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Superconducting Technology Program Sandia 1993 Annual Report

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ABSTRACT

Sandia's STP program is a four-part high-temperature superconductor (HTS) research and development program consisting of efforts in powder synthesis and process development, thallium-based HTS film development, wire and tape fabrication, and HTS motor design. The objective of this work is to develop high-temperature superconducting conductors (wire and tape) capable of meeting requirements for high-power electrical devices of interest to industry. The four research efforts currently underway are: 1) Process research on the material synthesis of high-temperature superconductors, 2) Investigation of the synthesis and processing of thallium-based high-temperature superconducting thick films, 3) Process development and characterization of high-temperature superconducting wire and tape, and 4) Cryogenic design of a high-temperature superconducting motor. This report outlines the research that has been performed during FY93 in each of these four areas. A brief background of each project is included to provide historical context and perspective. Major areas of research are described, although no attempt has been made to exhaustively include all work performed in each of these areas.

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1 Process Research on the Synthesis of High Temperature Superconductors

.1 Introduction

The objective of the materials synthesis and process development part of the Superconductivity Program for Electric Power Systems is to (1) develop reproducible, scalable techniques for the preparation and processing of high- T_c materials for use in bulk conductor applications and (2) provide high- T_c powders to support Sandia's initiatives in wire fabrication and thick film development. This part of Sandia's program emphasizes the chemical preparation of high- T_c powders based on a fundamental understanding of solution chemistry. Using liquidus and phase relations as our guide, we are also working on the development of powder processing strategies that optimize the properties of bulk superconductors. Initial synthesis and process development work was on the $\text{YBa}_2\text{Cu}_3\text{O}_x$ (123) system. The 123 powder synthesis technology is relatively mature and has been successfully transferred to US industry. However, fabrication of bulk conductors from this material has been limited by the presence of weak links which are not removable by commercially viable processing techniques. Therefore, we are now concentrating our research efforts on the Tl-Ba-Ca-Cu-O family of materials. This system offers many potential advantages in processing and performance over the 123 materials. We are developing powders with optimized properties that are required for our wire development work in the thallium high- T_c system. We are also developing processing strategies that build on Sandia's established expertise in the processing of thin films and the study of phase equilibria in the Tl-Ba-Ca-Cu-O system.

1.2 **Background**

Our superconductor synthesis work began shortly after the discovery of high- T_c superconductivity in 1986. The work was initiated in Sandia's BES Physics and Chemistry of Ceramics Project, which has as one of its main thrusts the study of the fundamental processes involved in the solution synthesis of ceramics. As a natural extension, we expanded our work to include the chemical synthesis of the newly discovered lanthanum cuprate and the rare earth barium copper oxide (123) superconductors. Throughout our synthesis efforts, we have emphasized the development of synthesis processes that produce fine, homogeneous powders with controlled stoichiometry, are reproducible and scalable, and are easily adaptable to other oxide systems.

The process we developed for the preparation of superconductor precursor powders is based on the simultaneous aqueous coprecipitation of metal hydroxide/carbonates. Briefly, the process involves the rapid mixing of a metal cation-containing solution with a precipitant solution in a continuous precipitation reactor system. Metal cation ratios and precipitant concentrations are controlled such that the carbonate concentration and pH conditions induce complete precipitation. The resulting hydroxycarbonate precipitate is amorphous and can be calcined to form a fine, controlled stoichiometry superconducting oxide powder. The co-ions for the metal cations (e.g., nitrate anions) and the precipitant anions (e.g., tetramethyl ammonia cations) were chosen such that their metal precursor salts are readily soluble in water, are easily pyrolyzable during calcination, and do not form complexes with the metal cations that prevent cation precipitation [J. A Voigt et al., in Mat. Res. Soc. Symp. Pro. 99 (MRS, 1988), p. 635]. A U. S. patent was issued for the process in June of 1989 (Patent No. 4,839,339). Because of the quality of the superconducting powder produced by this process, Superconductor Components, Inc. (SCI), Columbus, Ohio, actively pursued the acquisition of the technology. The transfer was completed in May, 1989 and SCI is

currently marketing bulk conductors and powders made by our process. We have continued to act as consultants to SCI as they work to further improve powder properties and to modify the process to develop and market new oxide materials.

With the discovery of the thallium-based high- T_c materials, we developed and patented a process that incorporates thallium into the superconductor precursor powder during precipitation (U.S. Patent No. 5,001,107, issued March 1991). The process involves the in situ oxidation of the highly soluble Tl^{+1} hydroxide species to the sparingly soluble Tl^{+3} form which precipitates as a hydrous oxide. The oxidation/precipitation of thallium is performed by using hydrogen peroxide as an oxidizing agent. The synthesis is carried out by adding H_2O_2 to the feed solution that contains the non-thallium metal cation constituents of the superconductor (i.e., Ba, Ca, and Cu). The thallium is provided by thallium (I) nitrate which is dissolved in a tetramethylammonium hydroxide/carbonate precipitant feed solution. The rapid mixing of the two feed solutions oxidizes the thallium and produces a controlled stoichiometry Tl-Ba-Ca-Cu hydroxycarbonate coprecipitate [J. A. Voigt et al., in High Temperature Superconducting Compounds: Processing and Related Issues (TMS Press, 1989) p. 291]. The precipitate is easily converted to the desired superconducting phase at 800°C to form a fine, highly reactive powder.

1.3 **Progress in FY93**

Synthesis and processing work in FY93 has dealt with the Tl-Ba-Ca-Cu-O family of high- T_c materials. Specifically, we have focused on the Tl-1223 phase which appears very attractive for wire applications at 77K, as indicated by the results of G.E. showing that the Tl-1223 phase can be fabricated in thick film form (film thickness $\sim 3 \mu m$) with high J_c 's, strong-links, and reasonable flux pinning characteristics at 77K. In order to develop processing protocols that optimize the superconducting properties of this phase in configurations suitable for power applications, we have investigated: (1) the

preparation of powder for use in powder-in-tube wire development; and (2) preparation and processing Tl-1223 thick films.

1.3.1 Powder Synthesis

In the area of powder synthesis, we have continued to use our coprecipitation process to prepare a wide range of Tl-free precursor powders, including powders of nominal metal cation stoichiometry of $\text{Tl}_0(\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3$, $\text{Tl}_0(\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3\text{Ag}_{0.37}$, and $\text{Tl}_0\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{Ag}_{0.37}$. In making quantities of powders sufficient for our wire development needs, our current powder processing facilities make it easier to incorporate the thallium in a second step rather than during precipitation. For most applications the Tl-1223 phase is formed by mixing Tl_2O_3 with the calcined precursor powder and reacting at high temperatures (920°C) for relatively short times (2 hours). We have concentrated on the lead and strontium substituted powders since these substitutions improve processability, superconducting properties, and stability of the Tl-1223 phase. These powders were used as part of on going collaborations with LANL and ANL directed at studying methods for fabricating Tl-1223 in phase pure form. The powders were also used as part of a collaboration with Intermagnetics General, Inc. and LANL dealing with processing of powder-in tube wires.

1.3.2 Thick Film Processing of Pb- and Sr-Substituted Tl-1223

The objectives of our work on the preparation of Pb- and Sr-substituted Tl-1223 thick films has several objectives. First, we wanted to determine if Tl-1223 phase could be formed on the time scale of a rapid thermal processing system, i. e., a few seconds to a few minutes. Initial work was done using LaAlO_3 substrates since they are relatively stable with respect to chemical interactions. Second, thermal processing strategies were investigated to determine if texture could be induced using precursor thick films

prepared by screen printing suspensions of our chemically-derived powders. The ability to prepare films with grain alignment is a critical first step to obtaining acceptable transport properties. Finally, we wanted to see if texture could be induced in thick films using substrates, such as silver, that are suitable for wire development. It is hoped that the processing insight gained from this work will lead to the development of appropriate processing protocols for preparing powder-in-tube Tl-1223 with aligned grain structures.

Precursor thick films were prepared by screen printing alcoholic slurries of the calcined coprecipitated precursor powders onto the desired substrate material. A commercial binder was added to the slurry to improve film integrity. For the LaAlO_3 substrates, a 1 to 1 powder mixture of Tl_2O_3 and the $\text{Tl}_6(\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3$ precursor were used, while for the silver substrates, a similar mixture was used only the precursor powder contained silver which was incorporated during the powder coprecipitation step. Before processing, the 20 μm thick films were subjected to a low temperature binder burnout step. The thick films were wrapped in gold foil with and without an additional thallium source of Tl_2O_3 and processed under various atmospheres (air, O_2 , and Ar) in a rapid thermal processing system.

Films on LaAlO_3 Substrates

Initial experiments were carried out using screen printed thick films on LaAlO_3 substrates with the objective being to explore superconductor phase development under rapid thermal processing conditions. The films were processed in O_2 with additional Tl_2O_3 as a thallium source in order to assure that all processing was done under conditions of a saturated thallos oxide vapor. Figure 1 shows typical Meissner results of a processing series in which the film samples were rapidly heated to 930°C, 940°C, or 950°C, held for two minutes, and cooled at a controlled rate. No additional anneals were done on these films. The figure shows, that for these relatively short

processing times, the Meissner signal at low temperatures and, hence, the fraction of the film that is superconducting increases with increasing processing temperature. In addition, the percentage of the desired TI-1223 phase in the superconducting fraction of the film also increases with increasing temperature. In this figure, the TI-1212 phase has a Meissner onset temperature below 75 K while the TI-1223 phase has an onset temperature above 100 K.

The phase development of this series of films is shown by the back scattered electron photomicrographs given in Figure 2. Also shown in the figure for comparison is a photomicrograph of the unfired film. It shows the precursor film to be composed of 1 μm primary particles that are packed to a uniform density. The contrast in the photomicrographs is due to the different scattering properties of the various phases present, i.e., the highest contrast phase in the unfired film is due to Ti_2O_3 particles. The TI-1212 phase (as determined by EDAX) indicated on the photomicrograph of the 930°C sample consists of tabular grains (low aspect ratio). Consistent with the Meissner results shown in Figure 1, the increasing amounts of TI-1223 is found with higher processing temperature. The TI-1223 phase consists of grains of platey morphologies (high aspect ratio).

The results described above and those of other experiments showed, that although the precursor films on LaAlO_3 could be readily converted to superconducting phases, no development of texture was apparent. For these studies processing temperatures and times were chosen such that there was relatively little sample melting during processing. The microstructural evolution in this case is shown schematically in Figure 3. Also shown in this figure is an alternate processing approach in which the film is subjected to significantly more melting. Under this condition it is expected that aligned microstructures can be developed during a second growth step out of the melt or the melt/quenched film. The following discussion of results deals with using this approach to texture development.

Figure 4 shows Meissner data for a rapidly thermally processed film on LaAlO_3 that was processed in O_2 at temperatures $> 960^\circ\text{C}$ for a short time followed by a controlled cool. At these temperatures significant precursor melting occurs. The Meissner data show that the superconducting portion of the film is a mixture of TI-1212 ($\approx 30\%$ with onset near 50 K) and TI-1223 ($\approx 70\%$ with onset above 100 K). Note the lower than typical $T_{c,\text{onset}}$ for these phases that is due to unoptimized thallium and oxygen content in the film. The film was subjected to no further anneals after the rapid thermal treatment. Unlike the previously discussed films, this film shows evidence of texture as shown by the photomicrograph in Figure 5. EDAX confirmed that the rectangular plates were the TI-1223 phase. There appears to be some preferential orientation among the plates in the a-direction with the single crystal LaAlO_3 substrate as might be expected due to the nearness of the phases in terms of lattice matching.

Magnetic hysteresis data and the calculated critical current densities shown in Figure 6 and 7 in which the magnetic field was applied perpendicular to the surface of the film indicate some strong linking of the grains. This linking is attributed to the presence of aligned TI-1223 based on similar measurements on a variety of TI-1212/TI-1223 thick films which showed little if any hysteretic behavior when there was no grain alignment present. The loop remains open out to the highest applied field of 5 tesla. Using this data, the Bean model was applied with film dimensions being used as the size parameters. The estimated critical current densities are shown in Figure 7 for temperatures of 5 K, 20 K, and 40 K. The magnetization J_c was still nearly 10^4 A/cm^2 at 5 K and a field of 5 tesla. The results shown in the figure are encouraging considering the film contains significant quantities of TI-1212 and has depressed critical temperatures for the two superconducting phases. The film was not continuous enough to do transport critical current measurements and there were regions that showed no alignment. Future work will be directed at improving processing to produce films with useful properties.

Films on Silver Substrates

The development of alignment in thick films of the Pb- and Sr-substituted TI-1223 material on silver has additional complications relative to using LaAlO_3 as the substrate. First, if the texture developed for the previously described LaAlO_3 case is due to lattice matching, the melt/growth process will not work on silver. Second, in order to get sufficient melting of the precursor film on LaAlO_3 in oxygen, temperatures in excess of 960°C were required. The melting point of silver in O_2 is 931°C due to the $\text{Ag}/\text{Ag}_2\text{O}$ eutectic. This difficulty can be avoided by processing under conditions of reduced oxygen partial pressures. For example by processing in argon, the melting point of silver is 962°C . In addition it is well documented that melting reactions in the cuprate superconductor systems are a function of the oxygen partial pressure, with melting temperatures decreasing with decreasing $P(\text{O}_2)$. Therefore, by going to a melt processing atmosphere of argon, temperatures significantly below the melting point of silver can be used. The ability to lower processing temperatures by using argon as the processing atmosphere is illustrated in Figure 8. The figure shows electron photomicrographs of two thick films of the same precursor stoichiometry $((\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3\text{Ag}_{0.37} + \text{Ti}_2\text{O}_3)$ that were rapidly thermally processed in air for 20 seconds at 910°C and in argon for 20 seconds at 890°C . As shown in the figure, both films are microstructurally similar being mainly composed of the TI-1212 phase. The lower temperature, argon processed sample has reacted somewhat more, as seen by the larger TI-1212 grains.

The melt/growth approach to processing thick films on silver substrates was carried out by first processing the gold wrapped samples under argon in the rapid thermal processing system. In this step, the films were heated to temperatures greater than 910°C for short times followed by a controlled cool. These conditions were chosen such that the films experienced a significant amount of melting. After the melt step, the films were annealed in a conventional furnace at 900°C for 1 hour followed by a

850°C, 3 hour hold, and then furnace cooled. The samples were wrapped in gold with a Ti_2O_3 source for all annealing runs.

The importance of the melting step in film processing is shown in Figures 9a- 9c. The figures show scanning electron micrographs of two films after the conventional furnace anneal at 900°C. The film shown in Figure 9b and Figure 9c was subjected to a rapid thermal melt step at 920°C for 20 seconds in argon prior to the anneal. The films have dramatically different microstructures. The furnace annealed-only film (Figure 9a) shows no evidence of texture and is composed of primarily the Sr- and Pb-substituted TI-1212 phase. The XRD pattern of this film (Figure 10) confirms this result and also shows that the film contains secondary phases of CaO, BaPbO_3 , and an undetermined impurity phase or phases. The intensities of the Sr- and Pb-substituted TI-1212 diffraction peaks indicate random orientation.

The film subjected to the melt step prior to the anneal, on the other hand, has regions of highly oriented TI-1223 platelets as shown in Figure 9b. It should be noted that this film was not superconducting after the 920°C melt step with no evidence of either TI-1212 or TI-1223. The film's microstructure at this stage of processing indicates significant melting and densification (compare micrographs shown in Figure 2a and Figure 11). This result confirms that the formation of the superconducting phase took place during the anneal step. Figure 9b shows that the relatively large TI-1223 plates ($> 10 \mu\text{m}$) in the annealed film are stacked several high and intermixed with much smaller TI-1212 grains and other impurity phases. Figure 9c shows a higher magnification of a TI-1223 texture region at the film/silver interface. The TI-1223 plates appear a-axis oriented in the plane of the silver substrate in a fashion similar to that LaAlO_3 case already discussed. The assemblage of plates has the desired "brick wall" appearance. As with the previously discussed film on LaAlO_3 , this film is far from phase pure as shown by its x-ray diffraction pattern given in Figure 10. The pattern is similar to that of the annealed-only film with the important difference being the

presence of Sr- and Pb-substituted Tl-1223. The only additional lines in the pattern are the 00l lines of Sr- and Pb-substituted Tl-1223, with the possible exception of the additional diffraction line of BaPbO₃. This indicates that although the volume fraction of the Tl-1223 phase is quite low, the Tl-1223 that is present is aligned with the c-axis perpendicular to the surface of the substrate which is consistent with the observations made from the photomicrographs.

The Meissner data, shown in Figure 12, for the melt/quenched/annealed film are consistent with x-ray diffraction results. The majority of the superconducting fraction of the film is Tl-1212 with an onset below 75 K and is approximately 16 percent Tl-1223 with an onset near 108 K. Despite the low volume fraction of Tl-1223, the fact that it is aligned gives the film good magnetic hysteresis out to 5 tesla at 5 K as given in Figure 13. Figure 14 shows the calculated critical current density of the film as a function of magnetic field at 5 and 20 K, again determined by application of the Bean model to the hysteresis data using the sample dimensions. The relatively weak dependence of the critical current density with field indicates the presence of strong links. The J_{cm} remains well above 10^4 /cm² at 5 K in an applied field of 5 tesla. The film's nonuniform coverage of the silver substrate made transport measurement not possible.

In summary, our initial work on the processing of Sr- and Pb-substituted Tl-1223 thick films has shown that it is possible to develop textured Tl-1223 on both LaAlO₃ and silver substrates through application of a melt processing step. Our inability to produce high quality, uniform, fully textured films is probably related to the lack of control in the single-zone gold foil wrapped sample technique. Future work will concentrate on using the 2-zone processing approach for the anneal step. The added control of this technique should greatly improve the quality of our films.

1.4 **Future Work**

Our future work in powder synthesis will continue to focus on the TI-material system. This work will support our drawing/rolling, hydrostatic, and coextrusion wire development studies being carried out as part of several collaborations with partners that include LANL, Nuclear Metals, Inc. (a private industrial partner in Concord, MA), AT&T and Intermagnetics General Corp. (IGC). In addition to powder development, we will carry out basic research in the processing of the TI-based materials in an effort to develop a fundamental understanding of how to optimize transport properties during wire fabrication.

We have initiated a study of the effects of composition on the $(\text{Ti,Pb})\text{-(Sr,Ba)-Ca-Cu-O}$ system with respect to critical temperature, phase development, texture development, transport properties, and the irreversibility line. This study is done in collaboration with IGC who will prepare powder-in-tube tapes using the powders prepared for the compositional matrix. Both Sandia and IGC will explore thermal processing and characterization of the tapes. As part of the compositional study, we will characterize sample microstructures through a contract with professor Yufei Gao at New Mexico Tech.

We will continue our thick film development work through newly established collaborations with GE and NREL. Our collaborators will carry out 2-zone thermal processing studies of thick films prepared using Sandia powder. In addition, we will carry out studies at Sandia that combine rapid thermal and 2-zone processing.

Fundamentals of phase formation in the Pb- and Sr-substituted Ti-1223 system will be investigated in 2-zone equilibrium experiments as developed by T. Aselage, SNL, for the undoped Ti-Ba-Ca-Cu-O system. This information will be used to develop thermal processing protocols for thick films and tapes.

Figure 1-1. Comparison of Meissner data for three $\approx 20 \mu\text{m}$ $\text{Ti}_x(\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3\text{O}_y$ thick films on LaAlO_3 following RTP processing in O_2 at 930°C, 940°C and 950°C, respectively.

Figure 1-2. Phase development of thick films rapidly heated to 930°C, 940°C, and 950°C, held for 2 minutes and cooled.

Grain alignment critical for achieving improved transport properties in Tl-1223 system.

Figure 1-3. Microstructural evolution for processing either by solid state sintering or partial melt growth.

Figure 1-4. Meissner data for a $\approx 20 \text{ }\mu\text{m}$ $\text{Ti}_x(\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3\text{O}_y$ thick film on LaAlO_3 following RTP processing in O_2 above 960°C with a controlled cool.

Figure 1-5. Thick film on LaAlO_3 rapidly processed at temperatures $> 960^\circ\text{C}$ showing evidence of texturing of the Tl-1223 phase.

Figure 1-6. Magnetic hysteresis data at 5 K for the same thick film shown in Figs. 4 and 5.

Figure 1-7. Critical current density J_{cm} versus applied field derived from magnetic hysteresis data (Fig. 6) using the film dimensions and the Bean model.

Figure 1-8. Two films with stoichiometry $((\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3\text{Ag}_{0.37} + \text{Ti}_2\text{O}_3)$ rapidly thermally processed in air for 20 s at 910°C and in argon for 20 s at 890°C . The reducing atmosphere results in increased melting even at lower temperatures.

Figure 1-9a. Thick film on Ag substrate furnace anneal only at 900°C/1hr, 850°C/3hr in O₂.

Figure 1-9b. Thick film subjected to rapid thermal melt step at 920 °C for 20 s in argon.

Figure 1-9c. Thick film subjected to a rapid thermal melt step at 920°C for 20s in argon.

Figure 1-10. X-ray diffraction pattern of melt processed film in Figs. 9b and 9c.

Figure 1-11. Thick film subjected to rapid melt processing at 920°C prior to 900°C anneal.

Figure 1-12. Meissner data for a $\approx 20 \mu\text{m}$ $\text{Tl}_x(\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3\text{O}_y$ thick film on Ag following RTP processing in Ar at 920°C and furnace annealing in O_2 at 900°C .

Figure 1-13. Magnetic hysteresis data at 5 K for the same thick film shown in Figs. 10-12.

Figure 1-14. Critical current density J_{cm} versus applied field derived from magnetic hysteresis data (Fig 13) using the film dimensions and the Bean model.

2 Growth of Thick Tl-Ba-Ca-Cu-O Superconducting Films Using Near-equilibrium Processing Conditions

2.1 Introduction

Thick film superconductors are an extension of the development of thin film conductors which have obtained high critical current densities even in the presence of high magnetic fields. However, thin film conductors are limited to small scale applications where epitaxial layers can be deposited on special substrates with matched lattice parameters. The deposition techniques such as magnetron sputtering, e-beam evaporation, and laser ablation are limited in the rate of deposition and the area of deposition. Thick films are an attempt to use bulk deposition and processing techniques to achieve thick layers with properties similar to those of the epitaxial thin films. Thick film deposition techniques include such methods as doctor blade coating, screen printing, spray pyrolysis and electrodeposition. Thick film processing has the advantage over wire and tape processing of having an exposed superconductor surface whose composition can be directly modified by such techniques as two-zone processing. These films are also capable of being analyzed directly for changes in composition and microstructure.

We have adopted the screen printing technique for producing thick films on various substrates including YSZ, LaAlO_3 , and silver. This method can produce coatings in the range from a few microns to several hundred microns. A commercial binder is used to suspend the superconductor powders and the binder is later burned off at temperatures below those that would affect the superconductor. The thick films are processed by conventional furnace annealing in enclosed gold capsules or in a two-zone furnace. The two-zone furnace allows the introduction of thallium into the superconductor by vapor phase transport of Tl_2O which is produced by the thermal decomposition of Tl_2O_3 . The Tl_2O vapor pressure is determined by the temperature of the Tl_2O_3 , which is controlled separately from the sample temperature, and by the

partial pressure of O_2 . The proper choice of these conditions determines the phase development of the thick film and the quality of the interfaces between the superconductor grains. Melt transitions and grain growth during this processing can lead to aligned superconductor grains (texturing) and to the development of strong links between the grains.

2.2 Background

Sandia was one of the first laboratories developing superconducting thin films after the initial discovery of HTS materials in 1986. Tl-Ba-Ca-Cu-O thin films were prepared by sequential electron-beam evaporation of the four metals onto $SrTiO_3$ or $LaAlO_3$ substrates followed by furnace annealing in an enclosed capsule with a separate Tl source. These polycrystalline Tl-Ba-Ca-Cu-O thin films produced in early 1988 exhibited superconducting transition temperatures (T_c 's) above 100 K and critical current densities (J_c 's) above 10^5 A/cm² at liquid nitrogen temperatures in large external magnetic fields. Further development of these HTS films has been funded since 1988 as a research project within the STP program at Sandia, concentrating on improvements in synthesis and processing techniques to yield uniform coverage over large-area substrates with maximum J_c values and vortex pinning strength.

The independent control of Tl and O_2 partial pressures has been found to be essential during post-deposition processing of Tl-based HTS thick films. Sintering or annealing samples in closed crucibles with bulk Tl-Ba-Ca-Cu-O pellets does not offer the desired control and is not a scalable process for manufacturing. These problems have been largely overcome by processing using the two-zone processing technique. Our program has benefited from the extensive early efforts at Sandia by T. L. Aselage in the study of the Tl-Ba-Ca-Cu-O high-temperature equilibrium phase diagram (funded by DOE Basic Energy Sciences under the Physics and Chemistry of Ceramics project). A two-zone furnace was developed for these studies in which the O_2 pressure is controlled in the gas which equilibrates over the sample in one heat zone

while the Ti_2O vapor pressure over the sample is varied by controlling the temperature of a Ti_2O source in a separate heat zone. Hence, independent control is possible of the three variables identified as controlling phase formation: sintering temperature, Ti partial pressure and O_2 partial pressure. Additional two-zone furnaces have been built on this early design and are being used for processing of both thin and thick films.

2.3 **Progress in FY93**

We have studied the growth of 10 - 20 μm thick TiBaCaCuO superconducting films by near-equilibrium processing. Precursor films were deposited on $\text{LaAlO}_3(100)$ substrates by screen-printing chemically-prepared Ti-free Ba-Ca-Cu-O powders. The films are doped with 5 wt.% Ag, which acts as a sintering aid. These precursor films are then thallinated and processed in a closed two-zone furnace. This allows separate control of the substrate temperature, oxygen partial pressure, and thallous oxide partial pressure, each of which is a critical variable for the formation of the Ti-superconducting phases. Film processing in both $p(\text{O}_2) = 63$ and 630 Torr is explored. The substrate temperature for every sample grown was 850°C for two hours. The Ti-oxide source temperature is varied.

Figure 1 shows the thallium intake of the resulting processed films as a function of Ti-oxide source temperature, for each of the oxygen partial pressures used. Energy dispersive X-ray (EDX) analysis was performed to measure film compositions. It is clear that oxygen partial pressure plays an important role in determining the Ti-oxide partial pressure since $p(\text{O}_2) \propto p(\text{TiO}_x) = \text{constant}$. Therefore, we find that films processed in lower $p(\text{O}_2)$ have a greater Ti-content than films processed in higher $p(\text{O}_2)$ for the same Ti-oxide source temperatures. It is worth noting the Ti-source temperatures needed to achieve a Ti content of 12.5 atomic percent, which is the amount of Ti present in the Ti-1223 phase. For $p(\text{O}_2) = 63$ Torr, a source temperature of 690°C is found; for $p(\text{O}_2) = 630$ Torr, a source temperature of 710°C is found.

Figure 2 shows the material phases present in films processed in $p(\text{O}_2) = 63$ Torr at different Tl-oxide source temperatures, as determined by x-ray diffraction (XRD). At the lowest source temperatures studied, below 690°C , the dominant phase is BaCuO_2 . The only superconducting phase detected is Tl-1212. Above 690°C , the BaCuO_2 phase begins to decompose, and the dominant phase appears to be the Tl-2212 superconducting phase. No evidence for the Tl-1223 occurs at $p(\text{O}_2) = 63$ Torr. Figure 3 shows the Meissner transitions for films processed at Tl-source temperatures of 690, 700, and 710°C . These results correlate well with the x-ray structural determinations. For the 690°C film, XRD found mainly BaCuO_2 and some Tl-1212. The Meissner measurement shows a weak superconducting transition below 70 K, which is representative of the Tl-1212 phase. At $T(\text{TlO}_x) = 700^\circ\text{C}$, we get our sharpest transition beginning near 100 K, representative of the Tl-2212 phase. This is the Tl-source temperature where the BaCuO_2 XRD peak falls off sharply. Higher $T(\text{TlO}_x)$ have the same superconducting transition temperature, but the strength of the signal falls off due to the preponderance of Tl-rich phases.

Figure 4 is the XRD determination of phases formed in processing in $p(\text{O}_2) = 630$ Torr. Again, we find that at the lowest Tl-source temperatures used that BaCuO_2 is the dominant phase. However, now we find that Tl-1223 also forms. This is the Tl-superconducting phase with the strongest magnetic flux pinning. It has a maximum appearance in the film processed at $T(\text{TlO}_x) = 710^\circ\text{C}$, which is precisely the source temperature that results in a Tl-content of 12.5% (Figure 1). At higher $T(\text{TlO}_x)$, Tl-2212 dominates. Figure 5 is the Meissner comparisons for films grown below, at, and above the $T(\text{TlO}_x)$ resulting in 12.5% Tl. We see that all the transitions occur slightly above 100 K. The film processed in $T(\text{TlO}_x) = 700^\circ\text{C}$ has the weakest transition, indicative of the large amount of BaCuO_2 present in it. The sharpest transition is for the film processed at $T(\text{TlO}_x) = 710^\circ\text{C}$, and is likely due to the significant portion of Tl-1223 formed. Above this Tl-source temperature, the signal broadens due to the formation of Tl-rich oxide phases, similar to that found for processing in $p(\text{O}_2) = 63$ Torr (Figure 3).

Scanning electron micrographs of the above films show poor grain connectivity, which accounts for the relatively weak superconducting transitions in even the best films.

This poor connectivity is reflected in the modest critical current densities achieved to date. Figure 6 shows magnetic hysteresis data taken at 5 K for the predominantly TI-2212 thick film prepared by two-zone processing at the TI-oxide source temperature of 700°C with $p(\text{O}_2) = 63$ Torr (Meissner data for this film are shown in Figure 2). Using the dimensions of the entire film and the Bean critical state model, hysteresis data yield the critical current density J_{cm} vs. applied field shown in Figure 7. Note that the low-field J_{cm} values are very promising for thick films. The collapse of hysteresis or rapid drop in J_{cm} above 2, 1 and 0.5 Tesla at 5, 10 and 20 K, respectively, indicates poor connectivity between grains (suppressed order parameter) rather than weak vortex pinning. We believe that partial melting during two-zone processing will significantly enhance the connectivity. Previous work indicates that partial melting of the films in the presence of TI-oxide vapor can result in smoother films of these phases. This requires higher substrate temperatures to be reached. We have been limited to 850°C by our current furnace. However, we have recently completed the assembly of a new two-zone furnace capable of achieving temperatures in excess of 900°C. New work will focus on using this new capability to grow higher quality thick films that demonstrate smoother surface morphology, higher yield of TI-1223, and consequently, significantly stronger superconducting transitions and higher critical current densities.

In summary, the two-zone furnace can control the resulting TI-content of films ~ 10 - 15 μm thick. Oxygen partial pressure plays an important role in the growth of TI-HTS thick films. For $p(\text{O}_2) = 63$ Torr, TI-1212 forms at low source temperature $T(\text{TiO}_x)$, and TI-2212 forms at higher $T(\text{TiO}_x)$. For $p(\text{O}_2) = 630$ Torr, TI-1223 forms at low $T(\text{TiO}_x)$, and TI-2212 forms at higher $T(\text{TiO}_x)$. In either low or high $p(\text{O}_2)$, at low $T(\text{TiO}_x)$, BaCuO_2 is difficult to decompose. At high $T(\text{TiO}_x)$, TI-rich oxides form. In general, superconducting transitions measured by magnetization confirm the TI-HTS phases determined by XRD. TI-1223 forms in $p(\text{O}_2) = 630$ Torr at low $T(\text{TiO}_x)$. In this

atmosphere, it is difficult to decompose the BaCuO_2 or to achieve any degree of melting between the grains. Future work will concentrate on substrate temperatures $> 850^\circ\text{C}$, as well as the study of processing schedules that will enhance both the decomposition of BaCuO_2 and the formation of larger grains of TI-1223.

2.4 Specific FY94 research objectives

The coming year's effort will be directed at expanding our understanding of the growth of thick film materials on LaAlO_3 substrates using a two-zone furnace approach. Previous work used Ba-Ca-Cu-O, doped with 5wt% silver, screen-printed precursors and a prototype furnace with the capability of achieving substrate temperatures of 850°C . We studied the effect of TI-O source temperatures, i.e. controlled TI-O partial pressures, and oxygen partial pressure on the growth of superconducting thick films. Although much was learned about the material phase diagram, it was clear that for batch-processing, higher substrate temperatures are required. Therefore, we are developing a new two-zone furnace capable of achieving substrate temperatures in excess of 920°C , uniform over several inches for larger area growth. This furnace will be used to study near-equilibrium growth of the Pb-Sr-doped TI-1223. LaAlO_3 substrates will be used to minimize substrate interactions so that the intrinsic properties of the films can be studied. These results will be applied to the study of these same films on silver substrates that also are currently being investigated. Higher temperature studies of undoped TI-1223 films will also be continued and compared with results for the doped films. The effect of the Pb,Sr additions on enhanced vortex pinning will be determined.

Figure 2-1. Thallium intake in processed films as a function of Tl-oxide source temperature for oxygen partial pressures of 63 Torr and 630 Torr.

Figure 2-2. XRD determination of phases present in films processed in $p(\text{O}_2) = 63$ Torr as a function of Tl-oxide source temperature.

Figure 2-3. Comparison of Meissner data for three $\approx 20 \mu\text{m}$ $\text{Ti}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_y/\text{Ag}$ thick films on LaAlO_3 following two-zone processing in $p(\text{O}_2) = 63 \text{ Torr}$ with Ti-oxide source temperatures of 690°C , 700°C and 710°C , respectively.

Figure 2-4. XRD determination of phases formed in $p(\text{O}_2) = 630$ Torr as a function of Tl-oxide source temperature.

Figure 2-5. Comparison of Meissner data for three $\approx 20 \text{ }\mu\text{m}$ $\text{Ti}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_y/\text{Ag}$ thick films on LaAlO_3 following two-zone processing in $p(\text{O}_2) = 630 \text{ Torr}$ with Ti-oxide source temperatures of 700°C , 710°C and 720°C , respectively.

Figure 2-6. Magnetic hysteresis data at 5 K for a $\approx 20 \mu\text{m}$ $\text{Ti}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_y/\text{Ag}$ thick film on LaAlO_3 following two-zone processing in $p(\text{O}_2) = 63$ Torr with a Ti-oxide source temperature of 700°C .

Figure 2-7. Critical current density J_{cm} versus applied field derived from magnetic hysteresis data (Fig. 6) using the film dimensions and the Bean model.

3 Process Development and Characterization of High-Temperature Superconducting Wire and Tape

3.1 Introduction

The objective of this project is to develop high-temperature superconducting wire capable of meeting the requirements for high-power electrical devices of interest to industry. Primary interest has been to develop materials and processes which can lead to the practical production of wires which can carry high currents in the presence of typical operating magnetic fields at liquid nitrogen temperature or higher, although lower temperature operation is considered for special applications. The thallium-based single layer system is considered the only viable material for operation in moderate magnetic fields at 77 K. In particular, the Pb- and Sr-substituted system is believed to have the best performance in magnetic field and most readily results in the TI-1223 phase desired. During the last year we have devoted our research efforts exclusively in this system, developing techniques in the areas of powder preparation, wire production, and process development.

Sandia's patented co-precipitation process has been used to produce fine-grained, stoichiometric precursor powders to which thallium has been added by bulk mixing with Tl_2O_3 or by vapor phase addition. These powders have been processed in bulk form to investigate microstructural development and also used in the production of silver-clad wires and tapes. Thermal processing schedules using both conventional furnace annealing and Rapid Thermal Annealing (RTP) have been investigated to determine conditions which might lead to the development of strong links and texture.

Wire development efforts have included conventional powder-in-tube (PIT) methods for producing wire and tape, as well as extrusion techniques for producing near theoretical dense core wires. Tapes have been produced using our powders in collaboration with Los Alamos and Argonne National Labs, as well as with Los Alamos

and Intermagnetics General Corporation (IGC). Under a four-way pilot center CRADA between Sandia, Los Alamos, Nuclear Metals, Inc. (NMI), and AT&T Bell Labs, wires were produced by coextrusion and hydrostatic extrusion methods. Conditions for stable extrusion were investigated for each method.

Sheath metallurgy has been found to be important in producing wires with uniform core diameters as well as protecting the core from adverse chemical interactions. Hardened silver alloys are believed to meet these requirements. We have investigated several silver alloys which were prepared in our metallurgy lab to determine if any adverse reactions occurred at typical processing temperatures.

3.2 Background

This is the first year for active collaboration in the area of wire development using the TI-based superconductors. Our coprecipitation powders have been shown to be superior to conventional mixed oxide prepared powders for the preparation of wires and tapes. Thus, several laboratories have shown interest in incorporating our powders into their wire development efforts. However, our collaboration in the area of extrusion goes back to 1989, when Sandia began discussions with Nuclear Metals, Inc. and Los Alamos regarding development of this technique. The first extrusion, using YBCO, were performed in 1990. At that time, AT&T expressed an interest in participating in this collaboration using a similar method of hydrostatic extrusion that they were developing. The first hydrostatic extrusions were performed in April, 1992.

3.3 Progress in FY93

This year had seen the complete conversion of our powder preparation, processing, and wire development efforts from the YBCO system to the TI-based superconductor system. We have demonstrated that our coprecipitation method can produce homogeneous, stoichiometric TI-free precursor powders which can be readily

converted to the desired TI-1223 phase both as bulk pellets and in the form of silver-clad wires/tapes. We have successfully produced powders with TI/Pb and Sr/Ba ratios which are believed to have superior performance in magnetic fields. Thermal processing techniques have been developed which resulted in linking of the superconducting grains as determined by transport and magnetization techniques. Microstructural characterization techniques such as SEM, Electron Microprobe and XRD have been used to determine secondary phase development as well as look for interactions between the superconductor and sheath material.

3.4 **Sandia/Los Alamos/IGC Tape Development Collaboration**

TI-1223 core tapes have been prepared in collaboration with Los Alamos and Intermagnetics General Corp. (IGC) using Sandia's coprecipitated Pb, Sr substituted TI-precursor powders. The powder was reacted at Los Alamos to form the nominal $(\text{Ti}_{0.5}\text{Pb}_{0.5})(\text{Sr}_{0.8}\text{Ba}_{0.2})_2\text{Ca}_2\text{Cu}_3\text{O}_x$ phase and sent to IGC for processing into tape (TW002B). Also, a Sr-only substituted powder was mixed with PbO_2 and Ti_2O_3 and formed into tape (TW001B). These samples were processed at Sandia using both conventional furnace annealing and RTP.

The shielding fraction data for tape TW002B (fully reacted core) are shown in Figure 1 while the Meissner data are shown in Figure 2. The mechanical deformation of the sintered powder during fabrication of this tape reduced the superconducting shielding fraction at 5 K from approximately 100% as-sintered to 25% as indicated by the high-field slope in Figure 1. In addition, the Meissner signal was degraded from a single, sharp transition with onset above 118 K to an extremely broad transition with two distinct onsets, a minor onset at 116 K and the major transition beginning near 105 K. A furnace anneal of this tape (TW002B) for 30 minutes at 885°C followed by 10 hours at 840°C, both in flowing O_2 , recovered a reasonably sharp, single Meissner transition beginning near 120 K. Further, this tape exhibited weak-linked superconducting shielding at 5 K with an estimated 700 A/cm² magnetization critical current at low field

before grain decoupling occurs. At higher fields, the decoupled grains produced a shielding fraction near 100%. Transport measurements on the furnace annealed sample showed a T_c onset of 118 K with a T_c zero of 112 K, but no measurable J_c . An RTP cycle of 30 seconds at 900°C in O₂ with no furnace annealing restored the superconductivity in a second piece of tape. The Meissner signal showed a reasonably sharp, single transition near 116 K and the shielding fraction at 5 K was near 100% in fields above 5 millitesla. An RTP cycle of 920°C for 1 minute in O₂ with no furnace annealing produced partial melting of the superconductor and tape. Although this process restored a single, reasonably sharp Meissner transition and a 5 K shielding fraction near 100%, the process resulted in only minor linking of the grains ($J_c < 100$ A/cm²). However, these data show that Rapid Thermal Processing did produce some intergranular connections between the grains and is capable of restoring the intragranular superconductivity after only a few seconds at temperature.

Similar furnace annealings (885°C/30min; 840°C/10hr) and RTP cycling of tape TW001B (unreacted core) were less successful. Figure 3 shows the shielding fraction data and Figure 4 shows the Meissner data for this tape. In all cases the Meissner signal consisted of two distinct transitions with onsets near 115 and 80 K. The 115 K transition is attributed to the Ti-1223 phase but contributes less than 20% of the Meissner signal in the furnace annealed sample and a smaller fraction in the two RTP tapes. The dominant, broad 80 K transition is assigned to the Ti-1212 phase for all tapes. The furnace annealed tape exhibits complete intragranular shielding above the weak-link decoupling regime at 5 K. The magnetization critical current at decoupling is roughly 300 A/cm². The two RTP tapes (900°C for 30 seconds and 920°C for 1 minute) exhibited intragranular shielding fractions near 100% and 60%, respectively, with negligible linking of the grains. These results indicate that tapes with unreacted cores may have to undergo a lower temperature conversion to the superconducting state prior to any attempt at melt-assisted processing. The conversion of the unreacted core to the reacted state was further examined at two furnace processing

conditions: 850°C/1hr/air and 910°C/1hr O₂. Figure 5 shows that the high-field slope of the magnetic shielding curve, which indicates the fraction of the material in the superconducting state, is higher for the 910°C processed sample, showing almost full conversion to the superconducting state. This indicates that higher processing temperatures are indeed necessary to produce fully reacted cores in these sealed tapes.

3.5 Sandia/Argonne/Los Alamos Wire Development Collaboration

Experiments have also been performed in collaboration with Argonne National Laboratory and Los Alamos National Laboratory using Sandia precursor powders of the Pb and Sr substituted Tl-1223 system. ANL reacted the precursor powders with Ti₂O₃ to produce superconducting powders with the nominal composition (Tl_{0.5}Pb_{0.5})(Sr_{0.8}Ba_{0.2})₂Ca₂Cu₃O_x. Thermal processing of pellets formed from this powder between 885°C to 910°C produced material with an onset temperature near 117 K, as determined by magnetization, but with the presence of a secondary phase with lower transition temperature. However, these samples still showed near 100% superconducting fraction. The powder-in-tube technique was used to produce wires (Figure 6) with a core diameter 0.15 cm using both fully reacted powders and unreacted powders (precursor powder plus Ti₂O₃). Initial processing of the unreacted core wires at 890°C in air for 1 hour then at 850°C in O₂ for 10 hours resulted in a zero resistance T_c of 115 K and a zero field J_c at 77 K of 700 A/cm² (I_c = 12 amps). At 20 K the wire showed an I_c of 30 amps (J_c = 1800 A/cm²).

Magnetization measurements were performed on these wire samples to look for the effects of secondary phases and to determine the limits of intergranular linking. Figure 7 shows the Meissner data for three wires: unreacted/annealed; reacted/as-drawn; reacted/annealed. The as-drawn wire with fully reacted Tl-1223 powder (solid triangles) had a relatively broad Meissner transition with an onset near 116 K and

showed evidence of a second transition near 95 K comprising about 30% of the sample which is attributed to the TI-1212 phase. Furnace annealing (open triangles) increased the onset to 117 K and produced a very sharp transition, removing any trace of a lower- T_c phase. Finally, furnace annealing a wire section containing unreacted powder (open circles) produced an equally sharp Meissner transition with an onset near 114 K. This demonstrated the feasibility of producing the TI-1223 phase from the precursor powder after tape production.

Figure 8 shows the diamagnetic shielding data at 5 K for these wire samples. As expected, the as-drawn wire shows a linear shielding moment versus applied magnetic field, indicating negligible intergranular shielding current. The furnace annealed wires with either reacted or initially unreacted cores showed evidence of intergranular linking up to fields of 7 mT and 4 mT respectively. An estimate of the intergranular shielding current using the critical state model gives a value of magnetic $J_c = 1500 \text{ A/cm}^2$ at 5 K for the furnace-annealed reacted core wire and $J_c = 1100 \text{ A/cm}^2$ for the unreacted core wire. These J_c values are in good agreement with the transport $J_{cs} \approx 1800 \text{ A/cm}^2$ measured at 20 K.

Portions of these drawn wires were sent to LANL for processing into rolled tapes (Figure 9). These tapes had core thicknesses of approximately 0.0032". These tapes were prepared with both fully reacted and unreacted cores. Thermal processing schedules were selected to investigate the conversion of the unreacted cores to the fully superconducting state using conventional furnace anneals. Also, Rapid Thermal Processing (RTP) experiments were performed to determine the effects of limited-time melt transients on phase formation and grain linking. RTP allows controlled transients into the melt regime of the superconductor powders so that grain interface melting may lead to better grain-to-grain contact, increased densification, possible nucleation of strong link interfaces, and the development of textured grain structures as seen for thick films of the same composition. Processing parameters which affect this grain development are RTP temperature, time, atmosphere as well as post-RTP annealing

parameters of temperature, time, and atmosphere. It has been seen that in an oxygen atmosphere the processing window is very narrow for the silver-clad conductors. The melt temperature of the silver drops to approximately 931°C the melt temperature of the superconductor increases to 920-930°C. Processing in a reducing or inert atmosphere increases the melt temperature of the silver to about 960°C while the superconductor melt temperature decreases by almost 50°C.

Initial experiments with the unreacted core tapes showed that the precursor powder transformed to the Tl-1223 state at 850°C in one hour in air with a T_c of 115 K and with no indication of other phases. Alternately, annealing in oxygen at 910°C for one hour followed by 850°C for 10 hours resulted in a T_c of 110 K and a transport J_c of 2×10^3 A/cm² at 77 K and 5×10^3 A/cm² at 20 K. Thus, the J_c results for the unreacted core tapes were a factor of two higher than seen for the drawn wires processed under similar conditions. The reacted core tapes showed even higher J_c values at 77 K, varying between 1.5×10^3 A/cm² and 2.6×10^3 A/cm² after processing at 890°C/1hr in O₂. These data are shown in Figure 10 along with data from subsequent runs.

Initial RTP schedules of 925°C/40s in air and 920°C/40s in O₂ were chosen to introduce melt transformations in these tapes. The RTP process by itself leaves the superconductor in a highly disordered, almost amorphous state. Subsequent annealing at temperatures below the melt temperature is necessary to allow phase development and grain growth. Post-RTP furnace annealing conditions of either 890°C/1hr or 900°C/1hr in O₂ were chosen, each followed by a lower temperature anneal at 850°C for 10 hour in O₂. These RTP samples showed significantly lower J_c values (500-800 A/cm²) although the T_c values were still between 110-113 K. No significant difference was seen between reacted core and unreacted core tapes. Apparently these RTP processing conditions did not result in sufficient melting.

Increased melt transformation was produced in these tapes by switching to processing in an argon atmosphere between 925°C and 945°C for 10-20 seconds followed by a 900°C/1hr/O₂; 850°C/3hr/O₂ anneal. All RTP temperatures resulted in T_cs near 112 K, however, only the 925°C RTP gave a measurable J_c value. This J_c was 3x10³ A/cm² at 77 K and increased to 7x10³ A/cm² at 18 K (Figure 10). These results show that although the processing window for RTP may be narrow, bulk melting of the superconductor can still give substantial J_c values. No attempt was made at this time to optimize the RTP or the post-RTP processing conditions, thus substantial improvements may still be possible.

Typical tape samples processed either by furnace annealing or by RTP were somewhat short (< 1 cm) and could not be completely sealed against TI loss during processing. An attempt to limit the TI loss was made by sealing the ends of one reacted core tape with Au foil and sealing the tape in a Au tube. This tape was furnace annealed at 850°C for 10 hours in O₂. The J_c increased to 3.5x10³ A/cm² at 77 K and reached 1.2x10⁴ A/cm² at 4.6 K (Figure 10). These results indicate that processing longer tape lengths may limit the end losses of TI and may result in even higher critical current values.

The development of the microstructure and secondary phases are important in determining the limits to grain connectivity and critical current density. Samples of the furnace annealed wires and the RTP tapes were examined by electron microprobe analysis to determine quantitatively the composition of both the superconducting and non-superconducting phases. Wires containing the fully reacted powder were furnace annealed in O₂ at 890°C/1hr, 890°C/10 hr, and 910°C/1hr, with each high-temp anneal followed by a low-temp 850°C/10hr anneal. Quantitative analysis showed only one superconducting phase with the composition (Tl_{0.6}Pb_{0.5})(Sr_{0.8-0.85}Ba_{0.2-0.25})₂Ca₂Cu₃O_x in all samples. The Sr/Ba ratio averaged 4.2:1 in all three samples. Table I summarizes the processing conditions and the measured Sr/Ba ratios. Several non-

superconducting secondary phases were identified. Figure 11 shows backscattered electron micrographs of two of these tapes showing several of the secondary phases. Table II lists the secondary phases identified and provides a code for phase identification in the micrographs. Silver was found throughout the bulk of the sample even though no silver was included as part of the starting powder. Obviously, diffusion of silver readily occurs at these temperatures which subsequently segregates as discrete particles during cooling. Two wires containing unreacted precursor powder also were reacted under similar conditions (see Table I). Again, only the TI-1223 phase was observed but the superconductor phase was Ba deficient with an average Sr/Ba ratio ranging from 5.8:1 to 6.4:1. Figure 12 shows a comparison of the individual grain superconducting phase elemental compositions in the reacted and unreacted core wires.

3.6 Compatibility Study of Superconductor and Sheath Material

Metal sheaths are a necessary component of potential superconducting wire or tape. The sheath provides mechanical support, environmental protection, and a conduction path for current in the case of local loss of a superconducting path. The silver based alloys are currently the only viable candidate metals which meet requirements of chemical compatibility with the superconductor, permeability to oxygen, high electrical conductivity, and mechanical deformability. However, the strength of pure silver is low and is mismatched with the mechanical deformation properties of the superconductor core. Oxide dispersal in a silver matrix has been used to harden these materials while maintaining the essential elements of the sheath material.

We have prepared a number of trial silver alloys using different oxide dispersals. Although the mechanical strength of the alloy was increased, the chemical compatibility of these alloys with the superconductor was not known. Therefore, a compatibility study was performed of candidate Ag alloy sheath materials and some of their constituents with the TI-1223 core material. Mixtures included Ag/Ti, Ag/HfO₂,

Consil 995, Ni, and TiO_2 . DTA was initially used to determine any interaction temperatures. Figure 13a and 13b shows a comparison of the DTA scans for the superconductor core (fully reacted) and a mixture of the superconductor and a silver-based alloy (Consil 995). The scans show that the presence of the silver reduced the melt temperature by approximately 30°C and increased the reaction during the melt. Mixtures of the TI-1223 and the sheath alloys were exposed to similar thermal heat treatments as would be given to a wire or tape ($900^\circ\text{C}/2\text{hr}/\text{O}_2$). The mixtures were then measured by magnetization to determine if any poisoning of the superconductor had occurred. Initial DTA results have shown that TiO_2 , although stable by itself up to 1000°C , reacts with the TI-based superconductor starting at approximately 860°C . The reactants are probably Sr and Ba titanates. Interactions with the Ag/Ti alloy powder were also seen, although to a lesser extent than for the pure TiO_2 . However, magnetization measurements showed that no deleterious reaction took place in any of the Ag alloy/superconductor mixtures. All samples showed a sharp T_c of 115 K as shown in Figures 14 and 15. The lack of measurable effect on T_c in the TiO_2 /TI(Pb,Sr)-1223 mixture, as determined by magnetization, indicates that the interactions with the superconductor must be localized near the TiO_2 particles. The lack of interaction of the constituent oxides with the superconductor indicates that these alloys are all viable candidates as sheath material in the TI-1223 system, although the Ti-based alloy is unstable at high temperatures. It was observed that all of the mixtures with Ag containing sheath material had greater shielding and Meissner signals. These results again point to the beneficial role of Ag in processing these materials.

3.7 Extrusion of TI-core Wires

The development of high intergranular transport current density is significantly affected by microstructural properties such as grain size, porosity(density), and secondary phases. Conventional powder-in-tube methods using wire drawing and rolling subject

the superconducting material to shear stresses which result in cracking and low density which subsequently requires long-time, high-temperature anneals to repair. We have investigated the effects of high-densification methods of wire production using the techniques of coextrusion and hydrostatic extrusion. These methods offer the advantage of uniformly compressing the superconducting powder in a protective, conductive sheath which leaves the ceramic material in a high-density, compressive state. This compressive state minimizes crack formation and propagation and offers the possibility of enhancing grain sintering at lower temperatures.

3.7.1 Coextrusion

The coextrusion technique is based on using a hydraulic ram to extrude a billet containing the superconductor core and sheath material through a reduction die to obtain a highly densified and elongated core encased in the outer extrusion billet material. In collaboration with Nuclear Metals, Inc., we have available extrusion presses ranging from 100 tons to 1400 tons capacity which allows extrusion over a wide range of geometries. Figure 16 shows a schematic diagram of the extrusion process.

The coextrusion effort this year has transitioned from extrusion of YBCO to the TI-based superconductors. The mechanical properties of the TI-based superconductors and the precursor oxides are similar to those of YBCO, in that no slip systems are active in the temperature ranges of the extrusion processes. Therefore, many of the requirements for matching mechanical properties of the superconductor core and the sheath material are similar for the two systems. Extrusions were initially performed in pure Ni, but the superconductor cores were not continuous. Apparently the large mismatch in extrusion constants between the core and the stiff nickel sheath was too great. Coextrusions were performed with a number of different sheath materials that had lower extrusion constants: Cu-10wt%Ni, sterling silver, and pure silver. Extrusion parameters were also varied, including superconductor core diameter, extrusion

temperature, and area reduction ratio. Figure 17 shows x-radiographs for some of these extrusions. The radiographs showed that for all extrusion conditions and billet material, the superconductor cores were continuous and uniform.

3.7.2 *Hydrostatic Extrusion*

This year the hydrostatic extrusion program was also switched to the TI-based superconductor system. These extrusions were performed in collaboration with Los Alamos and AT&T Bell Labs. Hydrostatic extrusions were performed at Bell Labs using the CdO hardened silver alloy Consil 900 as the billet material. Successful extrusions were performed at area reduction ratios up to 25:1 with a core diameter of 0.25", but higher reduction ratios with this core diameter stalled the extrusion press. Billets with core diameters of 0.20" were prepared and successful extrusions were performed at reduction ratios of 25:1, 45:1, and 100:1. Extrusion lengths of over 4 meters were obtained at the 100:1 reduction ratio. X-radiography showed the low extrusion ratio cores were continuous, although not very uniform in diameter, while the higher extrusion ratio cores were not completely continuous, although regions on continuity were obtained for lengths of about 1 meter. There were indications during the extrusion that hydrostatic conditions were not being maintained possibly due to the condition of the extrusion die. New extrusion dies are being made and should result in improved conditions leading to more uniform cores.

3.8 Future Work

Evidence has shown that texturing is necessary to achieve high current densities and strong links in the TI-based superconductors. Conventional sintering is not sufficient to achieve strong links, but instead, melt processing is required to produce platelets which can develop into a textured structure. Changes in the stoichiometry of the precursor powders can lead to enhanced melt-phase formation at lower processing temperatures. The Sandia coprecipitation process is uniquely capable of producing

powders with tailored stoichiometry in batch quantities. In collaboration with our ceramic processing group, an experimental matrix has been designed with variations in the TI/Pb ratio, the total TI/Pb content, and the Ca/Cu level to change liquid formation and subsequent grain growth. Thirteen powder compositions will be prepared in our processing laboratory. These powders will be provided to our industrial partner Intermagnetics General Corporation for processing as Ag-clad tapes. These tapes will be used to investigate grain development over a range of thermal processing conditions. Tapes, produced using both unreacted and reacted powders, will be processed using Rapid Thermal Processing techniques as well as conventional furnace annealing. These tapes will be characterized using our magnetization and transport capabilities while microstructural characterization using SEM and TEM will be performed in collaboration with New Mexico Tech.

Process development of the TI-1223 tapes will also be performed in collaboration with industrial partners in conjunction with our National Laboratory partners Los Alamos, Argonne, and NREL. With Los Alamos and IGC, we will continue to research the role of temperature and extended furnace annealing time on Ag-clad tapes to identify optimum grain growth conditions. Los Alamos will these tapes produced using our powders to examine the effects of pressing and sequential rolling/annealing steps. In collaboration with Argonne, we will be investigating the melt temperature of tapes produced using powders from our compositional design study. Argonne will be using their sound velocity apparatus to identify the melt temperature in long pieces of tape exposed to various atmospheres. We are also providing powders to NREL for processing into thick film tapes using their techniques of spray pyrolysis and electrodeposition. They will be using two-zone processing for conversion of the (Pb,Sr) precursor powder to the fully reacted TI(Pb,Sr)-1223 system. We will be performing characterization of these films using magnetization, transport, SEM, and TEM.

We are also continuing our extrusion efforts in collaboration with NMI, Bell Labs, and Los Alamos. We have established a working group of researchers from the four partners which meet quarterly to share research results and plan future development efforts. With NMI, we are planning coextrusions using combinations of silver and copper sheath materials. Techniques will be investigated to reduce the amount of silver necessary for protection of the core material. Coextrusions of multicore conductors will also be performed and characterized at Sandia using x-radiography and Computer Aided Tomography. NMI will provide dense coextruded cores of the Ti-based material with a silver sheath as the starting core for hydrostatic extrusion at Bell Labs. Denser, more uniform starting cores may result in improved stability during the hydrostatic extrusion. Hydrostatic extrusions are also planned using a new multitaper die which should reduce breakthrough pressures.

Table 3-1. Summary of Electron Microprobe Results for Processed Wires

<u>Powder</u>	<u>Processing (O₂)</u>	<u>Avg. Sr/Ba Ratio</u>
Unreacted	910 °C/1hr 850 °C/10hr	6.4:1
Unreacted	890 °C/1hr 850 °C/10hr	5.8:1
Reacted	910 °C/1hr 850 °C/10hr	4.2:1
Reacted	890 °C/1hr 850 °C/10hr	4.2:1
Reacted	890 °C/6hr 850 °C/10hr	4.2:1

Table 3-2. Non-superconducting Secondary Phase ID

<u>Code</u>	<u>Phase</u>
A	Silver
B	BaPbO ₂
C	CuO
SP	1223 Superconductor
SCC	(Sr,Ca)CuO ₂
CP	CaPbO ₂
CA	CaO or CaCO ₃
SB	(Sr _{0.66} ,Ba _{0.33})CO ₃
BS	(Ba _{0.66} ,Sr _{0.33})CO ₃

Figure 3-1. Shielding comparison of reacted core tape (TW002B). Comparison of furnace annealing and Rapid Thermal Processing.

Figure 3-2. Meissner comparison of reacted core tape (TW002B). Comparison of furnace annealing and Rapid Thermal Processing.

Figure 3-3. Shielding comparison of unreacted core tape (TW001B). Comparison of furnace annealing and Thermal Processing.

Figure 3-4. Meissner comparison of unreacted core tape (TW001B). Comparison of furnace annealing with RTP.

Figure 3-5. Shielding data for two furnace anneal temperatures for initially unreacted cores.

Figure 3-6. Drawn wire with TI-1223 core using PIT method.

Figure 3-7. Meissner data of drawn wires of Tl-1223, as drawn and annealed.

Figure 3-8. Shielding data for TI-1223 drawn wires, as-drawn and annealed.

Figure 3-9. Rolled tape with TI-1223 core using ANL drawn wire.

Figure 3-10. Transport critical current density for furnace annealed and RTP/furnace annealed tapes.

Reacted Core: 910C/1h

Unreacted Core: 910C/1h

Figure 3-11. Backscatter electron micrograph of annealed wires. See Table II. for secondary phase codes.

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Figure 3-12. Atomic composition of individual superconducting grains in reacted core and unreacted core tapes.

Figure 3-13a. DTA scan of fully reacted TI(Pb,Sr)-1223.

Figure 3-13b. DTA scan of mixture of TI(Pb,Sr)-1223 and Ag-based Consil 995.

Figure 3-14. Magnetization data for processed mixtures of sheath alloys with Tl-1223.

Figure 3-15. Magnetization data for mixtures of Tl-1223/TiO₂/silver and Tl-1223/HfO₂/silver.

Figure 3-16. Schematic of extrusion press.

Figure 3-17. X-radiographs of coextruded conductors with TI-1223 cores.

4 Cryogenic Design of a High-Temperature Superconducting Motor

4.1 Introduction

Development of high-temperature superconductor (HTS) motors can offer significant electrical power savings for industrial users, and material cost savings for the manufacturers. This program is tasked with investigating the cryogenic cooling of a HTS motor. The cooling of a superconducting motor presents a unique problem due to the rotational motions involved. The rotational motion establishes significant radial pressure gradients that complicate the flow distribution. Also, the strong radial acceleration may cause the two-phase flow to stratify at lower qualities (vapor mass fractions) than predicted by standard flow regime maps. Finally, the flow is susceptible to standard flow instabilities, such as a Ledinegg instability, and a Density Wave Oscillation. A computer model has been developed under this task to predict the flow distribution and stability of the cryogenic flow used to cool a superconducting motor.

4.2 Background

Sandia has participated with Reliance Electric to determine the feasibility of producing a 5000HP superconducting electric motor. Although Sandia has not directly dealt with HTS motor design, we have contributed to the program through our demonstrated capability in numerical modeling of thermal systems. Our modeling efforts of the axial channel cooled rotor began in 1992 and has progressed through several iterations of motor design.

Sandia's major responsibility is to aid in the modeling of the heat transfer aspects of the superconducting motor. The cooling of the superconducting materials in the field windings of the rotor presents a unique application of cryogenic engineering. Various motor design concepts are under consideration at Reliance Electric. As the

performance of HTSc materials improve, the operating temperature increases. The goal is to develop a motor that will operate at liquid nitrogen temperatures or higher. This would enable use of a single cycle refrigeration system, or a once through liquid nitrogen cooling system.

4.3 Progress in FY93

Various systems operating at intermediate temperatures (between liquid helium and liquid nitrogen) are being designed and/or constructed by Reliance Electric. The designs have considered both forced convective cooling and pool boiling designs. Figure 1 shows a potential forced convective cooling design. A multiphase-flow computer model of the cryogenic cooling has been developed to calculate the boiling heat transfer and phase distribution of the various cryogen coolants in the motor. It is important to accurately calculate both the gas and liquid phase velocities towards the exit to enable prediction of the pressure distribution within the rotor. The coolant flow through each of the axial coolant channels requires accurate prediction of the pressure distribution. The internal pressure field variations result in significant nonuniformities in the flow of the coolant. Due to the variable speed design, the flow distribution changes during operation. The model predicts the flow and temperature fields within the rotor. Figure 2 shows a typical result of the predicted temperature field of the rotor in Figure 1. It has been found that a cryogenic pump within the system is not required. The rotational motion causes the rotor to act as its own pump, drawing liquid from a liquid nitrogen reservoir and recycling the liquid portion of the flow that exits the rotor. The work at Sandia also identified a potential flow stability problem that is possible in some of the designs considered for the HTSc motor. This instability could lead to overheating of portions of the rotor and should be avoided.

Pool boiling designs are considered as an alternative to the forced convection design shown in Figure 1. In this design the coolant enters a central void in the rotor. The centripetal acceleration causes a flow of the coolant and subsequent removal of heat.

The large pressures generated by the centripetal acceleration often prevents boiling of the coolant (in a manner similar as found in earlier liquid helium low temperature superconducting generators), so pool boiling is somewhat a misnomer. Due to the complex multidimensional flow in a rotating environment, accurate analytical predictions of the various pool boiling designs developed are not possible. Therefore, the design process for the pool boiling options have centered on experiments conducted at Reliance Electric. Sandia has aided in the design of the experiments and the analysis of the results. So far the results have been very promising, indicating that this mode of cooling is possible.

A final design effort is the rotating coupler that will allow injection of the cryogen into the rotor. This device must operate with minimal heat and mass leakage. Heat and mass leakage will result in greater operational costs and frosting of the device. Sandia has aided in the design and analysis of a coupler for use in the HTSc motor project.

4.4 Future Work

The designs for this program have been predicated on the existence of superconducting wire that can operate at liquid nitrogen temperatures with high current levels and in the presence of magnetic fields in the 3-5 Tesla range. Since wires with these properties are still under development, continued design work on this project will be suspended. As superconducting wires become available with known properties, these studies will be resumed to accommodate realistic operating conditions.

Figure 4-1. Simplified geometry of the cryogenic forced convection rotor for a 5000-hp synchronous motor. Liquid nitrogen enters on the right, passes through the rotor center, is distributed radially in the inlet header, then flows axially back through the field winding. Liquid and vapor nitrogen are collected in the outlet header and exhausted.

Figure 4-2. Temperature distribution within the LN₂ rotor.

Distribution:

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DOE/HQ	R. Eaton
DOE/AL	R. Balthaser
NREL	R. McConnell
Battelle	James Dirks
ORNL	R. A. Hawsey
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BNL	D. O. Welch
Argonne	R. B. Poeppel
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Reliance	R. Schiferl
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