRYING CAPABILITY OF Hg-BASED SUPERCONDUCTING FILMS. Judy Wu, Y.Y. Xie, Z.W. Xing, Univ. of Kansas, Dept. of Physics and Astronomy, Lawrence, KS; T. Aytug, D.K. Christen, D.T. Verebelyi, M. Paranthaman, A. Goyal, Oak Ridge National Laboratory, Oak Ridge, TN; G. Danial, D. Larbalastier, Department of Physics, University of Wisconsin, Madison, WI.

High $I_c$s (critical currents) and high $J_c$s (critical current densities) are desirable for superconducting power related applications. This motivated us to conduct an experimental study to identify the current-limiting factors and to explore current-improving methods for the highest-$T_c$ Hg-based superconducting (Hg-HTS) films and coated conductors. Bi-crystal substrates were employed to study the effect of grain boundary on $J_c$s in Hg-HTS films, as well as their "precursor" Tl-HTS films, using both electrical transport and magnetic methods. Oxygen overdoping was applied to greatly improve grain-boundary connectivity and overall $J_c$s and $I_c$s. Various artificial defects were introduced into the films to improve the $J_c$s in an applied magnetic field. These included the linear defects generated with high-energy ion beam irradiation, the strain-induced linear growth defects on miscut substrates, and point defects via chemical doping and substitution. Significant enhancement of the in-field Jcs and Hirrs was obtained. To achieve high $I_c$s, Hg-HTS films of thickness as large as 3 microns have been fabricated. $I_c$s ~50 A/micron thickness for a cm wide film has been obtained at 100 K and high $I_c$s are expected when the processing conditions are optimized.

**2:30 PM E4.3**
CRITICAL CURRENT PROPERTIES OF Hg(Re)1223 SINGLE CRYSTALS WITH VARIOUS OXYGEN CONTENT. S. Ueda, J. Shimoyama, T. Sato, S. Horii, K. Otzschi and K. Kishio, Dept. of Superconductivity, Univ. of Tokyo, Tokyo, JAPAN.

Basic physical properties of Hg-based superconductors have not been well understood because of the difficulty in the synthesis of their large single crystals with high quality, while they show record-high $T_c$ fs. In the present study, we have successfully grown high quality single crystals of Hg(Re)1223 by the flux method using $BaZrO_3$ crucible, which is stable against molten Ba-Cu-O, and addition of $BaF_2$. The maximum size of the obtained crystal was $1\times1.1\times0.1 mm^3$ in dimension. X-ray diffraction analysis revealed that grown crystals are of HgRe1223 single phase with $c_0 = 15.7\AA$. In the TEM images, intergrowth of Hg(Re)1212 layer was found, however, it was less than 2% in each crystal. From the result of the temperature dependence of in-plane resistivity measurement, $T_c$(onset) was determined to be 133K and conduction of normal state exhibited an underdoped-like behavior. In the magnetic hysteresis curves for post-annealed Hg(Re)1223 single crystals under $H \parallel c$, large second peak effect was found with steep increase of magnetization at approximately 2-3kOe independent of temperatures. We deduce that the vortex state changes from 3D lattice to 2D glass at approximately 2-3kOe. On the other hand, the peak field monotonically increased with decreasing temperature, which suggests the existence of effective field-induced pinning sites in the present system. Irreversibility lines of the Hg(Re)1223 single crystals locate at higher fields than that of Re-free Hg1223. This strongly indicates that electromagnetic anisotropy is decreased by Re substitution as was predicted in our previous study on the polycrystalline Hg(Re)1223. Relationship between electromagnetic anisotropy and pinning performance in the present system will be discussed including the results on the variation of critical current properties with oxygen content.

**2:45 PM E4.4**
DIFFUSION DYNAMICS OF CATIONS AND ANIONS IN FABRICATION OF $HgBa_2CaCu_2O_{6+\delta}$ SUPERCONDUCTING FILMS USING CATION-EXCHANGE PROCESS. Y.Y. Xie, J.Z. Wu, University of Kansas, Department of Physics and Astronomy, Lawrence, KS; T. Aytug, D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

We report the experimental results on the study of the diffusion dynamics of Hg (Tl) cations and oxygen anions during conversions from $Tl_2Ba_2CaCu_2O_8$ (Tl-2212) precursor films to $HgBa_2CaCu_2O_{6+\delta}$ (Hg-1212) films. Different Hg-vapor annealing times and post oxygen annealing times were used to convert epitaxial Tl-2212 superconducting films into Hg-1212 films and to optimize the oxygen content in the films. The Hg-vapor annealing was carried out at ~700°C from several to 720 minutes, and oxygen annealing at ~300°C from 2 to 12 hours. Two time constants were found. A nearly complete Tl to Hg cation exchange can be obtained in a period as short as 45 min., an order of magnitude shorter than what has been used. The diffusion of anions (oxygen), on the other hand, takes several hours or longer at ~300°C in flowing oxygen to reach the optimal level. Hg-1212 films prepared using a shortened Hg-vapor annealing time (90-180 minutes) and a extended oxygen annealing time (>4 hours) have zero resistance transition temperature ($T_{c0}$) close to 120 K and critical current density ($J_c$) close to 1 $MA/cm^2$ at 100 K in self magnetic field. These $T_{c0}$ and $J_c$ values fall among those reported for high-quality Hg-1212 epitaxial films, which motivates us to employ these modified experimental conditions for development of Hg-1212 coated conductors since a smaller heat budget could minimize the interfacial reaction between the substrate and the film. The results on Hg-1212 coated conductor will also be discussed.

**3:30 PM *E4.5**
SUPERCONDUCTING THALLIUM OXIDE AND MERCURY OXIDE FILMS. Raghu N. Bhattacharya, Richard D. Blaugher, National Renewable Energy Laboratory, Golden, CO; Zhongwen Xing, Judy Z. Wu, Department of Physics and Astronomy, University of Kansas, Lawrence, KS; Jun Chen, Z.F. Ren, Boston College, Department of Physics, Boston, MA.

The primary technical challenge that must be satisfied to permit high-temperature superconductor (HTS) wire or tape in superconducting magnets or power-related applications is the successful demonstration of a low-cost, high-field, high-current-carrying wire or tape with acceptable mechanical properties. The thallium-based and mercury-based superconducting oxides are excellent candidates because of their high transition temperature reaching to 130 K and unique features in their growth morphology. In general, the electrodeposition of superconductor tapes for conductor applications has considerable practical potential, particularly in the fabrication of large non-planar devices. Electrodeposition is a potentially low-cost, non-vacuum process that has the ability to easily deposit superconductor precursor films or tapes at thickness up to 15 micron. Electrodeposited films of all the oxide superconductors have been demonstrated, with the Tl system emerging as most promising. Previously we reported on a two-layer electrodeposition process of thallium oxide superconductors that showed transport critical current density above $10^6$ $A/cm^2$ at 77 K in zero field on LAO substrates. We were not very successful of forming the 1223-TBSBCCO phase on Ag, Ag/Pd (10% Pd), and also on textured Ni. The Tl-2212 or Tl-1212 phase mostly formed on these metal substrates. The Tl-2212 or 1212 phases lack good magnetic-field-dependent properties. A recent study by Wu et al. [J.Z. Wu, S.L. Yan, and Y.Y. Xie, Appl. Phys. Lett., 74, 1469 (1999)] showed that cation exchange of Tl by Hg took place for both Tl-2212 and Tl-1212 phases. In this process, Hg replaced Tl by reacting a Tl oxide superconductor ($TlBa_2CaCu_2O_x$) in the presence of Hg vapor. The quality of the sample made in the cation-exchange process is superior to Hg-1212 synthesized directly by the conventional sealed-tube annealing process. Cation-exchanged Hg-1212 showed critical current densities nearly an order of magnitude higher than the best value reported on conventionally processed Hg-1212 thin films. Wu et. al used the sputtered deposited Tl-oxide films. We attempted to convert the thick electrodeposited Tl-oxide films into Hg-1212 by the cation-exchange process. In this meeting, we report our effort to convert an electrodeposited thick Tl-oxide film to Hg-oxide by the cation-exchange process.

**4:00 PM E4.6**
ATTEMPT OF FLUX-PINNING ENHANCEMENT BY WELL-ALIGNED CARBON NANOTUBES IN THALLIUM-BASED HIGH TC SUPERCONDUCTING THIN FILMS. D.Z. Wang, Z.P. Huang, S.X. Yang, J.Y. Lao, H.I. Ha, J.I. Oh, J. Moser, J.G. Wen, M. Naughton, Z.F. Ren, Boston College, Dept of Physics, Chestnut Hill, MA.

One way to improve Jc in magnetic field is to create flux-pinning centers. Random MgO nanorods and carbon nanotubes embedded in bulk superconductors have been reported to enhance flux-pinning, though the enhancement is not much. Recently we attempted to use well-aligned carbon nanotubes as flux-pinning centers in thallium-based thin films to enhance Jc. Synthesis of thallium-based thin films with well-aligned carbon nanotubes has been achieved successfully. Primary results are very promising. The combination of different size and density of well-aligned carbon nanotubes in different superconductor thin films are being investigated. Superconducting properties of these superconductor thin films with aligned carbon nanotubes will be reported.

**4:15 PM E4.7**
METASTABLE SUPERSTRUCTURES IN $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_{10-\delta}$ SUPERCONDUCTOR BASED ON TEM OBSERVATION AT ROOM TEMPERATURE. Li Yang, J.M. Vieira, Aveiro Univ, Dept of Ceramic and Glass Engineering, Aveiro, PORTUGAL; Kaibin Tang, Guien Zhou, Structure Research Lab, Univ of Science and Technology of China, Hefei, PR CHINA.

Three types of metastable modulations in $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_{10-\delta}$ ($T_c$=30K) are observed by electron diffraction at room temperature and reported in this paper. The modulations are sensitive to the irradiation of the electron beam and the sample storage time. After several minutes' exposure to the electron beam, type 3 modulation with the period $\tau_3$=1.360nm, orientated in the $[110]^*$ direction occurs and coexists with type 1 with $\tau_1$=0.605nm orientated in the $[310]^*$ direction The intensity of the type 2 modulation, with $\tau_2$=0.544nm orientated along the $[110]^*$ direction, is enhanced when it coexists with type 1 and/or type 3 modulation. However, when the sample is observed after 6 months storage, only type 2 modulation appears, and its intensity varies from one grain to another. It should be noted that no components of modulation along $[001]^*$ have been detected, and no differences in composition have been detected within the accuracy of the x-ray dispersive spectroscope in all the cases. Having the tetragonal symmetry (14/mmm) with a=0.384nm, c=2.864nm, the structure of $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_{10-\delta}$ resembles that of $YBa_2Cu_3O_{7-\delta}$ by inserting a fluorite type $Gd_{1.4}Ce_{0.6}$ layer instead of the Y layer and Ru ions residing in the Cu(1) site. $CuO_2$ layer and $RuO_2$ layer coexist as structural constituents in one unit cell. It has been proved that, in this compound, superconductivity is confined to the $CuO_2$ layer while magnetism is due to the $RuO_2$ layer. The metastable modulations display the interaction between the two layers from the structural point of view. Superconductivity and magnetism may exist simultaneously in unconventional superconductor $RuSr_2Gd_{1.4}Ce_{0.6}Cu_2O_{10-\delta}$, and they are decoupled from each other. Li Yang gratefully acknowledges the support from Research Contract PRAXIS/P/CTM/13142/98.

**4:30 PM E4.8**
TRANSPORT PROPERTIES OF $Hg(Re)Ba_2Ca_2Cu_3O_y$ SINGLE CRYSTALS. Shigeru Horii, Shinya Ueda, Keisuke Kameno, Kenji Otzschi, Jun-ichi Shimoyama, Kohji Kishio, Univ. of Tokyo, Dept. of Superconductivity, Tokyo, JAPAN.

$HgBa_2Ca_2Cu_3O_y$ (Hg1223) Phase shows the highest $T_c$ of approximately 135K under ambient pressure among all superconductors, while this chemical stability and flux pinning performance are poor. We have revealed these disadvantages became clear by substitution of Re for Hg from systematical measurements of Hg(Re)1223 polycrystalline samples. Further, recently, several groups reported growth of Hg(Re)1223 single crystals. However, using single crystals, fundamental physical properties of this phase have not understood yet. In this study, we have successfully grown high qualitiy Hg(Re)1223 single crystals in a $BaZrO_3$ crucible which is inactive to the flux liquid, and checked transport properties of the crystals with several carrier-doped conditions. For as-grown crystals, we observed superconductivity of $T_{c,zero}$=131K and metallic behavior in a whole temperature range below room temperature from inplane resistivity measurements. On the other hand, a localized behavior was observed from out-of-plane resistivity. Further, a sharp superconducting transion at 131K was also picked up, suggesting that intergrowth of 1212 and 1201 structures are free and this behavior conicides with a result of TEM observation. In this present study, we show inplane and out-of-plane resistivities, magnetoresistance and Hall effect for crystals with several carrier-doping conditions, and systematically discuss changes of electromagnetic anisotropy by Re-doping in Hg-system.

SESSION E5: POSTER SESSION
Chairs: Enrico Giannini and Chang-Beom Eom
Tuesday Evening, November 27, 2001
8:00 PM
Exhibition Hall D (Hynes)

**E5.1**
EFFECTS OF DOPING ON THE SUPERCONDUCTING PROPERTIES OF $MgB_2$. S.E. Lofland, Rowan Univ, Dept of Physics, Glassboro, NJ and Univ of Maryland, Dept of Physics, College Park, MD; M. Botsford, M. Wilhelm, and K.V. Ramanujachary, Rowan Univ, Dept. of Chemistry, Glassboro, NJ; S.D. Tyagi, Dept of Physics, Drexel Univ, Philadelphia, PA.

We have a completed a systematic study of the effects of doping $Mg_{1-x}A_xB_2$ (A = Na or Ca, $x \leq 0.4$) by microwave and ac susceptibility techniques. While Na substitutes for Mg, Ca substitution is difficult to realize. This is due to the fact that $CaB_6$ readily forms and is extremely stable. This is observed in x-rays and manifests itself in that the ac susceptibility and the microwave studies show no shift in $T_c$, only a drop in superconducting fraction. On the other hand, although Na also shows no change in $T_c$ it displays no change in the Meissner fraction for $x \leq 0.3$, only the superconducting transition is somewhat broadened. However, for $x > 0.3$, the superconductivity rapidly disappears. We discuss the effects of weak links on the microwave results in light of these observations.

**E5.2**
HIGH-TEMPERATURE SUPERCONDUCTIVITY IN GRAPHITE-SULFUR COMPOSITES: THEORETICAL ANALYSIS. Douglas S. Galvao, Bernardo Laks, Robson R. da Silva, Jose H.S. Torres, Yakov Kopelevich, Applied Physics Department, UNICAMP, Campinas, BRAZIL.

We address recently reported superconductivity in graphite-sulfur composites occurring below the critical temperature of $\sim$35 K. We analyze the electronic structure changes associated with the presence of sulfur atoms in 2D graphite layers. We have considered ordered and disordered sulfur atom distributions in various configurations. The density of states corresponding to these structures are calculated using the negative factor counting (NFC) coupled to a tight-binding hamiltonian. The NFC technique allows us to obtain the eigenvalues of very large matrices without direct diagonalization. This methodology has been proven to be very effective in the study of electronic structure of disordered organic materials. We have investigated possible p-doping and percolation effects due to presence of sulfur atoms. The calculated electronic changes are addressed in terms of the experimentally observed high temperature superconductivity.

**E5.3**
SYNTHESIS AND OPTICAL PROPERTIES OF MAGNESIUM DIBORIDE FILMS. H.M. Christen, H.Y. Zhai, M. Paranthaman, D.K. Christen, C. Cantoni, B.C. Sales, and D.H. Lowndes, Oak Ridge National Laboratory, Oak Ridge, TN; R.A. Kaindl, M.A. Carnahan, and D.S. Chemla, Lawrence Berkeley National Laboratory, Berkeley, CA.

Advances in our understanding of the recently discovered superconductor $MgB_2$ with $T_c \approx$39K hinge critically on the availability of high-quality samples. Despite the considerable effort that has been devoted to the growth of thin films of $MgB_2$, we still lack a complete understanding of the nucleation and growth mechanisms, and challenging issues remain related to magnesium volatility and reactivity with oxygen. Samples obtained by an ex-situ reaction of e-beam evaporated boron films with magnesium have been used to determine the far-infrared optical properties. These measurements indicate that a simple BCS picture of superconductivity is in fact inappropriate for $MgB_2$, and in particular reveal a superconducting gap $2\Delta$ about one-half of that expected from BCS theory. Further advances in the growth and processing of $MgB_2$ both by ex-situ (based on e-beam evaporation of boron) and in-situ approaches (based on pulsed laser deposition) will be discussed in detail. Research sponsored by the US Department of Energy under contract DE-AC05-00OR22735 with the Oak Ridge National Laboratory, managed by UT-Battelle, LLC, and by the DOE Office of Energy Efficiency and Renewable Energy, Office of Power Technologies - Superconductivity Program.

**E5.4**
MICROWAVE ELECTROMAGNETIC RESPONSE OF HTS AND $MgB_2$. P.V. Parimi, N. Hakim and S. Sridhar, Physics Department, Northeastern University, Boston, MA.

Multiple dielectric signatures have been observed in the microwave response of undoped HTS compounds which persist in to doped compounds. These results confirm a substantial localized charge contribution to the microwave absorption of HTS, in addition, they

indicate that several electronic states such as charge stripes and oxygen ordering are coupled to the underlying lattice instabilities rather than by purely magnetic or electronic interactions. In $La_2CuO_{4+x}$ and $La_{5/3}Sr_{1/3}NiO_4$ new dielectric transitions are observed, at common temperatures 32K and 245K, that are signatures of local lattice octahedra instabilities present in these isostructural compounds. The results reveal new aspects of the phase diagram of the perovskite cuprates and nickelates. Preliminary results of microwave impedance on thin film $MgB_2$ reveal $R_s$ that is higher than that of $YBa_2Cu_3O_{7-\delta}$. Detailed microwave analysis of $MgB_2$ film in comparison with HTS will be presented.

**E5.5**
HEAVY-ION DAMAGE TO MAGNESIUM DIBORIDE FILMS: ELECTRICAL TRANSPORT-CURRENT CHARACTERIZATION. H.R. Kerchner, C. Cantoni, M. Paranthaman, D.K. Christen, H.M. Christen, Oak Ridge National Laboratory, Oak Ridge, TN; and D.J. Miller, Argonne National Laboratory, Argonne, IL.

The use of magnesium diboride in superconducting magnets, transmission lines, or other large-scale applications depends on the transport-current characteristics of this material in magnetic field, and how they compare to the properties of conventional and high-temperature superconductors. Thin films of boride grown on sapphire substrates by pulsed laser deposition were exposed to Mg vapor to produce 0.5-$\mu$m thick layers of the metallic compound $MgB_2$. Four-terminal measurements of their voltage-current relations, E(J), were carried out before and after exposure to $B_\phi$=1 T and higher doses of 1-Gev U ions. These doses lowered critical temperatures $T_c \approx$39 K less than 0.1 degree. The 1 T dose raised the self-field critical current density, $J_c$, nearly by a factor of 2.5; higher doses added little. Unexpectedly the irradiation slightly reduced critical current densities in the presence of applied magnetic field greater than 0.1 T. The radiation effects upon thermally assisted flux-flow (TAFF) resistivity revealed by the E(J) slopes measured at temperatures near $T_c$ will be discussed.
Research sponsored by the U.S. Department of Energy under contract DE-AC05-00OR22725 with the Oak Ridge National Laboratory, managed by UT-Battelle, LLC.

**E5.6**
FLUX PENETRATION AND DYNAMICS AT RF IN SUPERCONDUCTING $MgB_2$. E. Niyanchi, N. Hakim, P.V. Parimi, S. Sridhar, Physics Department, Northeastern University, Boston, MA.

Radio frequency penetration depth of polycrystal $MgB_2$ has been measured and from the data the lower critical field $H_{c1}$ has been obtained. The $H_{c1}(T)$ determined from the experiment has a BCS-like temperature dependence, but the theoretically estimated $H_{c1}$ does not show an exponential dependence expected for BCS like superconductor for T < $T_c$. The pinning force constant $\alpha(T)$ is found to be two orders of magnitude higher than that of polycrystal $YBa_2Cu_3O_{7-\delta}$ suggesting a marginal contribution from grain boundaries. The $\alpha(T)$ however is lower than that of single crystal $YBa_2Cu_3O_{7-\delta}$. Field dependent penetration depth reveals an intermediate field scale $H_{c2\alpha}$ that lies between the upper and lower critical fields. This has been discussed in terms of softening of flux lattice and unusual temperature dependence of condensate fraction.
Work supported by ONR and NSF.

**E5.7**
THE EFFECT OF SHEATH MATERIALS ON THE SUPERCONDUCTIVITY OF $MgB_2$. W. Zhu, J.R. Cave, IREQ, Hydro Québec, Varennes, Québec, CANADA.

Several sheath materials, such as a silver-palladium alloy, inconel, and tantalum, have been used to fabricate $MgB_2$ tapes. The tapes are made with PIT method and heat-treated at various conditions in argon under 1 atmosphere. The microstructure and phase compositions of the tapes are examined by electron microscope and X-ray diffractometer. Current-voltage relations are characterized by the four-probe method at the temperatures of liquid helium and above. Resistive and inductive critical temperatures are measured by SQUID magnetometry. It is found that tantalum sheath has no effect on the Tc of the material; but the silver-palladium sheath decreases the Tc and hence the Jc of the tape under certain processing conditions, while inconel could destroy the superconductivity completely under certain circumstances.

**E5.8**
LITHIUM INSERTION IN SUPERCONDUCTING MAGNESIUM DIBORIDE. Emma Lasheras, Ainhoa Morata-Orrantia, E. Morán and M. Á.Alario-Franco, Laboratorio de Química del estado Sólido, Facultad de Ciencias Químicas, Universidad Complutense, Madrid, SPAIN.

Ever since superconductivity was discovered in $MgB_2$ (1), work has been performed in different directions so as to modify and, eventually, improve its already remarkable superconducting properties (2). We have been working in the intercalation of several cations in $MgB_2$ via Soft Chemistry, by means of both an electrochemical cell and the usual chemical reactions in solution, e.g. $MgB_2$ + x $LiC_4H_9$ (n-BuLi) $\rightarrow$ $Li_x$ $MgB_2$ $C_8H_{18}$ By this procedure, we have observed that lithium can be intercalated in varying amounts that are reflected in the unit cell volume and also that some superstructure lines appear. Concomitantly with these structural modifications, we have also observed that $T_c$ seems to decrease. Work supported by CICYT, Program Mat-98- 0729 References 1. Nagamatsu, J., Nakagawa, N., Muranaka, T. and Akimitsu, J., Nature 410, 63 (2001) 2. Hinks, D.G. Claus, H. and Jorgensen, J.D., Nature 411, 457 (2001) 3. There is a very useful web page concerning this type of materials: http://www.lanl.gov

**E5.9**
SUPERCONDUCTIVITY, SUPERSTRUCTURE, AND STRUCTURE ANOMALIES IN $Mg_{1-x}Al_xB_2$. J.Q. Li, L. Li, F.M. Liu, C. Dong, J.Y. Xiang and Z.X. Zhao, National Laboratory for Superconductivity, Institute of Physics, Chinese Academy of Sciences, Beijing, PR CHINA.

The appearance of a superstructure is identified to play a key role for the modifications in both superconductivity and structure transitions in the $Mg_{1-x}Al_xB_2$ system. This superstructure occurs along the $c$-axis direction, and can be well interpreted by Al-layer ordering. The optimal composition of the superstructure phase is $MgAlB_4$, a superconductor with Tc =12K. Raman spectrum of $MgAlB_4$ gives rise to a sharp peak at around 941cm$^{-1}$. Brief diagrams illustrating the superconductivity and structural features of $Mg_{1-x}Al_xB_2$(0<x<1) materials are presented.

**E5.10**
SUPERCONDUCTIVITY AND CRYSTAL LATTICE OF $MgB_2$ UNDER HIGH PRESSURE. Jie Tang, A. Matsushita, K. Togano, National Institute for Materials Science, Tsukuba, JAPAN; Lu-Chang Qin, JST-ICORP Nanotubulite Project, Tsukuba, JAPAN; H. Kito, H. Ihara, Electrotechnical Laboratory, Tsukuba, JAPAN.

Magnesium diboride ($MgB_2$) shows the highest transition temperature Tc among the binary superconductors. We have investigated the superconductivity and crystal lattice of this material under high pressure up to P=10 GPa. Four-probe measurement of the transition temperature Tc shows a linear decrease at a rate of 1.03 K/GPa following the equation Tc=39.6-1.03P. The material synthesized under 3 GPa pressure shows a very narrow transition width and this transition width stayed unchanged even under high pressure. The crystal lattice shows an anistropic compressibility measured from in situ X-ray diffraction under hydrostatic pressure up to 10 GPa using a diamond-anvil cell and its bulk mudulus was deduced to be 172 GPa from our measurement. It is much more compressible along the c-direction than the a/b direction. This is attributed to a weaker inter-plane bonding along the c-axis in comparison with a stronger in-plane bonding perpendicular to the c-axis. The pressure effect on Tc is well explained within the framework of the classical BCS theory based on the electron-phonon coupling mechanism.

**E5.11**
THE NATURE OF SURFACE OXIDES ON MAGNESIUM DIBORIDE. Chandana Meegoda and Michael Trenary, University of Illinois at Chicago, Dept of Chemistry, Chicago, IL; Yu Paderno, Academy of Sciences of Ukraine, Institute for Problems of Materials Science, Kiev, UKRAINE.

The recent discovery of superconductivity at 39 K in magnesium diboride ($MgB_2$) by Akimitsu and co-workers has stimulated a great deal of interest in this compound. Most of the recently published papers have focused on synthesis methods, measurements of bulk properties, and theoretical aspects of superconductivity in $MgB_2$. Here we report on the nature of the surface oxides present on polycrystalline $MgB_2$ samples as determined with high resolution X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES). The samples used in our study have a critical temperature of 39 K as revealed by magnetic susceptibility measurements. X-ray diffraction (XRD) shows that the samples are highly crystalline with the observed reflections assigned to the $MgB_2$ (100), (101), (002) and (110) planes. The $MgB_2$ grains have a size of 40 nm as determined from the XRD line widths using the Williamson-Hall method. Auger and XPS spectra show that $Ar^+$ sputtering significantly reduces the amount of surface oxide. The Mg Auger peak at 34 eV is characteristic of covalently bonded magnesium compounds. The surface oxides were characterized with XPS by examining the B 1s, Mg 1s, Mg 2s and Mg 2p regions. A product of Lorentzian and Gaussian functions was used to fit the spectra. The B 1s peak shows two components, one of which is assigned to $B_2O_3$. The other is due to the B atoms of $MgB_2$. Some elemental B may also be

present. Consistent with the bonding in metal borides, the Mg 2s and 2p peaks were found to be about 2 eV higher than those of the pure element. However for the Mg 1s peak, only a slight shift was observed.

**E5.12**
STRUCTURAL CHARACTERIZATION OF POLYCRYSTALLINE PEROVSKITE $MgCNi_3$ SUPERCONDUCTOR. X. Song[a], S.E. Babcock[a], L.D. Cooley[a], J. Jiang[a], A. Polyanskii[a], and D.C. Larbalestier[a] T. He[b], M.A. Hayward[b] and R.J. Cava[b]. [a]Applied Superconductivity Center, University of Wisconsin, Madison, WI. [b]Department of Chemistry and Princeton Materials Institute, Princeton University, Princeton, NJ.

The newly discovered superconductor $MgCNi_3$ exhibits a systematic change of its flux-pinning behavior with temperature that suggest a transition from grain-boundary flux pinning just below $T_c$ to pinning by a nanometer-scale distribution of core pinning sites at low temperature. Scanning electron microscopy, transmission electron diffraction, diffraction contrast imaging, high-resolution electron microscopy were used to study the microstructure of a polycrystalline $MgCNi_3$ sample on different length scales. Scanning electron microscopy revealed about 10 mm diameter grains whose boundaries generally were lined with residual graphite. TEM revealed an about 200nm sub-grain structure separated by a network of dislocation walls (i.e. low angle grain boundaries). Selected area diffraction patterns recorded along various zone axes from single sub-grains showed that the perovskite $MgCNi_3$ was the primary phase. Additional weaker spots in the diffractions patterns indexed consistently as a second phase with a simple cubic unit cell with lattice parameter of 0.47 nm, but as yet unknown structure. This second phase also was observed as a minor second phase constituent in X-ray diffraction patterns from the material, indicating that it is not present only as a thin surface layer on the TEM samples. The diffraction patterns show that this second phase maintains a cube to cube orientation relationship with the primary $MgCNi_3$ phase in spite of the large (about 25%) mismatch of the lattice parameters. Moire patterns observed by high-resolution TEM suggest that the second phase is finely dispersed. The Moire patterns are generated throughout most of the sample and confirm the presence of overlapping phases, even though the volume fraction of the second phase by X-ray appears to be small. This fine scale nanostructure is qualitatively consistent with the strong flux pinning exhibited by the compound.

**E5.13**
OBSERVATION OF VORTICES AND COLUMNAR DEFECTS BY 1-MV LORENTZ MICROSCOPY I. Hiroto Kasai[a,e], Osamu Kamimura[a,e], Tsuyoshi Matsuda[a,e], Ken Harada[a,e], Akira Tonomura[a,e], Satoru Okayasu[d], Masato Sasase[d], Yuri Nakayama[b,e], Jun-ichi Shimoyama[b,e], Kohji Kishio[b,e], Tetsuro Hanaguri[c,e] and Kouichi Kitazawa[c,e], [a]Advanced Research Laboratory, Hitachi Ltd., Hatoyama, Saitama, JAPAN; [b]Department of Applied Chemistry, University of Tokyo, Bunkyo-ku, Tokyo, JAPAN; [c]Department of Advanced Materials Science, School of Frontier Science, University of Tokyo, Bunkyo-ku, Tokyo, JAPAN; [d]Department of Material Science, Japan Atomic Energy Research Institute (JAERI), Tokai, Naka-gun, Ibaraki, JAPAN; [e]CREST, Japan Science and Technology Corporation (JST), Kawaguchi, Saitama, JAPAN.

An understanding of the dynamical behavior of individual vortices (magnetic flux lines) in a Type-II superconductor is important for developing practical applications of superconductivity, because the vortex flow generates heat and breaks down the superconducting state. There are no techniques, however, for observing the dynamics of vortices except for Lorentz microscopy using a field emission electron microscope [1]. Here we present the first observation of vortices using newly developed 1-MV field emission electron microscope [2] whose world highest brightness is 2 x $10^{10}$A/cm$^2$sr.
In case of vortices in high-temperature superconductor $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212), the observable specimen thickness with a 300-kV microscope is about 200 nm at maximum due to the limited penetration ability of electrons [3]. While 1-MeV electrons can be penetrate the 400-nm-thick specimen whose size is similar to the vortex diameter (twice the penetration depth of Bi-2212). Higher contrast and detail images of the vortices are able to be observed by the new microscope.
A single crystalline of Bi-2212 was grown by the standard floating-zone technique whose critical temperature $Tc$ was about 85 K [4]. The thin film was irradiated by 240-MeV $Au^{+15}$ ions with density of 5 x $10^6$ ions/cm$^2$ (corresponding to 1 G) whose irradiation direction was tilted by 70 degrees from the c-axis of Bi-2212 in order to obtain elongated size and enhanced contrast of the projected columns. Such a low dose ion irradiation was performed with a specially developed slit shutter system installed at the beam line of the Tandem accelerator of the Japan Atomic Energy Research Institute (JAERI). Then prepared thin film was set on a low-temperature stage of the electron microscope tilted by 30 degrees from electron beam. Finally, simultaneous observation of single vortex and columnar defect has been carried out successfully by Lorentz microscopy, and their interactions have also been investigated.
[1] K. Harada *et al.*, Nature **360** (1992) 51-53.
[2] T. Kawasaki *et al.*, Appl. Phys. Lett. **76** (2000) 1342.
[3] K. Harada *et al.*, Phys. Rev. Lett. **71** (1993) 3371.
[4] Y. Kotaka *et al.*, Physica **C235 – 240** (1994) 1529.

**E5.14**
OBSERVATION OF VORTICES AND COLUMNAR DEFECTS BY 1-MV LORENTZ MICROSCOPY II. Osamu Kamimura[a,e], Hiroto Kasai[a,e], Tsuyoshi Matsuda[a,e], Ken Harada[a,e], Akira Tonomura[a,e], Satoru Okayasu[b], Masato Sasase[b], Yuri Nakayama[c,e], Jun-ichi Shimoyama[c,e], Kohji Kishio[c,e], Tetsuro Hanaguri[d,e] and Kouichi Kitazawa[d,e], [a]Advanced Research Laboratory, Hitachi Ltd., Hatoyama, Saitama, JAPAN; [b]Department of Material Science, Japan Atomic Energy Research Institute (JAERI), Tokai, Naka-gun, Ibaraki, JAPAN; [c]Department of Applied Chemistry, University of Tokyo, Bunkyo-ku, Tokyo, JAPAN; [d]Department of Advanced Materials Science, School of Frontier Science, University of Tokyo, Bunkyo-ku, Tokyo, JAPAN; [e]CREST, Japan Science and Technology Corporation (JST), Kawaguchi, Saitama, JAPAN.

The individual vortices in $Bi_2Sr_2CaCu_2O_{8\delta}$ (Bi-2212) thin film with thickness of 400 nm and columnar defects produced by irradiation of 240-MeV $Au^{15+}$ ions were simultaneously observed by Lorentz microscopy using newly developed 1-MV field emission electron microscope [1]. To obtain clear images of columnar defects and their pinning effect for individual vortices [2], the thin films were irradiated to 70 degrees tilted to the specimen normal with extremely sparse dose (0.05 ions/$\mu$m$^2$).
There are two kinds of vortex images in a Lorentz micrograph; one is globular image which is ordinary vortex image in our many experiments, another is elongated image with lower contrast. Numerical simulation [3] reveals that the globular contrast corresponds to vortex penetrating the film perpendicular to the film plane and the elongated contrast corresponds to vortex trapped along tilted column. It means that we are successfully obtained the information of the vortices inside superconductors.
Furthermore, we found that the elongated images change their contrast depending on temperature. From their images and their dynamical behavior, we considered of vortex features at the columnar defects as follows; (1) Above 19 K to the critical temperature $T_c$ (elongated contrast), the vortices were trapped along the tilted columnar defects.
(2) From 12 K to 19 K (from elongated contrast to globular one), the vortices change their direction along the column to perpendicular to the film plane.
(3) Lower than 12K (globular contrast), the vortices was stand up perpendicular to the film plane.
These phenomena are considered that strong, high density and small pinning sites exist and become effective at lower temperature than 12 K. Then columnar defects seem to loose their pinning force and vortices at the columnar defects stand up perpendicularly. Precise investigation of the pinning mechanism is significant for the practical applications of the high-temperature superconductor.
[1] T. Kawasaki *et al.*, Appl. Phys. Lett. **76** (2000) 1342.
[2] K. Harada *et al.*, Phys. Rev. B **53** (1996) 9400.
[3] S. Fanesi *et al.*, Phys. Rev. B **59** (1999) 1426.

**E5.15**
THE RED-OX DECOMPOSITION OF THE SOLID SOLUTION BASED ON Bi-2212 PHASE. A.V. Knotko, A.G. Veresov, M.N. Pulkin, A.V. Garshev, V.I. Putlayev, Dept of Chemistry, Moscow State Univ, Moscow, RUSSIA.

To improve $j_c$ of superconducting materials is quite important from the standpoint of their technical application. Some methods to solve this problem exists, the most promising one consists in creation of nanoscale defects in a superconducting matrix acting as pinning centers with the sizes close to the coherence length of the superconductor. The decomposition of a solid solution during the internal oxidation can give a key to generation of effective pinning centers in cuprate superconductors. Such a technique uses the fact of faster oxygen diffusion compared to the cationic one in Bi-2212. The oxygen content and microstructure of $Bi_{2.1-x}Pb_xSr_2CaCu_2O_{8+d}$, $Bi_{2.1-x}Pb_xSr_2Ca_{1-y}Y_yCu_2O_{8+d}$ and $Bi_2Sr_{2-x}Ca_{1-y}Pr_{xy}Cu_2O_{8+d}$ solid solutions were studied with XRD, TG and TEM. It was found that the oxidation of the Pb - contained solutions at temperature below 700°C in air consisted of two distinct stages. The first swift stage was attributed to the diffusion of oxygen and the second sluggish one - to a cationic redistribution followed by precipitation of secondary phases. The spinodal-type microstructure and noticeable increasing of $j_c$ were observed for the solutions passed through the early stage of the oxidation. Significant deceleration of the first oxidation stage without any changes in the second stage was observed in the case of $Bi_{2.1-x}Pb_xSr_2Ca_{1-y}Y_yCu_2O_{8+d}$ solid solutions with increasing Y-content. Even stronger deceleration was found during the

oxidation of $Bi_2Sr_{2-x}Ca_{1-y}Pr_{x+y}Cu_2O_{8+d}$ solid solutions, the precipitation of secondary phases in the bulk was not detected in this case by XRD. However, TEM investigation showed the presence of round precipitates with sizes of about 6 nm. The relatively fast decomposition of the Pr-doped solutions was caused by annealing under reduction conditions (750 - 800°C, $N_2$-atmosphere). This work was supported by National Program "High-priority problems of condensed matter" (subprogram "Superconductivity", #96079), National Program of leading scientific schools support (#00-15-97435), Russian Foundation for Basic Research (#99-03-32627).

**E5.16**
EFFECT OF NANO-SIZED MgO ADDITION ON MICRO-STRUCTURAL DEVELOPMENT AND SUPERCONDUCTING PROPERTIES OF 61 MULTIFILAMENT BSCCO 2223 TAPES. Jae-Woong Ko, Jaimoo Yoo, Shin-Chul Kang, Hai-Doo Kim, Korea Institute of Machinery and Materials, Materials Engineering Dept., Changwon, Kyungnam, KOREA; Hyungsik Chung, Ajou Univ., Division of Mechanical and Industrial Engineering, Suwon, KOREA.

The effect of inert second phase on microstructural development and superconducting properties has been studied in order to increase Jc under magnetic field. In this study, nano-sized MgO particles were homogeneously mixed with BSCCO 2223 precursor using planetary ball milling and ultrasonification and then 61 multifilaments BSCCO 2223/MgO tapes were fabricated by PIT method. Microstructure and phase were analyzed by XRD, SEM and DTA. The critical current density was measured under magnetic field at 77K. Homogeneous distribution of MgO particles in BSCCO 2223 matrix led to increase in Jc under magnetic field.

**E5.17**
BSCCO CERAMICS WITH MICROINCLUSIONS OF THERMODYNAMICALLY COMPATIBLE PHASES. Pavel E. Kazin, Yuri D. Tretyakov, Vasili V. Lennikov, Marina A. Makarova, Andrey S. Karpov, Moscow State Univ, Dept of Chemistry, Moscow, RUSSIA; Martin Jansen, MPI Institut fuer Festkoerperforschung, Stuttgart, GERMANY.

Critical current density in BSCCO superconductors is strongly restricted on account of their low flux pinning ability. A promising way to solve this problem is connected with producing superconductor material containing fine inclusions acting as effective pinning centers. In the present work an original approach was elaborated in order to create submicron precipitates of thermodynamically compatible phases in the BSCCO matrix. Systems Bi-Sr-Ca-Cu-A-O (A=Mg, Al, In, Zr, Sn, P) were investigated and several A-containing oxides were found to be thermodynamically compatible with the Bi-2212 superconductor. The superconductor composites were prepared via melt-processing of either homogeneous precursors containing all the components or a mixture of two finely grained oxide precursors. The inclusions size, shape and spatial distribution depended on preparation conditions and nature of the dopant A. The inclusions of submicron equaxed particles (A=Mg, Al, Zr, Sn) and whiskers (A=In, P) were obtained. Application of chemical reaction between two oxide precursors led to formation of micron-sized particles with complex shape such as hollow perforated shells, serrated or star-like grains. The doping elements were found to be practically insoluble in solid Bi-2212. The composites exhibited Tc above 90 K and enhanced flux pinning at elevated temperatures in comparison with undoped superconductors.

**E5.18**
Abstract Withdrawn.

**E5.19**
INTRINSIC JOSEPHSON JUNCTIONS ON HIGH QUALITY BSCCO THIN FILMS GROWN BY MOCVD. Kazuhiro Endo, Hiroshi Sato, Hiroshi Akoh, Junji Itoh, National Institute of Advanced Industrial Science and Technology, Nanoelectronics Research Institute, Ibaraki, JAPAN; Kohji Kajimura, Japan Society for the Promotion of Machine Industry, Tokyo, JAPAN.

Much attention has been focused on intrinsic Josephson junctions observed in BSCCO single crystals from the point of view of Josephson characteristics control of artificial junctions and findings of new barrier materials as well as high frequency applications in the THz region. In order to improve integration for device applications, it is very important to fabricate Josephson junctions on high-quality thin films. We reports on the successful preparation of intrinsic Josephson junctions on high-quality BSCCO thin films grown by MOCVD. The surfaces of as-grown films were very smooth with a roughness of the order of half unit cell. In addition, there were no apparent grain boundaries, which offers a strong advantage for the improvement of integrgration. A typical I-V characteristic in the c-axis direction of a BSCCO film at 4.2K shows multiple resistive branches. The number of branches is consistent with that of junctions calculated from the height of a mesa structure. This indicates that BSCCO films consist of a series array of S($CuO_2$ layer) / I(SrO,BiO layers) / S($CuO_2$ layer) junction. The formation of stacked Josephson junctions was also confirmed by magnetic field dependence of Ic.

**E5.20**
ORIENTATION CONTROL OF HTS THIN FILMS. Kazuhiro Endo, Paul Fons, Junji Itoh, National Institute of Advanced Industrial Science and Technology, Nanoelectronics Research Institute, Ibaraki, JAPAN; Kohji Kajimura, Japan Society for the Promotion of Machine Industry, Tokyo, JAPAN.

The very short coherence length along the c-axis direction of high Tc superconductor (HTS) is a big problem for electronics application such as Josephson tunneling devices. Orientation control of HTS thin films is required, because the coherence length along non c-axis directions is longer in comparison with c-axis direction. For the first time, we have succeeded in growing bismuth-2223 films oriented along the [119] direction in situ by metalorganic chemical vapor deposition (MOCVD). Bismuth-2223 superconductor has the highest Tc among bismuth series oxide superconductors. The key to controlling the orientation is the use of the following growth conditions; substrates and growth temperatures. Here, (110) strontium titanate and (100) neodium galate single crystals were used as substrates. These substrates have favorable lattice matching with the (119) plane of bismuth-2223 superconductor, e.g., a misfit of -2.17 percent along the [100] direction and 1.96 percent along [110] for (110) strontium titanate, and a misfit of 0.4 percent along [010] and 1.25 percent along [001] for (100) neodium galate. A two step process was used involving a first stage in which growth was carried out at a lower temperature to favor heteroepitaxy of (119) oriented bismuth-2223 films and a second stage in which higher temperature growth was used to improve crystallinity and superconducting properties. The structure of the films was examined by X-ray diffraction. The surface morphology was observed by atomic force microscopy (AFM). The AFM images show a mountain-range-shaped morphology, which reflects the twinning growth of bismuth-2223 films. Similar AFM images were obtained for (103) oriented YBCO films grown by MOCVD. Finally, vicinal substrates were used in order to obtain twin-free bismuth-2223 films oriented along the [119] direction.

**E5.21**
INFLUENCE OF NICKEL DOPING ON THE PHASE FORMATION AND SUPERCONDUCTING PROPERTIES OF *Bi-Pb-Sr-Ca-Cu-O* CERAMICS. S.A. Halim, and A.I. Malik Univ, Putra Malaysia, Dept of Physics, Serdang, MALAYSIA.

The influence of nickel doping on the phase formation and superconducting properties of $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}Ni_xCu_3O_\delta$ with $x_{0.0to0.10}$ were investigated in detail, with the help of scanning electron microscopy, powder X-ray diffraction analysis, resistance and ac susceptibility characterizations. The measurements revealed that the transition temperatures and the formation of high-$T_c$ phase{2223} are slowly decreased with the increase of nickel content up to {0.05}. However beyond this concentration, the low-$T_c$ phase{2212} is dominant and a clear two step transition due to the mixed phases was observed. The results of phase formation and the variety in $T_c$ values are discussed on the bases of different superconducting phases and the weak-linked granular nature of the samples.

**E5.22**
Abstract Withdrawn.

**E5.23**
PREPARATION AND ELECTRONIC DEVICE PROPERTIES OF SINGLE CRYSTALLINE $La_{2-x}Sr_xCuO_4$ SUPERCONDUCTING FILM. A.T.M. Nazmul Islam, Ken Ashizawa, Satoshi Watauchi, Isao Tanaka, Institute of Inorganic Synthesis, Yamanashi University, Kofu, JAPAN.

The intrinsic Josephson junctions in $La_{2-x}Sr_xCuO_4$ single crystals are superior to Nb-SIS junctions, due to higher plasma frequency and smaller vortex quantum size. But the device processing using the bulk superconducting single crystals is complicated. $a_{tetra}$-axis orientated single-crystalline films of thickness of micron-order would be more desirable for the processing and development of electronic devices. In liquid phase epitaxial (LPE) technique there remains the possibility of contamination from crucible and hence disruption of the superconducting and intrinsic electronic device property of the films. So we have established a new LPE technique, infrared heated LPE (IR-LPE) technique using no crucible, where the techniques used in TSFZ method was applied to film preparation. $La_{2-x}Sr_xCuO_4$ single-crystalline films with $0.08 \leq x \leq 0.15$ and thickness less than 100 $\mu$m could successfully be prepared in this technique. $a_{tetra}$-axis orientated Zn-doped $La_2CuO_4$ single crystals of thickness ~1mm was used as substrate. Small electronic device was fabricated on the film by focus ion beam etching. The films were then characterized by their

superconducting and intrinsic Josephson junction property measurements. The prospect of use of IR-LPE film in electronic device applications was investigated. This work was partially supported by CREST, Japan Science and Technology (JST) Corporation, Japan.

**E5.24**
PREPARATION OF $[Ba_2CuO_2(CO_3)]_M[ACuO_2]_N$(A=Sr,Ca) FILMS BY MBE TECHNIQUE. Yutaka Adachi, Yoshio Matsui, Isao Sakaguchi, Hajime Haneda, Koichiro Takahashi, Advanced Materials Laboratory, National Institute for Materials Science, Tsukuba, JAPAN.

$[Ba_2CuO_2(CO_3)]_m[ACuO_2]_n$ superlattices containing oxycarbonate blocks as a charge reservoir have been prepared on $SrTiO_3$ using the molecular beam epitaxy technique. First, thin films of the oxycarbonate cuprate $Ba_2CuO_2(CO_3)$ have been prepared on $SrTiO_3$(001) using $NO_2$ as an oxidant and $CO_2$. The films have been grown at 500°C substrate temperature. At higher substrate temperature or at lower $CO_2$ pressure $Ba_2CuO_3$ was formed instead of $Ba_2CuO_2(CO_3)$, and the thin films become amorphous at lower temperature. XRD and RHEED observations indicated that $Ba_2CuO_2(CO_3)$ grew along the [001] crystal orientation on $SrTiO_3$(001) with following epitaxial relationship: $Ba_2CuO_2(CO_3)$[100]//$SrTiO_3$[110] and $Ba_2CuO_2(CO_3)$[110]//$SrTiO_3$ [100]. The depth profile of SIMS indicated the incorporation of carbon into the films. As-grown films behaved a semiconducting property in electrical measurement. No evidence of superconductivity has been observed down to 5K. Secondly, the oxycarbonate cuprates and infinite layers have been alternately stacked. It was confirmed that $Ba_2CuO_2(CO_3)$ was inserted between several unit cells of $SrCuO_2$.

**E5.25**
REVERSIBLE CONVERSION BETWEEN Hg-1212 AND Tl-1212 SUPERCONDUCTING THIN FILMS IN CATION EXCHANGE PROCESSES. Zhongwen Xing, J.Z. Wu, University of Kansas, Dept of Physics and Astronomy, Lawrence, KS.

In a recently developed cation exchange process, Tl-based high $T_c$ superconductors are used as "precursors" that are converted to Hg-based high-temperature superconductors when Tl cations are replaced by Hg cations, leaving the overall structure of the lattice nearly unchanged. We report in this experiment that such a conversion between $TlBa_2CaCu_2O_7$ (Tl-1212) and $HgBa_2CaCu_2O_6$ (Hg1212) is completely reversible and the direction of the conversion is determined by the population ratio of the Tl and Hg cations. The experiment was composed two steps. In step 1, a Hg1212 film was annealed in Tl vapor at 740°C~780°C to convert it to Tl-1212 films through cation exchange. In contrast, a control film of amorphous Tl-Ba-Ca-Cu-O became $Tl_2Ba_2CaCu_2O_9$ (Tl-2212). In step 2, the Tl-1212 film was annealed in Hg vapor to form the Hg-1212 films through cation exchange. The quality of the Hg-1212 film is almost identical to the starting Hg-1212 film. The resistance transition temperature ($T_c$) is close to 120K and critical current densities ($J_c$) of the Hg-1212 film is 1.8MA/cm$^2$ at 77K in zero field. This demonstrates that the conversion between Hg-1212 and Tl-1212 is reversible and the "1212" crystalline structure is stable during the cation exchange.

**E5.26**
NON-STOICHIOMETRY IN $La_2CuO_4$: THE $La_2Cu_{1-x}Sn_xO_{4+\delta}$ SOLID SOLUTION. Georges Dénès[a], Krzysztof Ruebenbauer[b], Glenn Taylor[a] and Brent Thompson[a], [a]Concordia Univ, Department of Chemistry and Biochemistry, Laboratory of Solid State Chemistry and Mössbauer Spectroscopy, Laboratories for Inorganic Materials, Montreal, Québec, CANADA; [b]Pedagogical University, Mössbauer Spectroscopy Laboratory, Cracow, POLAND.

"Useful" doping of $La_2CuO_4$ high $T_c$ superconductor have usually been restricted to substitution on the lanthanum site, usually by $M^{2+}$ divalent cations (alkaline earth metals), and this also resulted in non-stoichiometry on the oxygen site, which is further modified by the additional oxygen resulting from the oxidation of some Cu(II) to Cu(III) by annealing under oxygen. Most substitutions on the copper site, usually by transition metals, have resulted in a drastic decrease of $T_c$. Recently, we carried out a 10% substitution of copper by tin(IV), to give non-stoichiometric $La_2Cu_{1-x}Sn_xO_{4+\delta}$, where ideally $\delta$=0.10 if all the copper is in the 2 oxidation state. Microwave absorption measurements showed that, contrary to earlier substitutions on the copper site, 10% substitution by tin results in no decrease of $T_c$, which actually increases by 1K. We have now carried out an investigation of the material obtained versus the substitutional fraction x. It was found that the $La_2Cu_{1-x}Sn_xO_{4+\delta}$ solid solution exists for $0 \leq x \leq 0.12$. $^{119}$Sn Mössbauer Spectroscopy shows that all the tin is in the stannic form, and that at least two kinds of tin(IV) are present, with the coordination of one being much more distorted than the other.

**E5.27**
INFLUENCE OF LATERAL CRITICAL CURRENT DENSITY DISTRIBUTIONS ON AC TRANSPORT LOSSES FOR Ag-SHEATHED (Bi,Pb)-2223 MULTIFILAMENTARY TAPES WITH DIFFERENT FILAMENT ARRANGEMENTS. Ryoji Inada, Shusaku Sakamoto, Pingxiang Zhang, Akio Oota, Toyohashi Univ of Technology, Dept of Electrical and Electronic Engineering, Aichi, JAPAN; Hiroyuki Fujimoto, Railway Technical Research Institute, Tokyo, JAPAN.

The influence of the lateral critical current density ($J_c$) distributions on AC transport losses in self-fields at 77 K have been investigated on Ag-sheathed (Bi,Pb)-2223 multifilamentary tapes with different filament arrangements. Two deformation methods were applied to prepare the wires with different filament arrangements, one is an ordinary drawing process by using round dice, another is a rectangular deformation by a Turk's Head with four roller machine. Finally, the ordinary one-axial rolling process and heat treatments were applied to make all the multifilamentary tapes. The residual self-field distributions on the tape surface measured by scanning Hall probe depends on the configuration of each tape, because the lateral $J_c$-distributions are strongly influenced from the non-uniformity of the filament deformation in tapes caused in the rolling process. The measured AC transport losses of each tape are nearly independent of frequency between 50 and 500 Hz, indicating that the main contribution to losses comes from the hysteresis loss in the superconductor filaments. However, there is significant dependence of the loss values on the filament arrangements in each tape. Using the theory of Norris, these results are explained by the calculated results for the shape of field-free-core and flux-penetration regions, varying with filament arrangements and non-uniform lateral $J_c$-distributions.

**E5.28**
NEW DAMAGE MORPHOLOGIES AND THEIR FORMING MECHANISM IN HEAVY ION IRRADIATION. Daxiang Huang, Superconductivity Research Laboratory, ISTEC, Tokyo, JAPAN; Yukichi Sasaki, Japan Fine Ceramics Center, Nagoya, JAPAN; Yuichi Ikuhara, Engineering Research Institute, University of Tokyo, Tokyo, JAPAN.

230-MeV Au and 180-MeV Fe ion beams were used to bombard $Bi_2Sr_2CaCu_2O_x$ (Bi2212) single crystal sheets (~30$\mu$m thick) at room temperature using a Tandem accelerator. Cross section transmission electron microscopy observations were performed to analyze the damage evolution along the ion traces. Located in between the two typical irradiation-induced defect morphologies, parallel columnar defects and disordered cascade defects, we found three new types of transitive defect morphologies, large-angle-deflected columnar defects, cascade-defect-dotted columnar defects, and ordered cascade defects. Based on the analyses of the ion-energy deposition process along the ion traces, the forming mechanisms of these three new type defects were suggested. The columnar defect size was measured systematically along the ion traces using high-resolution electron microscopy, which leads us to study the ion velocity effects on the irradiation damage conveniently in a single ion-target system. In this system, the complicated influences on the irradiation damage due to the different target materials and the different ion species can be neglected. Then, a novel method was suggested by simply considering the dependence of damage efficiency on ion velocity, which can further minimize the influence of the ion effective charge and let us to understand the pure influence of ion velocity in the irradiation damage process. The application on the Au-Bi2212 system shows that there is a critical velocity $V_c \sim 0.057c$ (c, the velocity of light) at which the damage efficiency is maximum. This is a general phenomenon confirmed by reanalyzing the previous published data. From this observation, the irradiation damage process can be divided into two stages: the ion velocity controlled stage and the energy-density threshold controlled stage. In the Fe-irradiated sample, we found the long columnar defects with constant column size and the low threshold of stopping power for generating columnar defects. Combining with the Au irradiation data, a model is suggested which can explain the specific phenomena existed in the Fe-irradiated sample.

## SESSION E6: YBCO COATED CONDUCTORS I
Chairs: Robert A. Hawsey and Stephen R. Foltyn
Wednesday Morning, November 28, 2001
Room 200 (Hynes)

**8:30 AM *E6.1**
OVERVIEW OF U.S. DEPARTMENT OF ENERGY SUPERCONDUCTIVITY PROGRAM FOR ELECTRIC POWER. Marshall Reed, U.S. Department of Energy, Washington, DC.

A top priority of our government is to improve the energy delivery infrastructure. According to the report issued May 2001 by the

National Energy Policy Development (NEPD) Group, a group chaired by Vice-President Dick Cheney, "For the electricity we need, we must be ambitious. Transmission grids stand in need of repair, upgrading, and expansion if we put these connections in place, we'll go a long way to avoiding future blackouts." The U.S. Department of Energy (DOE) leads the U.S. national effort to develop high-temperature superconducting (HTS) wires and to demonstrate prototype electric power applications using the best wires available today. Superconductivity is such an important aspect of our nations energy future that the report recommended that the U.S. should "expand the Department's research and development on transmission reliability and superconductivity." Prototype superconducting power cables that will help upgrade our power grid when commercialized later this decade are being demonstrated now in Carrollton, Georgia and Detroit, Michigan. Research and development remains, however, if the superconducting wires are to have the electrical performance and low-cost features necessary for U.S. companies to fully commercialize new transmission, distribution, and generation technology that will help eliminate the bottlenecks in our urban areas and strengthen our grid. The industry-led Second Generation Wire Initiative is exploiting breakthroughs at Los Alamos and Oak Ridge national laboratories that promise unprecedented current carrying capacity in HTS wires. The most recent progress in the U.S. development of HTS "coated conductors" and in fielding prototype applications will be presented.

**9:00 AM *E6.2**
PROGRESS IN COATED CONDUCTOR DEVELOPMENT AT LOS ALAMOS NATIONAL LABORATORY. S.R. Foltyn, P.N. Arendt, P.C. Dowden, R.F. DePaula, J.R. Groves, L. Stan, H. Kung, T.G. Holesinger, Q.X. Jia, B.J. Gibbons, V. Matijasevic, J.Y. Coulter, E.J. Peterson, Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM.

Since 1993 Los Alamos has been working to advance the performance levels and demonstrate the commercial feasibility of coated conductors consisting of YBCO deposited on flexible metal. We have concentrated exclusively on the use of ion beam assisted deposition (IBAD) - as pioneered in 1991 by Fujikura, Ltd. and Lawrence Berkeley National Laboratory - and pulsed laser deposition (PLD) for the textured template and superconductor, respectively. In 1995 we demonstrated conductors with critical current density (Jc) of over 1 MA/cm2. More importantly we also showed that this Jc could be maintained for YBCO thickness levels that allowed the attainment of critical current (Ic) values of up to 200 A in a centimeter-wide tape. The next step was to adapt these processes to coat longer lengths of moving tape, an effort that culminated in 1999 with the fabrication of several one-meter-long conductors having Ic > 100 A. We are now pursuing further advances in three areas: improved performance, reduced cost, and longer length. For the first, we are developing processes that allow high Jc YBCO to be grown to thickness levels of 5 microns or more, and have demonstrated the potential to reach 600 A in a cm-wide tape. Second, because of its evolutionary texture development, IBAD of YSZ is the costliest step in our process: This economic issue has been addressed by switching to IBAD MgO, which forms a textured template 100 times faster than YSZ. We have now demonstrated YBCO performance levels on IBAD MgO in excess of 200 A/cm-width. Finally to produce longer lengths of tape for applications development, and in support of our industrial partners, we are developing a new facility with reel-to-reel tape transport systems to scale these successful processes up to lengths of 10-100 meters.

**9:30 AM E6.3**
GRAIN BOUNDARY FACETING IN $YBa_2Cu_3O_{7-x}$ BICRYSTAL THIN FILMS ON $SrTiO_3$ SUBSTRATES. Qiang Jin, Siu-Wai Chan, Columbia Univ, Dept of Applied Physics and Applied Mathematics, New York, NY.

The structure of grain boundaries in $YBa_2Cu_3O_{7-x}$ (YBCO) thin films deposited on [001] tilt $SrTiO_3$ bicrystal substrates has been characterized by transmission electron microscopy (TEM). It was found that the YBCO film boundaries were meandering along the relatively straight substrate boundaries. High -resolution lattice images indicated that the microscopic meandering of the film boundary essentially consisted of many straight segments of facets at the atomic scale. The observed facets correspond to low-index crystallographic planes in each of the adjacent grains. The orientations of the facets tend to have small deviation angles ($<28^\circ$) from the designed template boundary. The closure failure formed by the crystallographic planes of the facets in the two crystals compared with the misorientation angles are generally less than $2^\circ$. The factors controlling the formation of facets are discussed based on a three-criteria faceting model.

**9:45 AM E6.4**
PULSED ELECTRON-BEAM DEPOSITION - A NOVEL THIN FILM DEPOSITION TECHNIQUE FOR HIGH TEMPERATURE SUPERCONDUCTING THIN FILMS. K.S. Harshavardhan, M. Strikovski and J. Kim, Neocera, Inc., Beltsville, MD.

A novel thin film deposition technique based on pulsed electron beams is presented in this paper. Pulsed Electron-beam Deposition (PED) is a process in which a pulsed (100 ns) high power electron beam (approximately 1000 A, 15 keV) is incident on the surface of a target facilitating stoichiometric thin film deposition. Due to this unique feature, the Pulsed Electron-beam Deposition technique, as in the case of Pulsed Laser Deposition (PLD), provides a unique platform for depositing thin films of complex materials on a variety of technologically important substrates. Deposition rates between 0.5-1 nm/pulse can be achieved by this method. In contrast with PLD, where the ablation process is critically dependent on the optical absorption coefficient of the target material, in PED, the ablation depends only on the range of electrons in the target. For most of the solid state materials, this range is of the order of a few microns. Due to this unique difference in the beam-solid interaction mechanism, PED is expected to extend the range of materials that can be deposited as thin films. Our paper will present data obtained on epitaxial high-temperature superconducting (YBCO) films with superconducting transition temperatures in the range of 87-88 K and transition widths of 0.5 K. We will also present data on other oxide films relevant to superconducting technologies. We will compare the relative merits of this new deposition technique with relatively mature PLD technique.

**10:00 AM E6.5**
CONTINUOUS REEL-TO-REEL CHEMICAL SOLUTION DEPOSITION OF EPITAXIAL $Gd_2O_3$ BUFFER LAYERS ON BIAXIALLY TEXTURED Ni-W (3 at. %) ALLOY SUBSTRATES FOR THE FABRICATION OF HIGH-$J_c$ $YBa_2Cu_3O_{7-\delta}$ COATED CONDUCTORS. Tolga Aytug, M. Paranthaman, S. Sathyamurthy, B.W. Kang, D.B. Beach, A. Goyal, P.M. Martin, D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

A low-cost, non-vacuum reel-to reel dip-coating unit has been used to continuously deposit epitaxial $Gd_2O_3$ buffer layers on mechanically strengthened biaxially textured Ni-W (3 at. %) alloy tapes. A 2-methoxyethanol solution of gadolinium methoxyethoxide/acetate, prepared by an alkoxide sol-gel synthesis route, was used to coat the Ni-W (3 at. %) tapes. Due to its significance as a seed layer, optimum processing conditions (post-annealing speed and temperature) of $Gd_2O_3$ buffer layers have been studied. While highly textured films were obtained under reducing forming gas (96%Ar + 4% $H_2$) atmosphere at temperatures between 1100-1150°C, post-annealing speed did not significantly affect the crystalline quality of the $Gd_2O_3$. Scanning electron microscopy observations revealed a continuous, dense and crack-free surface morphology for these dip-coated buffers. The thickness of the $Gd_2O_3$ layers led to remarkable differences on the growth characteristics of the subsequent YSZ and $CeO_2$ layers deposited by rf-magnetron sputtering to complete the buffer-layer architecture. Epitaxial YBCO films grown by pulsed laser ablation on the $CeO_2$/YSZ/$Gd_2O_3$ /Ni-W (3 at. %) multilayer structure yielded self-field critical current densities ($J_c$) as high as $1.2 \times 10^6$ A/cm$^2$ at 77K. This performance attained on solution buffered Ni-W (3 at. %) alloy tapes is comparable to that of YBCO films grown on biaxially textured Ni substrates in which all buffer layers are deposited by vapor deposition techniques. Supported by the U.S. DOE, Division of Materials Sciences, Office of Science, and Office of Power Technologies-Superconductivity Program, Office of Energy Efficiency and Renewable Energy. The research was performed at the Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the USDOE under contract DE-AC05-00OR22725

**10:30 AM *E6.6**
PERCOLATION AND GRAIN BOUNDARIES AND THEIR EFFECT ON THE CURRENT DENSITY IN HTS CONDUCTORS. David Larbalestier, Matt Feldmann, George Daniels, Anatoly Polyanskii, and Alex Gurevich, University of Wisconsin-Madison, Madison, WI.

Magneto optical images of polycrystalline HTS conductors make it absolutely clear that the supercurrent path is controlled by many obstacles, notable amongst which are low angle grain boundaries. This is particularly clear for YBCO coated conductors, even for those with Jc(77K, 0T) values exceeding 2 MA/cm2. Coupled study of polycrystalline tracks and single grain boundaries makes it clear that dissipation is highly localized in such CC and that a zero field critical current density measurement does not put the whole sample into a critical state. Careful comparison of single GBs in bicrystals grown on STO and of single GBS isolated within CC suggests very similar behavior, even though the highest values of Jc exceed 5 MA/cm2 within individual CC grains. We will summarize recent results aimed at understanding the coupling between the intergrain and intragrain current densities in coated conductors. *Work carried out in collaboration with American Superconductor Corporation and Oak Ridge National Laboratory staff.

**11:00 AM E6.7**
DEPOSITION OF YBCO THIN FILM ON METALLIC SUBSTRATES BY A SOL GEL METHOD. Donglu Shi, Yongli Xu, Shaun McCllelen, and Relva Buchanan, Dept. of MS&E, University of Cincinnati, Cincinnati, OH; Lumin Wang and Shixin Wang, Dept. of Nuclear Engineering and Radiological Science, University of Michigan, Ann Arbor, MI.

A unique Sol Gel process has been developed without using fluorine in our laboratory to deposit YBCO thin films on various substrates including silver and silver alloy for conductor development. On single crystal substrates such as $LaAlO_3$ and YSZ, the growth has been found to be epitaxial. Both rocking curve XRD and HRTEM experimental results have shown consistent structural behaviors of the thin films that the YBCO growth is coherent with the lattice structure of the substrate. Transport resistivity measurements have shown sharp Tc's in these sol gel thin films indicating their high qualities. The growth mechanisms and sol gel chemistry will be discussed in this presentation.

**11:15 AM E6.8**
NANOPARTICLE FORMATION FROM $Y_1Ba_2Cu_3O_{7-X}$ FOR POTENTIAL FLUX PINNING MECHANISM. Paul N. Barnes, P. Terry Murray, Tim Haugan, Propulsion Directorate, Air Force Research Laboratory, Wright-Patterson AFB, OH; Richard Rogow, Glen P. Perram, Department of Engineering Physics, Air Force Institute of Technology, Wright-Patterson AFB, OH.

An initial report of nanoparticle formation from by laser ablation of $Y_1Ba_2Cu_3O_{7-X}$ (YBCO) will be presented. The nanoparticles were created by irradiating a target of bulk YBCO with a KrF excimer laser in a pulsed laser deposition setup used for making thin film YBCO. Background pressures of 5 Torr O2 were used. The nanoparticles ranged in size from ~3 to 5 nm for the conditions used with a laser energy of 50 mJ per pulse. Characterization of the laser plume was performed using optical emission spectroscopy. Although the visible plume emission is very weak under the conditions used to create the nanoparticles, the plume remained collisionally dynamic at the high pressure and low laser energy used. Use of the nanoparticle formation for flux pinning will be discussed.

SESSION E7: YBCO COATED CONDUCTORS II
Chairs: Darren Verebelyi and M. Parans Paranthaman
Wednesday Afternoon, November 28, 2001
Room 200 (Hynes)

**1:30 PM *E7.1**
PHASE AND MICROSTRUCTURE EVOLUTION OF HIGH CRITICAL CURRENT DENSITY TFA-MOD YBCO COATED CONDUCTORS. Yutaka Yamada, Takeshi Araki, Haruhiko Kurosaki, SeokBeom Kim, Toyotaka Yuasa, Yuh Shiohara, Izumi Hirabayashi, Superconductivity Research Laboratory, Nagoya, JAPAN; Yasuhiro Iijima, Takashi Saito, Fujikura Ltd., Tokyo, JAPAN; Junko Shibata, Yuichi Ikuhara, Tokyo University, Tokyo, JAPAN; Takeharu Katoh and Tsukasa Hirayama, Japan Fine Ceramic Center, Nagoya, JAPAN.

We have obtained high $J_c$ values for TFA-MOD processed YBCO, for example, 7.8 MA/cm$^2$ on LaAlOA/cm$^3$ substrates and 2.5 MA/cm$^2$ on metallic IBAD substrates. Using resistivity measurements during firing up to 800°C, AFM and TEM studies, phase change has been investigated for the high $J_c$ TFA-YBCO samples. Precursors consisted of fine CuO and amorphous Y-Ba-Cu-O-F was further oxidized in the firing of a low pressure oxygen atmosphere at a temperature as low as 600°C and then seemed to be transformed into Y123 at 670°C. Moreover, the microstructures formed in an early stage and a terminate stage at 800°C firing differed each other. The former was considered to be more textured and uniform structure than the latter and thus the interface region near the substrate was estimated to have higher $J_c$ than the above nominal $J_c$ of 7.8 MA/cm$^2$ and 2.5 MA/cm$^2$.

**2:00 PM *E7.2**
RECENT ADVANCES IN THE FABRICATION OF HIGH-Jc TAPES BY EPITAXIAL DEPOSITION OF YBCO ON RABITS. A. Goyal, B.W. Kang, D.F. Lee, M. Paranthaman, C. Cantoni, K.L. More, L. Heatherly, K. Leonard, S. Sathyamurthy, R. Feenstra, J.R. Thompson, D.K. Christen, F.A. List, M. Kowaleski, T. Aytug, D.M. Kroeger and N. Rutter.

Progress made in the fabrication of high-Jc tapes by epitaxial deposition of YBCO on rolling assisted biaxially textured substrates (RABiTS) is reported. A high critical current density of 4.4 MA/cm2 at 77K, self-field was obtained on a single crystal Ni foil made by a modified RABiTS approach. Similar results are also obtained for intragranular, transport Jc measurements on typical, epitaxial YBCO films on RABiTS. This provides a significant motivation for improving the texture of the metal template used in the RABiTS process. Recent progress made in developing substrates with improved textures will be reported. Buffer layer deposition on such substrates and the importance of controlling surface segregation of impurities for obtaining high quality epitaxial films will be discussed. It has been suggested that Ca-doping may mitigate the effects of grain boundaries in coated conductors. We summarize here our results on Ca-doping of grain boundaries in the YBCO films on RABiTS. It is also of interest to grow thick YBCO films with high Ic/width. Efforts at fabricating thick YBCO films on RABiTS using pulsed laser deposition will be summarized. Mechanisms of Jc reduction with increasing YBCO thickness will be discussed. Lastly, a status report on reel-to-reel fabrication of meter long YBCO samples using the ex-situ BaF2/TFA process on RABiTS will be given. Research sponsored by US Department of Energy, Office of Energy Efficiency and Renewable Energy, Office of Power Technologies, Superconductivity Program under contract DE-AC05-96OR22464.

**2:30 PM E7.3**
COATED CONDUCTORS WITH HIGH STRENGTH BIAXIALLY TEXTURED Ni-ALLOY SUBSTRATES. Bernd de Boer, Vadlamanyi S. Sarma, Laura Fernandez G.-R., Norman Reger, Joerg Eickemeyer, Bernhard Holzapfel, IFW Dresden, Institute of Metallic Materials, GERMANY; W. Prusseit, THEVA GmbH, GERMANY.

To increase the overall current density of the YBCO coated conductor either the film thickness can be increased or the substrate thickness can be reduced. Unfortunately the superconducting film quality deteriorates with increasing thickness and therefore the total current is limited. The reduction of the substrate tape thickness is limited by the material strength. By enhancing the strength, the tape thickness can be further reduced and thereby the current density of the coated conductor improved. The metallurgical ways of strengthening RABiTS kind of substrate materials are constraint by the necessity of the very strong cube texture. In this contribution two feasible ways of substrate strengthening are proposed. By solid solution strengthening the tape yield strength can be enhanced up to a factor of four in comparison to pure Ni. The strengthening is limited by the solute concentration at which the cube texture deteriorates. Besides strengthening, Cr- and V-solutes in Ni reduce the Curie temperature below 77K what is necessary for ac applications. The formation of Cr- or V-oxides at the surface before coating is prevented by the PVD of a thin Ni layer in HV. Another way of strengthening is by introducing finely dispersed particles into the tape matrix. In order to maintain a strong cube texture the particles have to be formed after the recrystallization annealing by internal oxidation of solute Al. The room temperature yield strength of these tapes reaches up to five times the value of pure Ni. A further enhancement of the strength is possible by combining solid solution hardening with particle strengthening. Also these tapes have been coated with a Ni layer before buffer and YBCO deposition. Coating of the tapes have been carried out by PLD or thermal coevaporation.

**2:45 PM E7.4**
X-RAY DIFFRACTION MAPPING OF YBCO SUPER-CONDUCTING TAPE ON A MESOSTRUCTURAL SCALE. E.J. Peterson, Los Alamos National Laboratory, Superconductivity Technology Center, Los Alamos, NM; U. Preckwinkle, B.N. Herrington, Bruker Axs, Inc. Madison WI; F.M. Mueller, D.J. Brown, G.W. Brown, M.E. Hawley, J.Y. Coulter, S.R. Foltyn, P.N. Arendt, and D.E. Peterson, Los Alamos National Laboratory, Superconductivity Technology Center, Los Alamos, NM.

In the production of second-generation superconducting tapes, the quantification of substrate and superconductor in-plane texture by x-ray diffraction has been found to be one of the best ways to predict the final tape current transport performance. However, it is also possible to obtain crystallographic information regarding superconductor oxygen stoichiometry, cation disorder, and overall crystal quality through the analysis of x-ray diffraction peak positions, intensities, and shapes. With the advent of 2-dimensional x-ray detectors and focused x-ray sources it is possible to collect this information on a relatively fine spatial scale and in reasonable time frames. We present here the results of mesostructural (approximately 1 mm resolution) x-ray diffraction mapping over a 3.3 cm x 1 cm region of YBCO coated conductor tape that had been produced by combined IBAD/PLD techniques. The x-ray diffraction mapping results are compared to images of the supercurrent paths, as obtained using a magnetic field scanning technique. Correlations are made with regard to features in the diffraction maps and the supercurrent images.

**3:00 PM E7.5**
TRANSPORT PROPERTIES OF Cr-PATTERNED $YBa_2Cu_3O_7$ THIN FILMS. Rajesh Chopdekar, Cornell Univ, Dept of Applied and Engineering Physics, Ithaca, NY; Darren Dale, Yuri Suzuki, Cornell Univ, Dept of Materials Science and Engineering, Ithaca, NY.

Epitaxial thin films of $YBa_2Cu_3O_7$ (YBCO) have been deposited and patterned on (100) $SrTiO_3$ and (110) $NdGaO_3$ substrates. Wet etchants used in conventional photolithography react too quickly with YBCO thin films, resulting in undercutting of patterned structures. We have implemented a technique to pattern thin lines from films without wet or dry etching. Chromium is evaporated onto the substrates and subsequently patterned. YBCO is then deposited by pulsed laser deposition, which results in an epitaxial YBCO film on patterned areas and randomly oriented YBCO deposited on chromium. An annealing step is required for the chromium to diffuse through the undesired YBCO in order to render those sections insulating. Rutherford Backscattering Spectrometry performed after annealing confirms Cr diffusion up through the undesired YBCO areas. YBCO lines patterned with this technique exhibit resistivity and critical temperatures similar to those of the unpatterned films. As we decrease the width of the YBCO lines, the effect of the edges, where there is chromium-reacted material, on transport becomes more pronounced. The resolution of the patterning technique and the profile of superconducting versus non-superconducting materials at the edges will be explored.

**3:30 PM *E7.6**
SCALE UP OF YBCO COATED CONDUCTOR PROCESSES AT IGC-SUPERPOWER. V. Selvamanickam, IGC-SuperPower, Schenectady, NY.

IGC-SuperPower has established pilot-scale facilities for scale up of Coated Conductor processes to manufacturing. These facilities have been designed for continuous processing of substrates, buffer layers, and superconducting layers over time periods of a week. To enable continuous processing over long time periods and with high throughput, the processing equipment have been designed for providing substantial quantities of source material, with long deposition zones, and ratings for continuous, long operation cycles. Using a reel to reel polishing equipment, tens of meters of substrates have been polished continuously with a high degree of surface finish. Uniform in-plane texture has been achieved in lengths of buffer layers deposited on these substrates. High current densities have been attained in YBCO deposited on the buffered-substrates. The overall activities on scale up of Coated Conductor processes at IGC-SuperPower will be summarized in this presentation. Part of the work was performed under a CRADA with Los Alamos and Argonne National Labs.

**4:00 PM *E7.7**
PROGRESS IN YBCO COATED CONDUCTORS. Suresh Annavarapu, Martin Rupich, Cees Thieme, Qi Li, Sky Cui, Nguyet Nguyen, Sharon Lu, Wei Zhang, Darren Verebelyi, Joe Lynch, and Elliot Thompson, American Superconductor, Westborough, MA.

Progress in the development of economically and technically viable YBCO coated conductor is reported. Biaxially textured metal substrates have been fabricated using reel-to-reel deformation texturing processes. Epitaxial buffer layers have been deposited on deformation textured metal substrates using a combination of reel-to-reel electron-beam evaporation, metal-organic deposition and magnetron sputtering processes. Epitaxial YBCO films have been deposited on lengths of buffered textured substrates using a reel-to-reel solution deposition process. Structure property relationships will be presented and critical issues for future progress will be identified.

**4:30 PM E7.8**
THE PROGRESS ON LOW-COST, HIGH-QUALITY, HIGH-TEMPERATURE SUPERCONDUCTING TAPES DEPOSITED BY THE COMBUSTION CHEMICAL VAPOR DEPOSITION PROCESS. Shara S. Shoup, Marvis K. White, Steve L. Krebs, Adam C. King, Dave S. Mattox, Ian H. Campbell, MicroCoating Technologies, Inc., Chamblee, GA; Ken R. Marken, Seung Hong, Joe Sowa, Bolek Czabaj, Oxford Superconducting Technology, Carteret, NJ; M. Paranthaman, Hans M. Christen, Hong-Ying Zhai, Oak Ridge National Laboratory, Oak Ridge, TN.

The innovative Combustion Chemical Vapor Deposition (CCVD) process is a non-vacuum technique that is being investigated to enable next generation products in several application areas. One such area is high-temperature superconductors (HTS). In combination with the Rolling Assisted Biaxially Textured Substrate (RABiTS) technology, the CCVD process has significant promise to provide low-cost, high-quality lengths of YBCO coated conductor. The CCVD technology has considerable potential to overcome many of the shortcomings of traditional vapor deposition techniques while yielding equal and/or better quality coatings at a lower cost. As a result, capital requirements and operating costs are reduced at least tenfold when compared to competing vacuum-based technologies (e.g. sputtering and MOCVD). The ability to deposit thin films in the open atmosphere enables continuous, production line manufacturing. Consequently, throughput potential is far greater than with conventional thin-film technologies, most of which are generally restricted to batch processing. Such advantages can help decrease the cost of fabricating HTS tape over traditional deposition technologies and thus help meet DOE's target cost goals for making HTS tape competitive with copper. The CCVD technology has been used to deposit both buffer layer coatings as well as YBCO superconducting layers. A buffer layer architecture of strontium titanate and ceria have been deposited by CCVD on textured nickel substrates and optimized to appropriate thicknesses and microstructures to provide templates for growing YBCO with high critical current density values. Films of YBCO superconductors have been grown on single crystal substrates with critical current densities over 1 million amperes per square centimeter. Work is currently in progress to combine both the buffer layer and superconductor technologies to produce high-quality coupons of HTS tape made entirely by the non-vacuum CCVD process. An up-date on the progress made towards scaling the CCVD process will also be given.

**4:45 PM E7.9**
STRUCTURE CHARACTERISTICS OF YBCO COATED CONDUCTORS BASED ON ISD MgO PROCESS. Y.L. Tang, D.J. Miller, Materials Science Division, Argonne National Laboratory, IL; B.H. Ma, R.E. Koritala and U. Balachandran, Energy Technology Division, Argonne National Laboratory, IL.

The inclined-substrate deposition (ISD) method for growth of biaxially textured MgO is of interest due to its applications in coated conductors based on HTS. The ISD method is especially attractive since it offers the potential to produce high-quality biaxially textured layer in a simple and efficient process. In this work, high-resolution electron microscopy (HREM) of YBCO coated conductors based on ISD MgO on Hastelloy tapes was carried out to study both the structure and individual layers and especially the interfaces between them. The results of these studies will be presented with an emphasis on controlling interface reactions.

SESSION E8: POSTER SESSION
Chairs: Venkat Selvamanickam and Suresh Annavarapu
Wednesday Evening, November 28, 2001
8:00 PM
Exhibition Hall D (Hynes)

**E8.1**
A FLUX PINNING MODEL FOR CRITICAL CURRENTS AT LOW-ANGLE GRAIN BOUNDARIES. D. Agassi, Naval Surface Warfare Center, White Oak, MD; S.J. Pennycook, Oak Ridge National Laboratory, Oak Ridge, TN.

Determination of the factors that limit the current carrying capacity of low angle grain boundaries is critical for improved performance of wires and tapes. In the high angle regime the exponential drop in critical current has been successfully modeled as transmission through a continuous Josephson junction with a width determined through a structural unit description of grain boundary atomic structure [1]. At lower angles, however, the situation is more complicated as the boundary comprises an array of discrete dislocation cores. Previous models have used a geometric approach [2] or a Ginsburg-Landau formulation [3] to predict the angular dependence, and are appropriate where the coherence length is large compared to the dislocation spacing in the boundary. However, in the technologically important low field, low temperature, low angle regime, flux pinning is expected to determine the critical current. We present flux pinning calculations [4] for the two limiting descriptions of the bad zone around a grain boundary dislocation, the strain model and the band-bending model. We find significant differences in the angular dependence of critical current that provides insight into the origins of the very different experimental results reported by different groups and also into the improvement in critical current possible through doping. Critical currents parallel to the boundary are predicted to be close to the depairing limit in agreement with recent experiments. [1] N.D. Browning, J.P. Buban, P.D. Nellist, D.P. Norton, M.F. Chisholm and S.J. Pennycook, Physica C 294 (1998) 183. [2] M.F. Chisholm and S.J. Pennycook, Nature 351 (1991) 47. [3] A. Gurevich and E.A. Pashitskii, Phys. Rev. B 57 (1998) 13878 [4] D. Agassi, C.S. Pande and R.A. Masumura, Phys. Rev. B 52 (1995) 16237.

**E8.2**
NEW CONDUCTIVE IBAD-BUFFER FOR HTS APPLICATIONS. Karola Thiele, Sibylle Sievers, Herbert C. Freyhardt, Institut für Materialphysik, Universität Göttingen, GERMANY; Jürgen Dzick, Jörg Hoffmann, Zentrum für Funktionswerkstoffe GmbH Göttingen, GERMANY.

The use of biaxially textured yttria-stabilized zirconia (YSZ) thin

films deposited by an ion-beam-assisted deposition process (IBAD) as a buffer layer for HTS has been widely studied and coated conductors of up to 10m in length have been sucessfully prepared. Two points require further investigation: Firstly, the IBAD mechanisms are not fully understood. Therefore, it is important to follow the texture evolution of IBAD-YSZ films during film growth in detail and to use other IBAD-materials for comparison. Secondly, YSZ is an insulator and, therefore, e.g. a gold shunt is necessary for protecting the HTS film in case of switching to the normal state; an electrically conductive buffer would act as a bypass for the electrical current itself. Both points are shown to be fulfilled with a new electrically conductive buffer material that can be textured by an IBAD process and which serves as a suitable template for YBCO. Biaxially textured conductive buffer layers were deposited by an IBAD process. The deposition conditions (e.g. ion-beam parameters and the angle of incidence) were varied to optimize the in-plane texture. Investigations (TEM and x-ray) of the texture evolution of these IBAD films during film growth were carried out. There are results which can be explained by mechanisms similar to IBAD-YSZ, such as the in-plane texture improvement with increasing film thickness, but also findings which are contrary to the ones obtained for YSZ, such as etching rates and dependences on the incident beam angle. An in-plane texture of 15° FWHM for a 900 nm thick film has been achieved. The quality of the buffer has been proven by the subsequent deposition of YBCO-films deposited by pulsed laser deposition and thermal coevaporation. So far, a $j_C$ of 0.31 MA/cm$^2$ (77K, 0T) has been observed. Part of this work was supported by the German BMBF.

**E8.3**
HIGH Jc YBCO FILMS BY IN-SITU ULTRASONIC SPRAY PYROLYSIS. Annarita Ferreri, Andrey Berenov, Dept. Materials, Imperial College, London, UNITED KINGDOM; Garry Perkins, Yury Bugoslawsky, Anthony D. Caplin, Dept Physics, Imperial College, London, UNITED KINGDOM; Judith L. MacManus-Driscoll, Dept. Materials, Imperial College, London, UNITED KINGDOM.

Superconducting $YBa_2Cu_3O_{7-x}$ thin films were processed by means of in-situ ultrasonic mist pyrolysis, on single crystal oxide substrates. Growth temperatures lower than 900°C have been utilised to spray nitrate precursors. Epitaxial growth of biaxially aligned (<1.5°), 1.5 micron films has been achieved, with Jc's of >4x105Acm$^{-2}$ at 77K, with growth rates of ~0.4 micron/min.

**E8.4**
STUDY OF NiO FILMS GROWN ON PURE Ni, NIi-Cr AND Ni-V SUBSTRATE FOR THE FORMATION OF A BUFFER LAYER COMPONENT FOR REBCO COATED CONDUCTOR. Z. Lockman, J.L. MacManus-Driscoll, Imperial College, London, UNITED KINGDOM; W. Goldacker, R. Nast, Forschungszentrum Karlsruhe, Technik und Umwelt, GERMANY; B. de Boer, Institute of Metallic Materials, IFW Dresden, GERMANY.

In this work, thermal oxidation of rolling assisted biaxially textured substrates {RABiTS}: Pure Ni, Ni-10%Cr, Ni-13%Cr and Ni-9%V has been studied. It was possible to form predominantly cube-textured NiO for both short and long annealing times with a drop of the texture for intermediate times. The texture evolution with time is related to competing nucleation and growth processes on the bare substrate and on the developing NiO surface. For the thin oxide, the best texture was obtained on Ni- 13%Cr and for longer oxidation times the best texture was obtained for pure Ni and Ni-9%V.

**E8.5**
INITIAL GROWTH STUDIES OF REBaCuO THIN FILMS. Guus Rijnders, Victor Leca, Mark Huijben, Martijn Dekkers, Sybolt Harkema, Horst Rogalla, Dave H.A. Blank, Univ of Twente, Low Temperature Div and MESA Research Inst, NETHERLANDS; Sara Bals and Gustaav van Tendeloo, EMAT, Univ of Antwerp, BELGIUM.

Depending on the deposition method, like standard pulsed laser deposition, block-by-block and interval deposition, the structural properties of REBaCuO thin films have been investigated. As is known from literature, the initial growth plays a crucial role in the occurrence of antiphase boundaries, grainboundaries, pseudo-morphic growth and oxidation process. Using well-defined (single terminated) substrates, this initial growth has been studied in-situ using high pressure reflective high energy electron diffraction and ex-situ X-Ray diffraction, Atomic Force Microscopy and Transmission Electron Microscopy. In this contribution an overview is given of the data obtained using these different growth techniques. Besides the above-mentioned properties, special attention will be paid to cation disorder. Furthermore, a comparison will be made in terms of superconducting properties as well as surface morphology.

**E8.6**
BREAKING OF Cu-O CHAINS IN $PrBa_2Cu_3O_7$ UPON Al-DOPING. Mingji Jin[a,c], Q.Y. Chen[a], C.L. Chen[a], Chong Wang[a], Y.S. Song[a], Hye-Won Seo[a], Lixi Yuan[a], W.K. Chu[a], Udom Tipparach[b], T.P. Chen[b] and K.S. No[c]. [a]Texas Center for Superconductivity and Dept. of Physics, Univ. of Houston, TX; [b]Dept. of Physics, Univ. of N. Dakota, ND; [c]Dept. of Mat. Science and Eng., Korea Adv. Inst. of Sci. and Tech., Taejon, SOUTH KOREA.

We have vacuum-deposited c-axis and (110) oriented $PrBa_2(Cu_{0.8}Al_{0.2})_3O_7$ thin films on (001) $LaAlO_3$ and (110) $SrTiO_3$ substrates by rf-sputtering and pulsed laser deposition. From the temperature dependence of resistivities for both the target and the films, we found that the electrical conduction was mainly through the variable range phonon- assisted hopping mechanism. This suggests that Al-doping in this material has given rise to extensive impurity states in the band gap, which, in turn, localized the charge carriers, especially at lower temperatures. The resistivity of the doped PBCO is higher than the pure PBCO, ranged from ~1 Ω-cm at room temperature to about five orders of magnitudes higher at ~30K. The electronic structure of the samples was studied through measuring the optical absorption coefficients vs. photon energy from which the band gap and defect state energies were inferred. Then the soft x-ray emission spectra were obtained for pure and Al-doped PBCO thin films. The shift in Cu L$\alpha$ peak position and the change of Cu L$\alpha$/L$\beta$ intensity ratio in soft x-ray emission spectra after Al doping indicate the change of Cu-O bonding in the lattice due to the partial Al substitution. Also, the Raman spectra for pure and Al-doped PBCO thin films were obtained. An additional peak near the apical oxygen peak was detected in the Al-doped PBCO thin film. This occurs as a result of the broken symmetry of Cu-O chains caused by the partial replacement of the Al atoms with the Cu ions on the Cu-O chains, indicating that the Al atoms primarily substitute the Cu sites on the Cu-O chains.

**E8.7**
STRAIN, NON-STOICHIOMETRY AND DOPING AT LOW-ANGLE GRAIN BOUNDARIES IN YBCO. G. Duscher, Oak Ridge National Laboratory, Oak Ridge, TN and North Carolina State University, Raleigh NC; M. Kim, C. Cantoni, D.K. Christen, Oak Ridge National Laboratory, Oak Ridge TN; A. Franceschetti, S.T. Pantelides, and S.J. Pennycook, Oak Ridge National Laboratory, Oak Ridge TN, and Vanderbilt University, Nashville TN.

The origin of the non-superconducting zone around dislocation cores in YBCO has been probed by a combination of atomic-resolution Z-contrast microscopy, electron energy loss spectroscopy (EELS) and first-principles theory. As in the case of the model perovskite SrTiO3 [1], EELS and theory both show that grain boundaries in YBCO are intrinsically non-stoichiometric. This is the origin of band-bending. Hole depletion is seen up to 2 nm from the cores, even in relatively strain-free regions. We show further that Ca segregates to the dislocation cores without changing the atomic arrangement. The strain field is therefore identical to the undoped boundary, but changes are found in the extent of the hole depletion. These results show that at least in the specific samples studied, band bending is the major factor defining the extent of the non-superconducting zone, and implies significant potential for improvements in critical current through doping. [1] M. Kim, G. Duscher, N.D. Browning, K. Sohlberg, S.T. Pantelides and S.J. Pennycook, Phys. Rev. Letts. 86 (2001) 4056.

**E8.8**
FAST MOD PROCESSING OF YBCO FILMS. Igor Seleznev and Michael J. Cima, Massachusetts Institute of Technology, Dept of Materials Science and Engineering, Cambridge, MA.

Coated conductors using epitaxial thin films of $YBa_2Cu_3O_{7-x}$ (YBCO) high temperature superconductor will only be widely used in large-scale electrical devices if the YBCO can be grown relatively fast on different substrates. Films of YBCO were prepared on lanthanum aluminate (LAO) single crystals and $CeO_2$-buffered YSZ single crystal substrates by metal organic deposition (MOD) process. MOD has many advantages over different vacuum techniques because it can be used to deposit chemically uniform films on large areas and exhibits relatively high growth speeds of films. A difficulty with MOD process is, however, the first low temperature anneal when liquid solution deposited on a substrate is converted to an oxyfluoride film. A new method was developed that dramatically decreases the low temperature anneal time from hours to approximately 15 minutes. This method was used for the deposition of 0.8-micron thick films. Films prepared by this method have strong c-axis texture and exhibit good electrical properties and Jc of 1 MA/cm$^2$. An additional decrease of conversion time of YBCO films can be reached by using low vacuum during high temperature conversion. Low vacuum furnace was build for those purposes. Conversion of films in vacuum speeds the conversion due to the elimination of any mass transfer limitations in the gas phase. Production of high critical current density films required that the water partial pressure be carefully controlled during reaction. This method was successfully applied. Films that carried more that 1 MA/cm$^2$ were prepared by using that method.

**E8.9**
(110)-ORIENTED GRAIN BOUNDARIES IN $YBa_2Cu_3O_7$-BASED COATED CONDUCTORS. B.J. Gibbons, H. Kung, Q.X. Jia, R.J. Hammon, A.A. Zurek, and S.R. Foltyn, Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM.

Grain boundaries in $YBa_2Cu_3O_7$ (YBCO)-based coated conductors can significantly affect the current transport properties; therefore, the study of such boundaries and their interactions with flux-bearing vortices is critical. It has been shown for [001] tilt boundaries with (100) grain boundary planes that above 5° - 7° misorientation, the boundary dislocations begin to close off the supercurrent transport in the conductor. We have shown previously that small-angle [001] tilt boundaries with (110) grain boundary planes exhibit partial grain boundary dislocations separated by stacking faults. Interestingly, these grain boundary structures have twice the number of dislocations and shorter core channel widths than Frank's geometrical rule predicts. In order to study the latter effect on controlled samples, single (110)-oriented grain boundaries were fabricated on $Y_2O_3$-$ZrO_2$ (YSZ) bicrystal substrates. Pulsed laser deposition was used to deposit ≈ 300 nm of YBCO on ≈ 20 nm of $CeO_2$ on these bicrystals with boundary angles from 0° to 45°. Transport property measurements, as well as results of a transmission electron microscopy study will be presented.

**E8.10**
ENHANCEMENT OF TWIN REFINEMENT FOR HIGH CRITICAL CURRENT DENSITY AND STRONG FLUX PINNING IN YBCO BY QUENCHING AND MELT GROWTH METHOD. Linfeng Mei, Oratai Jongprateep, Siu-Wai Chan, Columbia University, Department of Applied Physics and Applied Mathematics, New York, NY.

Earlier, we achieved twin refinement in melt-textured growth (MTG) YBCO by combining the effect of $PtO_2$ doping and increase of isothermal annealing temperature from 450°C to 680°C. By increasing annealing temperature, the twin boundaries in MTG YBCO are greatly refined - from twin spacing of 1900 $\AA$ at annealing temperature of 450°C to 540 $\AA$ at annealing temperature of 680°C. The superconducting properties significantly improved: critical current density $J_c$ and maximum flux pinning force $F_c$ can be enhanced by 200% –from $J_c$ of 9,800 A/cm$^2$ to 32,000 A/cm$^2$ at 77 K and 1 T, and from $F_c$ of $1.17 \times 10^8$ N/m$^3$ to $3.35 \times 10^8$ N/m$^3$. Recent study has shown that processing of YBCO by quenching and melt growth QMG method can further enhance twin refinement and increase superconducting properties. Twin structures studied by TEM reveal that, for samples annealed at the same temperature, twin boundaries are more refined in QMG than those of MTG. The twin spacing is 10% - 20% lower in QMG samples. The maximum flux pinning force can be enhanced by 100% in QMG samples. The critical current density is also improved from 12,500 A/cm$^2$ for MTG sample oxygenated at 500°C to 25,390 A/ cm$^2$ for QMG oxygenated at the same temperature.

**E8.11**
THEORETICAL MODELS OF THE THERMODYNAMICS OF INTERMEDIATE PHASES IN THE $BaF_2$ PROCESS FOR SYNTHESIS OF $YBa_2Cu_3O_7$ THICK FILMS. H.B. Su and D.O. Welch, Brookhaven National Laboratory, Materials and Chemical Sciences Division, Upton, NY.

The so-called $BaF_2$ process for $YBa_2Cu_3O_7$ coated conductor fabrication shows great promise, but still obstacles remain to fabricating 5$\mu$m thick films because of the onset of formation on non-c-axis-aligned crystallites. To understand the mechanisms involved requires an understanding of the thermodynamics of the (Y,Ba) oxy-fluoride and (Y,Ba)$CuO_x$ (the "T phase") transitional phases which have recently been discovered by detailed studies at BNL of the $BaF_2$ process using transmission electron microscopy. Shell models of interatomic bonding were used to calculate lattice energies, Debye temperatures, and other parameters required to estimate the Gibbs free energies of the oxy-fluoride and T phases as a function of temperature as well as fluoride and oxygen partial pressures. The relative stability of the transitional phases and its effect on the nucleation of c-axis or a-axis YBCO crystallite will be discussed.

**E8.12**
FLUX-PINNING OF $YBa_2Cu_3O_{7-x}/Y_2BaCuO_5$ MULTILAYER THIN FILM STRUCTURES PROCESSED BY PULSED LASER DEPOSITION. Timothy Haugan, Brent Cobb, Paul Barnes, Iman Maartense, Lyle Brunke, Air Force Research Laboratory, Wright-Patterson AFB, OH.

A multilayer thin film structure of $YBa_2Cu_3O_7$ (123) superconductor interleaved with non-superconducting $Y_2BaCuO_5$ (211) phase was studied for the purpose of increasing flux-pinning of the $YBa_2Cu_3O_{7-x}$ superconductor. Initial results showed significant improvement for Jc in applied magnetic fields, e.g. over 100% higher at 0.5-2 T, 70 K. A multi-layer structure of 35 layers of {∼80 angstrom $YBa_2Cu_3O_{7-x}$ / ∼12 angstrom $Y_2BaCuO_5$} was fabricated by (248 nm) pulsed laser deposition at 300 mTorr O2 pressure and 785°C substrate temperature. This multilayer structure showed dramatic improvement in flux-pinning at 4 to 77 K. However, for the unoptimized process, the Tc of the composite was reduced to ∼87 K from 92 K for pure 123 phase. This multilayer film showed no cracking and unusual surface smoothness, probably as a consequence of the multilayer growth process. The significance of these results for long length applications of coated conductors will be discussed.

**E8.13**
ORIENTATION AND STRAIN MAPPING IN RABiTS USING X-RAY MICROBEAM DIFFRACTION. John Budai, David Norton, Gene Ice, Bennett Larson, Jon Tischler, Wenge Yang, Chan Park, Oak Ridge National Laboratory, Oak Ridge, TN; Walter Lowe, Howard University, Washington, DC.

We have used a newly developed x-ray microbeam technique to investigate the crystallographic alignment (texture) and strain in each layer of RABiTS samples. Polychromatic x-rays were focussed to <1$\mu$m diameter using elliptical Kirkpatrick-Baez mirrors at the Advanced Photon Source synchrotron, MHATT-CAT beamline. Since x-rays penetrate all layers, white microbeam diffraction simultaneously provides CCD Laue patterns from each layer of the multilayer RABiTS samples. Analysis of these patterns yields real-space maps of the local lattice structure, crystallographic orientation, and the strain tensor with micron-spatial resolution. The advantages and disadvantages of x-ray microbeam patterns will be compared with electron backscattered patterns (EBSP). Our orientation results show that that successive deposited layers are not strictly epitaxial; rather, each heteroepitaxial layer exhibits a tilting toward the surface normal. The tilting behavior of $CeO_2$ buffer layers at typical growth temperatures can be described by a ledge growth model which incorporates both elastic deformation at steps and interfacial misfit dislocations. Note that tilting is desirable in applications since it decreases the grain boundary misorientation angles and enhances Jc. Strain results show that the microstrain, like the macrostrain, is biaxial and is consistent with differential thermal contraction.
Research sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC, MHATT-CAT beamline funded by the U.S. Department of Energy, Basic Energy Sciences.

**E8.14**
FABRICATION OF SUPERCONDUCTING $YBa_2Cu_3O_y$ THIN FILMS ON YSZ SUBSTRATES WITH AND WITHOUT A $Eu_2CuO_4$ BUFFER LAYER. C.Y. Ben Yau[a,b], C.D. Beling[b], J. Gao[b], R.S. Newrock[a], [a]Department of Physics, University of Cincinnati, Cincinnati, OH; [b]Department of Physics, University of Hong Kong, Hong Kong, CHINA.

A careful study of $Eu_2CuO_4$ (ECO) thin films indicates that the use of ECO as an insulating buffer layer would result in a dramatic improvement of epitaxy, surface quality and interface condition of superconducting $YBa_2Cu_3O_7$ (YBCO) thin films grown on Yttrium-stabilized $ZrO_2$ (YSZ) (001) substrates. ECO films are highly c-axis oriented and well-crystallized, as shown by X-ray diffraction $\theta$ - $2\theta$ scan and rocking curves FWHM ∼0.25°. Buffered YBCO thin films are superconducting at a $T_c$ = 88 K with improved crystallinity, as observed from X-ray diffraction of different samples. Moreover, the buffered superconducting thin films possess an extremely smooth surface (average roughness less than 1∼1.5 nm) and a Stranski-Krastanov growth mode was observed under atomic force microscopy. Also, the interfaces of the buffered YBCO thin films on YSZ substrates are improved, as shown by positron data obtained by positron annihilation spectroscopy which is a non-destructive method to probe the bulk and the interface of semiconducting or superconducting thin films and makes use of the positron-electron annihilation of positron beams with varied implantation energy.

**E8.15**
VAPOR PRESSURE AND THERMODYNAMICS OF HIGH $T_c$ SYSTEMS. Lawrence Cook, Ralph Klein, and Winnie Wong-Ng, NIST, Gaithersburg, MD.

Volatile components present in high $T_c$ systems and their precursors include $O_2$, $CO_2$, $F_2$, PbO, and Mg. The role of oxygen is well known in the oxidation/reduction of YBCO and related superconductors, and oxygen also plays a role in the processing of BSCCO materials. For some high $T_c$ processing routes, alkaline-earth carbonates are used as precursors, resulting in production of $CO_2$. For other processing routes, the use of fluoride precursors requires consideration of fluoride-containing species such as HF, when water is added to the system. For BSCCO materials, where Pb is added to optimize

processing yield, volatility of PbO can lead to changes in stoichiometry. Most recently, interest in $MgB_2$-based superconductors has led to an appreciation of the importance of Mg partial pressure in the processing of these materials. For all such systems, it is important to consider phase equilibria as a function of the appropriate partial pressures. Equilibrium partial pressures over selected systems have been measured in our laboratory using Knudsen effusion thermogravimetry. We have also used solution calorimetry to measure selected heats of formation, which can be directly related to vapor pressures. These data provide an important handle in dealing with the thermodynamics of complex high $T_c$ systems, for which generation of an integrated thermodynamic/phase equilibrium database is our goal.

**E8.16**
PHASE RELATIONS IN THE Ba-R-Cu-O (R = Nd, Sm, AND Y) SYSTEMS. Winnie Wong-Ng, Julia Suh and Lawrence Cook, Ceramics Division, NIST, Gaithersburg, MD.

Phase diagrams provide guidelines for processing of high-temperature superconductors. There is a need for phase diagrams for the second-generation coated conductors prepared under reduced conditions. These flexible coated conductors are based on the barium yttrium copper oxide and the lanthanide substituted materials. The phase diagrams of the carbonate-free Ba-Nd-Cu-O, Ba-Sm-Cu-O, and the Ba-Y-Cu-O systems were constructed under atmospherically-controlled conditions (purified air and 0.1% $O_2$). A special experimental procedure was used for preparing BaO starting material and for the handling and heat-treatment of samples. A discussion of the crystal chemistry and phase relationships of phases found in these systems will be presented. A comprehensive comparison of selected diagrams prepared under different atmospheric conditions will be summarized.

**E8.17**
THE EFFECT OF PHOTODOPING ON THE LOCAL STRUCTURE OF YBCO(y 0.3, 0.94) FILMS. A.Yu. Ignatov, T.A. Tyson, L.M. Dieng, NJIT, Dept. of Physics, Newark, NJ.

We report on c-axis polarized Cu K-edge x-ray absorption measurements of YBa2Cu3O6 y films (y 0.3 and 0.94). Optimally doped sample (y 0.94) do not show any appreciable change in its local structure at photon doses up to 1022 cm-2. In contrast, XANES of the underdoped sample (y 0.3) reveals marked changes that are reminiscent of those found with oxygen doping. Cu K-edge multiple-scattering simulations of YBa2Cu3O6.5 suggest that the observed experimental behavior can be understand assuming local ordering of O(1) atoms in the chains. We also studied relaxation of photodoped states by measuring resistance and XANES spectra vs. time after photodoping. Changes of major XANES features could be scaled to the resistance. EXAFS analysis indicate that, on average, the Cu(1)-O(4) and Cu(2)-O(4) distributions tend to be broader in photodoped state. Fits to the experimental data suggest a double-peak Cu1-O1 distribution with a typical separation between the peaks of 0.15 A. Though the c-axis measurements do not probe the electronic and local structure of the Cu1-O11 y planes directly, EXAFS data are in line with a microscopic model suggesting partial photoinduced diffusion of O1 atoms from a mostly disordered uniform arrangement to a locally ordered states where they group into short-range chains of [O1-Cu1-O1 ]n. Preliminary Cu K-edge DAFS results, enabling local probe around Cu(1) sites, will be presented. This work is supported by NSF Career Grant DMR-9733862.

**E8.18**
HOW TO OPTIMIZE CRITICAL CURRENT PERFORMANCE OF RE123 MATERIALS BY CONTROLLING OXYGEN CONTENT. Jun-ichi Shimoyama, Shigeru Horii, Kenji Otzschi and Kohji Kishio, Department of Superconductivity, University of Tokyo, Tokyo, JAPAN.

In order to optimize the critical current performance of RE123 materials, such as coated conductors and bulk magnets, post-annealing under suitable conditions is essentially important, because resulting oxygen defect concentration determines $T_c$, pinning sites and electromagnetic anisotropy. Oxygen nonstoichiometry of RE123 compounds had been eagerly studied before 1990, however, more quantitative information has been required for the recent RE123 materials. Therefore, in the present study, oxygen nonstoichiometry of RE123 compounds with various RE elements was precisely investigated as functions of temperature and $P(O_2)$, and dependence of their superconducting properties, such as $T_c$, irreversibility field and $J_c$, on oxygen content was systematically studied. Thermo-gravimetric measurements of the RE123 sintered bulk specimen were performed using an electro-micro-balance. Thermodynamic quantities, $h_O$ and $s_O$, were calculated from the nonstoichiometry data. Superconducting properties of sintered bulk, melt-solidified bulk and single crystals with various oxygen contents were measured by using a SQUID magnetometer. Oxygen nonstoichimetry behavior was found to slightly depend on the RE element and it was largest in Y123. $h_O$ and $s_O$ were also dependent on RE element. The highest $T_c$ was found at similar oxygen content, however, the equilibrium conditions, temperature and $P(O_2)$, were different in each RE123. Dramatic difference was found in $T_c$ variation with oxygen content at slightly carrier underdoped region. Oxygen content range which gives $T_c \geq 90$K was systematically increased with a decrease of ionic size of RE, while the highest $T_c$ monotonically decreases. This difference was found to affect the flux pinning behaviors in melt-solidified bulk and single crystals. These results strongly suggested that the post-annealing conditions of RE123 materials should be optimized independently for improving critical current performance in each system.

**E8.19**
EFFECT OF HIGH MAGNETIC FIELD DURING MELT-SOLIDIFICATION PROCESS ON CRYSTAL ALIGNMENT OF REBCO BULK. R. Yamagiwa, S. Horii, H. Eto, K. Otzschi, J. Shimoyama, K. Kishio, Dept. of Superconductivity, Univ. of Tokyo, Tokyo, JAPAN.

Recently, several papers reported that application of high magnetic fields during the crystal growth process is effective for improving the crystallinity of RE-Ba-Cu-O (REBCO) melt-solidified bulk. However, most of those studies were made on the epitaxially grown large domain from the top seeded crystal and, therefore, essential effect of magnetic field on crystal orientation and crystallinity of REBCO has not been well clarified yet. In the present study, we have synthesized REBCO ( RE = Y, Nd, Sm, Gd, Dy, Ho, Er ) bulk samples by the melt-solidification method without seeding under various magnetic fields, in order to evaluate the crystal alignment effect of applied fields which is believed to originate from anisotropic magnetic moment of REBCO. Disk-shaped samples with 10mm $\phi$ x 10mm were fabricated by slow-cooling from peritectic temperature at a rate of 5°C/h under various magnetic fields ($H_a$) up to 8T. Crystal alignment was evaluated by the X-ray diffraction, magnetization measurement using a SQUID magnetometer and observation of microstructure. In the case of YBCO, apparent effect of applied fields was not found in the domain size of the samples. Average domain size was approximately 1.5 x 1.5 x 1.5$mm^3$. Magnetization hysteresis width, $\Delta M_{H \parallel a}$ (85K, 5kOe), of YBCO was almost constant up to $\mu_0 H_a$ = 6T, while $\Delta M_{\perp a}$ drastically decreased with magnetic fields below $\mu_0 H_a$ = 4T and kept small values for samples with higher $H_a$. The ratio, $\Delta M_{\parallel a} / \Delta M_{\perp a}$, monotonically increased with an increase of $H_a$. Relationship between anisotropic magnetic moment of RE ion in the crystal and alignment effect by applied magnetic fields will be systematically discussed.

**E8.20**
SELF OXIDIZED Ni AND Ni ALLOYS AS TEMPLATE FOR HTS. Cirsten Brandt, Sibylle Sievers, Jörg Hoffmann, Herbert C. Freyhardt, Universitaet Goettingen, GERMANY.

To prepare long HTS-coated tapes a highly textured buffer layer is necessary. The buffer layer should suppress the diffusion of the metal-ions of the substrate into the superconductor and serve as a template for the superconductor. The RABiTS process is a standard method to produce long-length pretextured Ni or Ni-alloy substrate tapes. Typically oxides like YSZ, $CeO_2$ or MgO are deposited as a buffer layer. As an alternative procedure buffer layers can be prepared by self oxide epitaxy (SOE). Pretextured Ni and Ni-alloys were oxidized to generate epitaxial and highly textured NiO on RABiTS tapes. In this contribution growth mechanisms of the NiO were investigated. Therefore, we oxidized Ni, NiCr, and NiV RABiTS tapes as well as the corresponding single crystalline [001]< 100 > films at different temperatures and oxygen pressures. Two possible out-of-plane orientations were observed. Beside the desired NiO(100) also NiO(111) could be detected. Obviously the particular kinetics determines, which orientation is favored. Both orientations are biaxially textured. NiO(100) grows in-plane with two different epitaxial relations, the 0° and the 45° epitaxy. Which of these orientations is dominant can be controlled by the oxygen pressure. Furthermore, the unexpected occurence of $\mu$m-sized quadratic holes in the oxidized Ni films will be discussed.

**E8.21**
TEXTURE BY THE KILOMETER. E.D. Specht, F.A. List, Oak Ridge National Lab, Oak Ridge, TN.

An x-ray diffraction system is described for the analysis of tapes up to 20 m in length. The tape is spooled from reel to reel while mounted on a four-circle diffractometer, allowing rocking curves, phi scans, theta / 2-theta scans and pole figures to be collected at any point on the tape. Examples are presented for superconducting films grown epitaxially on biaxially textured substrates, including quality assurance, designed experiments, and location of defects. Work sponsored by the Division of Materials Sciences and by the Office of

Energy Efficiency and Renewable Energy, Office of Utility Technology-Superconductivity Program. Oak Ridge National Laboratory (ORNL) is operated by UT-Battelle, LLC, for the U.S. Department of Energy under contract DE-AC05-00OR22725.

**E8.22**
SPATIAL DISTRIBUTION ANALYSIS OF SUPERCONDUCTING TRANSITION TEMPERATURE IN EPITAXIAL $YBa_2Cu_3O_7$ FILM USING VARIABLE TEMPERATURE SCANNING LASER MICROSCOPY. S. Seo and C. Kwon, Department of Physics and Astronomy, California State University-Long Beach, Long Beach, CA; B.H. Park and Q.X. Jia, Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM.

We have investigated spatial distribution of superconducting properties using Variable Temperature Scanning Laser Microscope (VTSLM). The scanning laser microscope is "a hot spot" scanning technique where a laser beam is scanned across a sample to create a local heating, and the voltage response, $\delta$V, due to the laser beam is measured from the sample using a lock-in amplifier with respect to the position of the beam. We used an epitaxial $YBa_2Cu_3O_7$ film photolithographically patterned to a 300 $\mu$m-wide and 2.0 mm-long bridge. Since the voltage response, $\delta$V(x,y) is proportional to dR/dT(x,y), we obtain the spatial distribution of superconducting transition in VTSLM images. In the resistive transition region, we find a strong correlation between the VTSLM images and the resistance of the sample. With decreasing resistance, the area with large $\delta$V(x,y) shifts towards the ends of the bridge. This indicates that the resistive transition is not uniform and both ends of the bridge have lower transition temperature ($T_c$). VTSLM images taken below superconducting transition temperature and near the critical current ($I_c$) show that the resistive areas appear initially at the both ends of the bridge. VTSLM technique enables us to image the local superconducting properties and to identify the weaker superconducting areas. The effects of laser output power, bias currents, and thermal linkage between the sample and the sample block will also be discussed.

**E8.23**
EFFECTS OF ION BEAMS ON THE EARLY STAGES OF MgO GROWTH. Luis A. Zepeda-Ruiz and D.J. Srolovitz, Princeton Materials Institute & Department of Mechanical and Aerospace Engineering, Princeton University, Princeton, NJ.

Biaxially textured MgO grown on amorphous substrates is technologically important because of its potential applications as a heteroepitaxial template for high current superconducting films. The necessary degree of texture is accomplished by using ion beam assisted deposition (IBAD) to create polycrystalline films with a preferred in-plane and out-of-plane grain orientation. There is a substantial body of evidence that suggests that much of the in-plane texture is established prior to island coalescence. In this presentation, we examine the effects of an ion beam in selecting MgO island orientation. We performed three-dimensional molecular dynamics simulations to examine the sputter yield and in-plane texture as a function of ion beam orientation for small MgO islands. While channeling does occur in small islands, the difference in sputter yield between channeling and non-channeling orientations is much smaller in small islands than in larger islands or a continuous film. The ion beam is capable of disrupting the entire island structure at small island sizes, while channeling dependent sputtering occurs at larger island size. The main role of the ion beam at small island sizes is to effectively modify the critical island size for growth. Since less sputtering occurs for channeling orientations, this yields an orientation-dependent stable island nucleation rate. We have developed an analytical model for this effect that is parameterized in terms of our molecular dynamics sputter yield data. This model predicts the evolution of texture by preferred island nucleation.

**E8.24**
PHASE DIAGRAM STUDIES IN THE SrO-CuO-$Ti_2O_3$ SYSTEM; APPLICATIONS TO YBCO COATED CONDUCTORS. A. Ayala, T.G. Holesinger, E.J. Peterson, M. Archuleta, Superconductivity Technology Center, Los Alamos National Laboratory.

$SrTiO_3$ (STO) is a candidate buffer layer material for use in YBCO coated conductors. However, the interactions with YBCO are not yet fully understood and little information exists in the way of phase diagrams. With this in mind, the phase diagram for the pseudo-ternary system SrO-CuO-$Ti_2O_3$ was investigated with an emphasis on the region surrounding STO. Solid state and solution methods were used to prepare bulk samples. Solubility limits and phase assemblages were determined by x-ray diffraction and electron microscopy in the temperature range of 1000°C to 1100°C in oxygen partial pressures of 1%, 10%, and 100%. The influence of the phase diagram on the processing of coated conductors will be discussed.

**E8.25**
X-RAY CHARACTERIZATION YBCO WITH VARIOUS CRITICAL PARAMETERS. B.N. Kodess, I.P. Jouravlev, E.M. Roysenblat, Dept. Mater. Science, ICS&E at Denver, CO; Department of Crystals Metrology, VNIIMS, Moscow, RUSSIA.

The investigation of structural parameters of YBCO-ceramics with the various contents of oxygen has been carried out. The interplay between sample structural parameters and critical parameters of a superconducting state (Tc) is analyzed at various levels of a structure - microstructural and atomic, and electronic. The characterization has been made with new iterative procedure of determination of Braggs' profiles parameters, taking into account extremely complicated diffraction pattern YBCO (many overlapping peaks, especially at high angles). The application of this technique (which we used earlier for refinement of single crystal structural parameters) to polycrystalline samples allowed to increase number of separated peaks up to about 220 for CuK alpha-radiation and to reveal the angular dependency of full width at half maximum of profile (FWHM), including reflections of various orders. The multistage refinement allowed to determine peaks positions (connected with change of staking faults, vacancion sub-lattice and macro-stresses contribution), ratio of integrated intensities (the long-range and short-range order) and FWHM (connected with mosaic block and defects of a various type contribution) with a high accuracy. The parameters of time stability are determined using Reference Material Standard of superconducting ceramics of composition and properties. More detail analysis of a rhombic phase diffraction pattern has been made for composition with maximal Tc {YBa2Cu3O6.93; a=3,8188, b=3,8851, c=11,6781}, which has a greatly of overlapping peaks because of close value of the triple parameter b and parameter c. Also the data were collected for composition YBa2Cu3O6.33 which has overlapping peaks caused by approximately equal lattice parameters a and b.

**E8.26**
Abstract Withdrawn.

**E8.27**
LOCAL STRUCTURE OF SUPERCONDUCTING MATERIALS: HALOGENATED YBCO AND $MgC_xNi_3$. L.M. Dieng, A.A. Ignatov, T.A. Tyson, NJIT, Dept. of Physics, Newark, NJ; M. Croft, Dept. of Physics, Rutgers University, Piscataway, NJ; R.J. Cava, Dept. of Chemistry and Princeton Materials Institute, Princeton University, Princeton, NJ.

The recovery of superconductivity by chlorination, iodination and bromination was investigated in underdoped single crystal YBCO. The spatially resolved local structural changes in halogenated samples have been measured by x-ray absorption spectroscopy (XAFS) and compared to those in normal crystals. The local structure about the Y, Ba, Cu and Br/Cl/I sites was determined. Evidence for charge transfer was derived from the near edge spectra (XANES). We address the issue of the incorporation of Br/Cl/I into the lattice and develop structural models for the recovery of superconductivity. We also investigated the local atomic and electronic structure on the new perovskite superconductor $MgC_xNi_3$ in order to examine the role of local distortions. This research is supported by National Science Foundation Career Grant DMR-9733862.

**E8.28**
$LaTiO_3$ CONDUCTIVE BUFFER LAYERS ON BIAXIALLY TEXTURED Cu FOR COATED CONDUCTORS. Kyunghoon Kim, Mitesh Patel, and David Norton, University of Florida, Department of MS&E, Gainesville, FL; John Budai, Brian Sales, Matthew Chisholm, Aytug Tolga, Claudia Cantoni, and David Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

In recent years, significant effort has focused on HTS coated conductors based on Rolling Assisted Biaxially Textured Substrates (RABiTS). For a functional conductor, it is likely important to provide a suitable conducting shunt for traversing current around localized breaks in the superconducting film. With metallic substrates, shorting the superconducting film to the substrate via a conducting buffer layer is one approach being considered. While much of the interest has focused on Ni and Ni-based alloys, other biaxially textured metals present intriguing advantages. In particular, copper is attractive as it is non-magnetic and exhibits a higher conductivity than Ni at 77 K, thus providing better fault protection for similar substrate thickness. Unfortunately, little is known about the epitaxial growth of oxides on Cu. Epitaxial growth of oxides on Cu present major challenges due to the large difference in thermal expansion coefficient as compared to superconducting oxides. In this talk, we will describe the growth of $LaTiO_3$, which is a metallic pseudo-perovskite, as a suitable conductive buffer layer on Ni and Cu for $YBa_2Cu_3O_7$ growth. Oxide film growth is explored using reactive sputter deposition and pulsed laser deposition. Characterization of the films will include X-ray diffraction and scanning electron microscopy.

In addition, we will describe properties of superconducting films deposited on the buffered substrates.

**E8.29**
Abstract Withdrawn.

**E8.30**
LIQUID PHASE AND BUFFER LAYER CONSIDERATION FOR HIGH RATE IN-SITU YBCO DEPOSITION. Tsuyoshi Ohnishi, William Jo, Ann Marshall, Jeong-uk Huh, Robert H. Hammond, and M.R. Beasley, Stanford University, Geballe Laboratory for Advanced Materials, Stanford, CA; Richard E. Ericson, 3M Corporation, St. Paul, NM.

We are developing a high rate (up to 10 nm/s), in-situ YBCO film synthesis process using an electron beam deposition method with a view toward coated conductors for electrical power applications. It seems that Ba-Cu-O liquid flux is important to grow not only bulk crystal but also film form of ReBCO [1]. We have also confirmed the effectiveness of the liquid phase to grow high Jc YBCO in our high rate in-situ deposition. In this talk, optimization of the liquid phase, i.e. composition and formation temperature, based on thermodynamic stability and lowering melting point with doping will be presented. Growth of buffer layers suitable for the liquid phase assisted film growth on artificial textured metal tapes will be also discussed. [1] M. Kawasaki, D.B. Choi, T. Ito, K.S. Yun, and H. Koinuma, Proceedings of the third symposium on atomic scale surface and interface dynamics. 3, (1999) 151.

**E8.31**
INVESTIGATION OF MAGNETIZATION LOOPS IN ULTRATHIN YBCO FILMS. Lyuba Delimova, Ivan Liniichuk, Ioffe Inst, Dept of Solid State Electronics, St. Petersburg, RUSSIA; Erkki Lähderanta, Univ of Turku, Wihuri Phys Lab, Univ of Vaasa, Dept of Physics, FINLAND; Konstantin Traito, Ioffe Inst, Dept of Solid State Physics, St. Petersburg, RUSSIA.

The interest in fabricating high-quality ultrathin YBCO films is associated with their fundamental HTSC properties and possible microwave and microelectronic applications. The investigation of magnetic hysteresis loops in HTSC materials is a widely used tool to estimate the critical current density and its dependence on magnetic field, yielding information about superconducting properties relevant to applications. We study magnetization loops in strip-shaped YBCO films with a thickness about 10 nm, deposited on a $SrTiO_3$ substrate with and without an YBaCuNbO buffer layer. The zero resistance critical temperatures of the films studied are $T_c$=74,79,87 K. The magnetization loops are measured in magnetic fields up to B=1 T applied perpendicular to the film surface in the temperature range of 2.5-50 K. The critical current determined from the opening of the hysteresis loop is on the order of $(1-2)\cdot 10^6 A/cm^2$ at 2.5 K and B=0. It is field independent up to 0.1 T and decreases down to $\approx 10^4 A/cm^2$ at 1 T. Magnetization M(B) of all the samples is found to show a sharp minimum (dip) around zero magnetic field. The dip width of 0.1 mT is temperature independent. The anomaly of M(B) behavior occurs in the same field range where an anomaly of ac surface impedance Z(B) of thin YBCO films has been found recently. In the framework of the 2D limit of the critical state model, this dip of M(B) can be associated with anomalous weak links having a non-Fraunhofer-like field dependence of the critical current.

**E8.32**
ENHANCEMENT OF FLUX PINNING IN MELT TEXTURED $Y_{1-x}Nd_xBa_2Cu_3O_{7-y}$ SUPERCONDUCTORS. Guiwen Qiao, Yutao Xing, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, CHINA.

The textured $Y_{1-x}Nd_xBa_2Cu_3O_{7-y}$ superconducting samples have been fabricated by the modified MTG method. XRD analysis shows that the samples have good orientation along the (001) direction. Nd substituted Y in the samples homogeneously and it has little effects on the samples microstructures. The field dependence of the irreversibility temperature in various MTG $Y_{1-x}Nd_xBa_2Cu_3O_{7-y}$ samples at 5k was investigated. The ZFC and FC M-T curves were measured by a dc SQUID magnetometer. It was found that the magnetization of FC curve is very small compared with the ZFC curve. This means the pinning forces acting on the flux are very strong. The irreversibility line of the samples show that small amount of Nd substitution for Y can enhance the flux pinning properties of these YBCO superconductors especially in higher fields. The irreversibility temperature of the sample with the substitution amount of 0.01 is increased by 8k compared with the sample of zero substitution in high fields; in very low fields, the Tir is almost the same. We believe that the substituted areas have poor superconducting property with the matrix. When the substitution amount is small, it has little harmful effects to the sample. In large fields, the few substituted areas in the sample lost their superconductivity and then they can act as flux pinning centers in the sample. So introducing a second phase, which has the same crystal structure with the matrix into high Tc superconductors may be a prospective way to improve their flux pinning properties.

**E8.33**
Abstract Withdrawn.

**E8.34**
MECHANICAL PROPERTIES OF CUBE TEXTURED Ni/Ni-1.5at%Al COMPOSITE TAPES FOR HTS COATING APPLICATIONS. V.S. Sarma, Bernd de Boer, Bernhard Holzapfel, Institute for Solid State and Materials Research, Dresden, GERMANY.

The development of mechanically stronger(~500 MPa yield strength) highly cube textured substrates is of great technological importance for increasing the engineering current density of the coated HTS conductor tapes. Nickel substrates are thought to be suitable candidates for this in view of their excellent oxidation resistance and ability to form strong cube texture after heavy rolling and annealing. However nickel is very soft (yield strength ~40 MPa). The mechanical properties can be increased through solid solution and precipitation strengthening methods. However the addition of alloying elements in larger amounts (eg: Cr, V, Al ) required for solid solution strengthening results in deterioration of cube texture. A more promising method to improve strength is through internal oxidation of Ni-Al alloy. Results on the internally oxidised Ni-1at%Al and Ni-1.5at%Al alloys show that yield strength levels of the order of 200 MPa can be achieved through this method in 40$\mu$m tapes. To avoid the formation of $Al_2O_3$ at the surface which is detrimental for further deposition, Ni-Ni/Ni-1.5at% Al composite tapes are prepared by rod in tube hot extrusion and further hot and cold rolling to 40 and 80$\mu$m thickness. The tapes were recrystallized and texture was investigated. Further the tapes will be internally oxidized to obtain $Al_2O_3$ precipitation for increasing the strength. The present paper reports the texture development and mechanical properties of these composite tapes.

**E8.35**
IMAGING TRANSPORT CURRENT DISTRIBUTION IN HIGH TEMPERATURE SUPERCONDUCTORS USING ROOM TEMPERATURE SCANNING LASER MICROSCOPE. C. Kwon, S. Seo, and B.E. Klein, Department of Physics and Astronomy, California State University-Long Beach, Long Beach, CA; B.H. Park and Q.X. Jia, Superconductivity Technology Center, Los Alamos National Laboratory, Los Alamos, NM.

We report the feasibility of room temperature scanning laser microscopy (RTSLM) for the study of high temperature superconducting films. The scanning laser microscope is "a hot spot" scanning technique where a laser beam is scanned across a sample to create local heating, and the voltage response, $\delta V(x,y)$, due to the laser beam is measured from the sample using a lock-in amplifier with respect to the position of the beam. RTSLM images from $SmBa_2Cu_3O_7$ and $YBa_2Cu_3O_7$ thin films show that the ac-voltage response exists only in the section of the bridge where the transport current produces a voltage drop. A photolithographically defined 60 $\mu$m x 60 $\mu$m void in a 300 $\mu$m-wide bridge was clearly visible in a RTSLM image giving the spatial resolution smaller than 60 $\mu$m. In addition, the void disturbs the transport current distribution beyond itself generating an elongated shape void of 64 $\mu$m x 85 $\mu$m with the longer side along the direction of current flow in the RTSLM image. Our results indicate that the RTSLM is a useful tool to investigate the transport current distribution in high temperature superconductors.

**E8.36**
THE EFFECT OF MAGNETIC FIELD ON THE ANOMALOUS MICROWAVE Q OF YBCO-ON-MgO RESONATORS. S.H. Park, Department of Electrical Engineering and Computer Sciences, MIT, Cambridge, MA and Air Force Research Laboratory, Hanscom AFB, Bedford, MA; D.E. Oates, MIT Lincoln Laboratory, Lexington, MA; J.S. Derov, Air Force Research Laboratory, Hanscom AFB, Bedford, MA; G. Dresselhaus, Department of Physics, MIT, Cambridge, MA and Air Force Research Laboratory, Hanscom AFB, Bedford, MA; M. Dresselhaus, Department of Physics, MIT, Cambridge, MA.

Several reports of anomalous microwave surface impedance in YBCO thin films have appeared in the literature. The anomaly consists of a decrease in both the real and imaginary parts of the surface impedance as either the microwave power or the dc magnetic field is increased. We have previously observed an increase of Q as the microwave power is increased for both YBCO and niobium resonators on MgO substrates at low temperatures. This effect closely resembles the reported anomalous surface impedance. In this study, we measured the Q vs microwave power in a resonator as a function of dc magnetic field up to 0.1 T for temperatures between 5 and 70 K. The

measurements were done at a frequency of 3.7 GHz in a suspended microstrip resonator. The external DC magnetic field was perpendicular to the Cu-O plane of the YBCO film. The power was varied from -55 to 18 dBm. The anomalous Q behavior was not affected by the magnetic field. The anomaly was much less pronounced at 20 K than at 5 K and completely gone at 70 K. The anomalous Q behavior was absent on sapphire and lanthanum aluminate substrates. We conclude that a low-temperature nonlinear dielectric loss in the MgO is the source of the anomalous Q. The absence of any magnetic field dependence rules out magnetic origins. We will discuss the various mechanisms that could explain this previously unobserved nonlinear dielectric loss.
This work was supported by the Air Force Office of Scientific Research.

**E8.37**
DEVELOPMENT OF HTS RF DEVICES USING SINGLE-DOMAIN YBCO FOR WIRELESS TELECOMMUNICATIONS. Donglu Shi, Yongli Xu, Altan Ferendeci and David Mast, Dept. of MS&E, Dept. of Electrical & Computer Engineering and Computer Science, and Dept. of Physics, University of Cincinnati, Cincinnati, OH.

Using a novel laser technique, unique RF devices have been designed and developed using single-domain YBCO for wireless telecommunications. The uniqueness of these devices lies on the 3-D architecture of the single-domain structures. In contrast to the thin film devices where the structures are limited 2-D patterns, the newly designed RF cavities and circuitry utilize the processing freedom in the development of 3-D structures. These advantages will enable the design of lightweight, tunable, and compact RF devices in base station filters. In this presentation we will show current experimental results in single-domain YBCO growth, device design, and RF property characterization.

SESSION E9: YBCO COATED CONDUCTORS III
Chairs: Martin W. Rupich and Victor A. Maroni
Thursday Morning, November 29, 2001
Room 200 (Hynes)

**8:30 AM *E9.1**
FABRICATION BY INCLINED SUBSTRATE DEPOSITION OF BIAXIALLY TEXTURED BUFFER LAYER FOR COATED CONDUCTORS. U. Balachandran, B. Ma, M. Li, B.L. Fisher, R.E. Koritala, R. Erck, and S.E. Dorris, Energy Technology Division Argonne National Laboratory, Argonne, IL.

Inclined substrate deposition (ISD) offers the potential for rapid production of high-quality biaxially textured buffer layers suitable for YBCO-coated conductors. We have grown biaxially textured MgO films by ISD at deposition rates in the range 20-100 Å/sec. Columnar grain structures with a roofing-tile-shaped surface were observed in these films. X-ray pole figure analysis revealed that the (002) planes of these ISD-MgO films are tilted at an angle from substrate normal. A small phi-scan full-width at half maximum (FWHM) of $\approx 9^\circ$ was observed on MgO films deposited with an inclination angle of 55°. YBCO films were grown on ISD-MgO buffered Hastelloy substrates by pulsed laser deposition. We obtained a critical current density of $\approx 2 \times 10^5$ A/cm$^2$ at 77 K in self-field on 0.5-mm-thick, 0.5-cm-wide, and 1-cm-long samples. Details of ISD-MgO film fabrication and characterization, as well as the results of YBCO deposited on the ISD-MgO buffer layer, will be presented.

**9:00 AM *E9.2**
CONTINUOUS YBCO - TAPE COATING BY THERMAL EVAPORATION. Ralf Nemetschek, Werner Prusseit, THEVA GmbH, Eching-Dietersheim, GERMANY; B. Holzapfel, J. Eickemeyer, B. deBoer, IFW Dresden, GERMANY; U. Miller, Plansee AG, Reutte, AUSTRIA; E. Maher, Oxford Superconductivity plc, UNITED KINGDOM.

Currently, due to simultaneous large area coating thermal evaporation allows the highest YBCO volume deposition rates among various coating techniques. The simultaneous large area deposition translates into long length when the deposition area is closely filled up with metal tape. On short RABiTS samples thermal evaporation has already established critical current densities in excess of 1MA/cm$^2$ at 77K. Recently, the deposition technique has been scaled up using a multi-turn tape winder for a simultaneously coatable length of about 4 meters of 1 cm wide tape. The reel to reel tape deposition system is designed for long term continuous operation based on a vapor composition control by atomic absorption spectroscopy and in situ refillable evaporation sources. In the initial setup phase the system has been able to reproduced the above results on small samples. The first results on continuously moving tape substrate coating up to a length of several meters will be reported.

**9:30 AM E9.3**
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.

Films of $YBa_2Cu_3O_{7-x}$ (YBCO) were prepared on lanthanum aluminate (LAO) single crystals, buffered (500 nm $Y_2O_3$, 50 nm $CeO_2$) Ni-single crystal substrates and $CeO_2$-buffered YSZ single crystal substrates by metal organic deposition (MOD) process. Glassy films were converted to epitaxial YBCO films, by heating in wet atmosphere of $N_2/O_2$ mixture. A novel technique was used to determine kinetics of conversion of films from glassy state to crystalline YBCO films. The concentration of residual fluoride in the partially converted films was examined using fluoride ion selective electrode. Influence of different factors on the conversion rate of the oxyfluoride films, including high and low vapor pressure of water, temperature and substrate material was investigated using that method. Results obtained with the electrode measurements were compared to results obtained by as X-ray analysis and in situ resistivity measurements. Analysis of data received by different techniques showed that fluoride concentration measurement method is a fast and accurate analysis that can be successfully used for the investigation of conversion kinetics of the films. Based on the fluorine analysis of the growth rate at different temperatures activation energy was estimated at 2.6 eV. This result was compared to theoretical thermodynamic calculations.

**9:45 AM E9.4**
DEVELOPMENT OF A HIGH RATE, ECONOMICALLY VIABLE BUFFER DEPOSITION PROCESS FOR YBCO COATED CONDUCTORS. M. Paranthaman, T. Aytug, B.W. Kang, S. Sathyamurthy, P.M. Martin, A. Goyal, D.M. Kroeger, R. Feenstra, and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

A low-cost, high rate, reactive sputter deposition process has been developed to fabricate epitaxial YSZ buffer layers on $CeO_2$-buffered Ni (100) substrates. For fabricating high current YBCO conductors on practical substrates, present experience suggests that the buffer layer thickness should be at least 200 nm to 500 nm. In the standard RABiTS architecture of $CeO_2$ (sputtered)/ YSZ (sputtered)/ $CeO_2$ (ebeam)/Ni, the typical deposition rate for YSZ from an oxide source is 0.5-1 Å/sec. But for producing long lengths of buffered Ni substrates, we need deposition rates in the order of 20-30 Å/sec. We have reactively sputter deposited YSZ layers epitaxially on ebeam $CeO_2$-buffered Ni substrates at reasonably high rates from an alloy target. YBCO films with a $J_c$ of over 1 MA/cm$^2$ at 77 K and self-field have been obtained using pulsed laser deposition on reactively sputtered YSZ layers with $CeO_2$ cap layers. We will discuss in detail about issues related to scale-up of reactively sputter deposition process.
Supported by the U.S. DOE, Division of Materials Sciences, Office of Science, and Office of Power Technologies-Superconductivity Program, Office of Energy Efficiency and Renewable Energy. The research was performed at the Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the USDOE under contract DE-AC05-00OR22725

**10:00 AM E9.5**
A NEW COMMERCIALLY-VIABLE METHOD OF FABRICATING ROBUST OXIDE BUFFER LAYERS FOR YBCO SUPER-CONDUCTING TAPE. Ilwon Kim, Scott A. Barnett, John Rechner, and Sankar Sambasivan, Applied Thin Films, Inc., Evanston, IL; Amit Goyal, Oak Ridge National Laboratory, Oak Ridge, TN.

A novel technique has been developed for fabricating oxide buffers for YBCO coated conductor application. We have demonstrated smooth, crack-, pore- free Yttria-Stabilized Zirconia buffer layers on biaxially textured Ni and Ni-alloy substrates. High quality YBCO layers have been deposited on this buffers with Pulsed Laser Deposition. YBCO films with sputtered ceria intermediate layers reproducibly exhibited Jc values of 1 MA/cm$^2$. The approach avoids difficulties normally observed in initial oxide deposition on the metal tape and potentially allows upto $\approx$ 100 times higher processing rates compared to competing techniques such as RF sputtering. In addition to the processing and superconductivity data, results of structural characterization including x-ray diffraction and High Resolution Transmission Electron Microscopy will be discussed.

**10:30 AM *E9.6**
ENGINEERED MICROSTRUCTURES AND TRANSPORT PROPERTIES IN YBCO COATED CONDUCTORS.
Terry Holesinger, Brady J. Gibbons, J. Yates Coulter, and Stephen R. Foltyn.

Each process used to deposit or make the bi-axially textured template,

additional buffer layer(s), and the superconductor creates interfaces along which defects or interfacial reactions may result. These defects can be additive and propagate through the entire film structure to affect the growth and properties of the superconducting film. Defects within the films and their corresponding transport properties have been correlated with the differences in composition and thickness of the underlying buffer layer materials. We show how this knowledge can be used to maximize critical current densities and engineer specific defect structures for improved transport properties in applied fields.

**11:00 AM *E9.7**
PHASE RELATIONSHIPS IN THE Ba-Y-Cu-F-O-H SYSTEM. Winnie Wong-Ng, Lawrence Cook, Igor Levin, Mark Vaudin, Julia Suh, James Cline, Ceramics Division, NIST, Gaithersburg, MD; Ron Feenstra, Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN.

*Exsitu* methods using e-beam co-evaporated $BaF_2$-Y-Cu-precursor films on RABiTS, followed by post-annealing in the presence of water vapor, have demonstrated the potential of producing high quality, long length $Ba_2YCu_3O_{6+x}$ (Y-213) superconductors. However, the details of phase equilibria and the phase evolution of the Y-213 phase in the multi-component Ba-Y-Cu-F-O-H system, are not completely understood. It is important to determine the role of liquid; specifically, whether an intermediate fluorine-containing low-temperature liquid forms, and whether its formation plays a role in governing the formation of the Y-213 phase. We accomplished the design and setup of the laboratory apparatus (atmospheric controlled furnaces, DTA, preparation of BaO), and sample handling procedure necessary for atmospheric-controlled experiments. Since the interplay of phase equilibria and kinetics is important in this system, we have investigated the melting equilibria involving Y-213 under reduced processing atmospheres, and have also successfully employed the *in situ* high temperature x-ray diffraction to follow the phase formation of the Y-213 phase, starting from amorphous $BaF_2$-Y-Cu films supplied by ORNL. Results of the melting equilibria and phase formation of Y-213 in the presence of water vapor will be discussed.

**11:30 AM E9.8**
EFFECT OF SULFUR SURFACE STRUCTURE ON NUCLEATION OF OXIDE SEED LAYERS ON TEXTURED METALS FOR COATED CONDUCTOR APPLICATION. C. Cantoni, D.K. Christen, A. Goyal, D.M. Zehner, H.M. Christen, Oak Ridge National Laboratory, Oak Ridge, TN; D.P. Norton, University of Florida, Department of Materials Science and Engineering, Gainesville, FL.

RABiTS technology is based on epitaxial deposition of oxide buffer layers on a textured metal surface. This process is intrinsically complex because of the different chemical and electronic nature of the two surfaces involved. Changes in the atomistic structure and free energy of the metal surface determined by chemisorbed species can have a dramatic influence on the seed layer nucleation and ultimately determine the film crystallographic orientation. We have shown that $\{100\}<100>$ textured Ni substrates are characterized by a stable c(2 $\times$ 2) superstructure formed by sulfur, and that such superstructure is responsible for the (200) epitaxial growth of YSZ on the metal. Here we investigate further the mechanism of formation of c(2 $\times$ 2)-S on Ni and its effect on nucleation of other oxide seed layers such as $CeO_2$ and STO. We will present a study of the surface structure and oxidation conditions in biaxially textured Ni alloys such as Ni-3% W and Ni-13% Cr and will address the possibility of oxide seed layer nucleation directly on the Ni-13% Cr surface. Preliminary data on epitaxial cube-on-cube growth of STO on $\{100\}<100>$ textured Cu will be also discussed. Deposition of perovskite buffer layers on Cu tapes is a very interesting approach for coated conductor applications because 1) Cu would provide a non-magnetic substrate less reactive with oxygen than Ni-13%Cr, and 2) a RABiTS architecture consisting of Cu and perovskite conducting buffer layers will provide thermal stability to the HTS tape without the need for an additional cap layer, therefore increasing the tape engineering critical current $J_E$.
Research sponsored by the U.S. Department of Energy under contract DE-AC05-00OR22725 with the Oak Ridge National Laboratory, managed by UT-Battelle, LLC.

**11:45 AM E9.9**
THEORETICAL MODELS OF SPACE CHARGES, STRAIN FIELDS, AND Ca-DOPING AT GRAIN BOUNDARIES IN $YBa_2Cu_3O_{7-x}$. David O. Welch, Brookhaven National Laboratory, Upton, NY; H.B. Su, SUNY-Stony Brook, Stony Brook, NY.

In this paper we will discuss statistical thermodynamic and kinetic models of the origin of space charges and band-bending effects at tilt grain boundaries in $YBa_2Cu_3O_{7-x}$ and the effects of Ca doping upon them. Shell models of interatomic forces are used to calculate the energetics of various relevant point defects and defect clusters as well as their energies of binding to grain boundary dislocations. Open system thermodynamics, including stress effects, will be used to model equilibrium states of Ca segregation, oxygen content and degree of order at various temperatures and oxygen partial pressures. Kinetic considerations and non-equilibrium states will be discussed.

## SESSION E10: YBCO COATED CONDUCTORS IV
Chairs: U. (Balu) Balachandran and Kaname Matsumoto
Thursday Afternoon, November 29, 2001
Room 200 (Hynes)

**1:30 PM *E10.1**
SURFACE-OXIDATION EPITAXY METHOD FOR CRITICAL CURRENT CONTROL OF YBCO COATED CONDUCTORS. Kaname Matsumoto, Kozo Osamura, Kyoto Univ, Dept of Materials Science, Kyoto, JAPAN; Izumi Hirabayashi, ISTEC, Superconductivity Research Laboratory, Nagoya, JAPAN.

The principle and application of surface-oxidation epitaxy (SOE) method, which is one of the methods for preparing long-length biaxially textured template for coated conductors, are discussed. Biaxially oriented surface-oxidized NiO/Ni tape of 50 m length has already been produced by SOE. In-plane texture (delta-phi) of the NiO layer was 12-15 degrees throughout the whole length of the tape. Though biaxial orientation mechanism in the NiO layer on the cube-textured Ni tape is not yet clear, the difference of the growth rate of different NiO crystal plane seems to be a reason of the orientation. In the initial stage of SOE process, (001)NiO grains and (111)NiO grains coexist. However, the (001) grains coalesce and bury (111) grains, and eventually covers all Ni surfaces. The (001)NiO grains grow by keeping epitaxial relation with (001)Ni surface so that the biaxial orientation of NiO layer realizes. Large Jc improvement in YBCO films deposited SOE-NiO tapes has been obtained by using 50 nm MgO cap layer on bare NiO surface. The surface roughness of NiO was greatly improved, since a wettability of MgO for NiO is excellent. MgO was also very effective in diffusion prevention of the Ni element. On the other hand, control of the exact YBCO film deposition condition is necessary in order to form high Jc YBCO films on MgO, because the YBCO grains with cube-on-cube and with 45-degrees rotated orientation are easy to coexist. Consequently, $SrTiO_3$, $BaTiO_3$, YSZ, etc., which have better lattice misfit to YBCO than that of MgO, were examined as new cap layers. If we use one of these materials for cap layer, YBCO deposition condition will be eased. This is very important for fabricating long coated conductors.

**2:00 PM *E10.2**
CHARACTERIZATION OF THE CHEMISTRY AND PHYSICS OF COATED CONDUCTOR EMBODIMENTS BY MOLECULAR SPECTROSCOPY AND SYNCHROTRON X-RAY METHODS. V.A. Maroni, K. Venkataraman, M. Li, Y. Jee, B. Ma, H. You, J.H. Je, W.G. Cullen, Argonne National Laboratory, Argonne, IL; C. Thieme, M.W. Rupich, S. Cui, and S. Annavarapu, American Superconductor, Westborough, MA.

Molecular spectroscopy and synchrotron x-ray diffraction/scattering methods are being used to investigate the microstructural and phase chemical properties of coated conductor specimens prepared by a variety of fabrication pathways that included steps such as inclined substrate deposition of seed layers, pulsed laser deposition of buffer and YBCO layers, and metal-organic deposition of YBCO. The purpose of the studies is to determine how interface physics and chemistry influence the nucleation and propagation of biaxial texture, the development of strain from layer to layer (substrate through to superconductor), and, ultimately, the current carrying capacity of the conductor. Defect structures in the superconductor layer and the presence of second phases were found to play an important role in each case. This presentation will report on (1) the application of Raman microscopy as a primary characterization tool for analysis of the YBCO layer (the technique detects/determines and gives spatial information concerning cation disorder, defect structures, impurity phases, c-axis alignment, approximate YBCO oxygen stoichiometry, and the nature and approximate magnitude of strain fields); (2) the implementation of synchrotron x-ray reflectivity methods to examine the surface roughness and epitaxy of unseeded and seeded substrates, including the detection of sub-micron oxide films on textured metal substrates, the determination of surface roughness parameters before and after seed layer deposition, the measurement of substrate and seed layer grain size, and the examination of mosaic distributions; and (3) the use of molecular spectroscopy and x-ray diffraction methods in combination to measure the thickness of buffer layers and to determine the nature and magnitude of stain fields at buffer layer/substrate interfaces.
Work performed at the Argonne National Laboratory was sponsored by the U.S. Department of Energy (DOE), Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, under Contract W-31-109-ENG-38.

**2:30 PM E10.3**
STRONG CORRELATION BETWEEN TRANSPORT CURRENT DENSITIES AND SPATIALLY-RESOLVED X-RAY ANALYSIS IN YBCO SUPERCONDUCTING TAPES. F.M. Mueller, D.J. Brown, G.W. Brown, M.E. Hawley, J.Y. Coulter, S.R. Foltyn, P.N. Arendt, E.J. Peterson, D.E. Peterson, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM; U. Preckwinkle, B.N. Herrington, Bruker Axs, Inc, Madison, WI.

Transport current pathway information is useful for improving the current conduction of high temperature superconducting tapes because it can be correlated with data from other characterization techniques such as spatially-resolved X-ray Analysis, to optimize tape production methods. The current density (J) pathways can be determined by inverting magnetic field data measured at the surface of the superconductor, but care must be taken to avoid subtle errors in the inversion process. We have developed improved inversion techniques that allow us to compare the spatial dependence of the current density pathways with a variety of current-limiting defects present in superconducting tapes. In particular, our methods more adequately treat the high wave-number noise inherent in these inversion processes. We will compare directly the spatially resolved vector paths of J with X-ray data. The correlation is high. This improved inversion process should lead to improvement in YBCO coated conductor tapes.

**2:45 PM E10.4**
MANIPULATING THE NUCLEATION AND GROWTH OF REBaCuO THIN FILMS. Guus Rijnders and Dave H.A. Blank, Univ of Twente, Low Temperature Div, MESA Research Inst, Enschede, THE NETHERLANDS.

The nucleation and growth of the cuprate high Tc materials, especially the REBaCuO compounds, have been subject to many studies in the last ten years. In the case of deposition techniques such as pulsed laser deposition (PLD) or sputter deposition mainly the growth mode was studied. Depending on the growth conditions, a multiple level 2D or spiral growth has been observed introducing grainboundaries and increased roughening of the film surface. This limits applications based on multi-layer technology. Starting with atomically smooth substrates, i.e., $TiO_2$ terminated strontium-titanate, the initial growth of $REBa_2Cu_3O_{7-d}$ using high-pressure RHEED is studied. With standard PLD conditions, the RHEED intensity drops significantly during the initial growth and nucleation of sub-unitcell blocks is observed by AFM and TEM [1]. As a consequence, defects such as are present in the film. In this presentation growth manipulation using a new growth method, pulsed laser interval deposition of sub-unit cells, will be discussed. With this technique, control and modification of nucleation and growth are possible. Special attention will be paid to the perovskite stacking sequence at the interface with the substrate and its impact on the occurrence of antiphase boundaries during film growth. [1] S. Bals, G. Rijnders, D.H.A. Blank, G. van Tendeloo, Physica C 355 (2001) 225.

**3:00 PM E10.5**
FABRICATION OF HIGH $J_c$ YBCO COATED CONDUCTORS USING SOL-GEL BUFFER LAYERS ON NICKEL AND NICKEL ALLOY SUBSTRATES. S. Sathyamurthy, M. Paranthaman, B.W. Kang, T. Aytug, A. Goyal, P.M. Martin, and D.K. Christen, Oak Ridge National Laboratory, Oak Ridge, TN.

Sol-gel processing of $La_2Zr_2O_7$ (LZO) buffer layers on biaxially textured Ni-3 at.% W alloy substrates using a continuous reel-to-reel dip-coating unit has been studied. The epitaxial LZO films obtained have a strong cube texture and uniform microstructure. The effect of increasing the annealing speed on the texture, microstructure and the carbon content retained in the film were studied. On top of the LZO films, epitaxial layers of Yttria Stabilized Zirconia (YSZ) and Ceria ($CeO_2$) were deposited using rf sputtering, and YBCO films were then deposited using Pulsed Laser Deposition (PLD). A critical current density ($J_c$) of 1.9 MA/cm$^2$ at 77K and self-field and 0.34 MA/cm$^2$ at 77K and 0.5T have been obtained on these films. These values are comparable to those obtained on YBCO films deposited on all-vacuum deposited buffer layers, and the highest ever obtained using solution seed layers. The use of these sol-gel films as single buffer layers for coated conductor processing has been explored. Critical current density of 0.2 MA/cm$^2$, on textured nickel substrates at 77K and self-field, has been measured on these samples. Microstructural analysis of these samples reveals the presence of significant amount of nickel oxide. The processing thicker, denser LZO buffer layer to combat the problem of nickel oxidation using modified sol-gel synthesis is currently being explored. The results obtained from this process will be discussed in detail.
This research was sponsored by the U.S. DOE, Division of Materials Sciences, Office of Science, Office of Energy Efficiency and Renewable Energy, and Office of Power Technologies-Superconductivity Program. This research was performed at the Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the USDOE under contract DE-AC05-00OR22725. Sincere acknowledgements also extended to Oak Ridge Associated Universities for making this work possible.

**3:30 PM *E10.6**
FABRICATION OF REBCO COATED CONDUCTORS BY LIQUID PHASE EPITAXY. Xiaoding Qi, Zainovia Lockman, Musood Soorie and Judith L. MacManus-Driscoll.

Liquid phase epitaxy (LPE) has been used to grow mixed REBCO (RE=Y, Yb, Er) thick films for coated conductor applications. Growth on single crystal substrates has produced very encouraging results. $T_c$'s of 91 K and $J_c$'s close to 1 MA/cm$^2$ have been achieved in several micrometer thick films, with a growth rate of 1 $\mu$m/min. However, the growth of REBCO thick films on metallic substrates is much more difficult. There is an urgent need to find a suitable metallic substrate and its buffer layers, which can meet the special conditions of the LPE growth. The most important requirements of the buffer layer and substrate are a close lattice-match, thermal expansion match and resistance to corrosion by the flux. In this presentation, we will report the results of LPE growth of mixed REBCO's on $RE_2CuO_4$ on surface oxidation epitaxy NiO on RabiTs Ni substrates. Each processing step is highly scaleable.

**4:00 PM E10.7**
PEAK EFFECT AT MICROWAVE FREQUENCIES IN SWIFT HEAVY ION IRRADIATED Y-123 THIN FILMS - INVESTIGATION OF VORTEX DYNAMICS. Tamalika Banerjee, R. Pinto, TIFR, Dept of CMP & MS, Mumbai, INDIA; Avinash Bhangale, Institute of Science, Dept of Physics, Mumbai, INDIA; Pratap Raychaudhuri, University of Birmingham, School of Physics and Astronomy, UNITED KINGDOM; D. Kanjilal, Nuclear Science Centre, New Delhi, INDIA.

The vortex dynamics at microwave frequencies in Y-123 films has been studied. We observe a peak in the microwave (4.8 GHz) surface resistance in some films in magnetic fields (up to 0.8 T) whereas a few films do not show a pronounced peak in their surface resistance. This is associated with the 'peak effect' phenomenon and reflects the order-disorder transformation of the flux-line lattice near the transition temperature [1]. Introduction of artificial pinning centers like columnar defects as a result of irradiation with 200 MeV Ag ion leads to the suppression of the peak in films exhibiting 'peak effect'. Interestingly, a peak is seen to develop in those films that initially did not show a peak in the surface resistance before irradiation. These results and the dynamics of the vortices at microwave frequencies for the unirradiated and irradiated films will be discussed.
[1] A.R. Bhangale, P. Raychaudhuri, S. Sarkar, T. Banerjee, S.S. Bhagwat, V.S. Shirodhkar and R. Pinto, Phys. Rev. B(Rapid) **63**, (2001) 180502R.

**4:15 PM E10.8**
PHASE STABILITY, MICROSTRUCTURE, AND CRITICAL CURRENTS OF YBCO FILMS BY THE IN-SITU E-BEAM METHOD. William Jo, T. Ohnishi, J-U. Huh, R.H. Hammond, and M.R. Beasley, Geballe Laboratory for Advanced Materials, Stanford University, Stanford, CA.

Synthesis of $YBa_2Cu_3O_{7-x}$ films by the in-situ e-beam method is promising for manufacturing scale-up of coated conductors, since it allows deposition with large area, high rate, and thick growth due to low pressure and, a finding that under certain conditions the growth is via a liquid flux. Formation of stable $YBa_2Cu_3O_{7-x}$ at low pressure ($5x10^{-5}$ torr) results from a thermodynamic stability driven by atomic oxygen instead of molecular oxygen. Growth temperature is also found to be an essential parameter to determine phase stability of the films. From TEM studies, layer-by-layer and/or island growth have been observed. It is found that critical currents of the films show a strong dependence on microstructure: high critical currents 3 - 5 MA/cm$^2$ in faulted island grown grains and low in perfect layer-by-layer structures. The faulted microstructure is believed to supply enough pinning centers of vortices and be transparent for oxygen diffusion while layer-by- layer structures show larger lattice constant, indicating that they are oxygen-deficient. Growth on metal tape substrates with high Jc and high rate is a challenge because of reaction and diffusion at the high temperature needed so far for the liquid flux assisted growth. At lower temperature the challenges are different and will be discussed.

**4:30 PM E10.9**
CHARACTERIZING TRANSPORT CURRENT DEFECTS IN 1 CM WIDE YBCO COATED CONDUCTORS. G.W. Brown, M.E. Hawley, Structure-Property Relations (MST-8), Los Alamos National Laboratory, Los Alamos, NM; E.J. Peterson, J.Y. Coulter, P.C. Dowden, P.N. Arendt, S.R. Foltyn, F.M. Mueller, Superconductivity Technology Center (MST-STC), Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

An important aspect of YBCO coated conductor progress is the identification and elimination of isolated regions of low $J_c$ that have been found in otherwise high $J_c$ tapes. In the past these regions have been observed with 4-point probe measurements taken at 1 cm intervals along the tape length. Now we have been able to characterize these regions in greater detail using low temperature scanning magnetoresistance microscopy to map the current pathways in the defective region. These results help us to identify the extent and overall nature of these defects in terms of their effect on the current flow, the property of interest we wish to optimize. In this work we have imaged 5 cm long sections from "good" and "bad" regions of a 1 meter long YBCO coated conductor. The current density map from the "good" region had the expected edge-peaked structure, similar to that found in our previous work on high quality test samples grown on single crystal substrates. The map from the "bad" region showed the current density to be confined to half the width of the conductor with some additional defects and inhomogeneities in the adjacent broader current flow regions. The implications of these structures as well as general correlation of their details with spatially resolved x-ray analysis and electron microscopy results will be presented and discussed.