

AD-A259 415



2

ANNUAL REPORT

N00014-88-C-0681

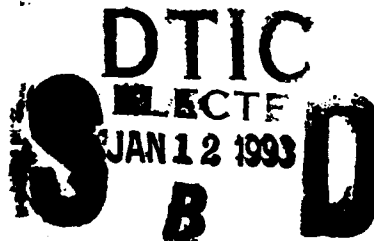
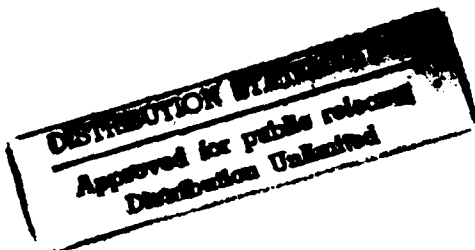
CNR

DEVELOPMENT OF ELEMENTS OF A HIGH Tc
SUPERCONDUCTING CABLE

Dr. Kenneth W. Lay
518-387-6147
FAX 518-387-7495

GE CORPORATE RESEARCH & DEVELOPMENT
PO BOX 8
Schenectady, NY 12301

July 1, 1991 - June 30, 1992



13
p4p

93-00660



93 1 11 004

PROGRAM SUMMARY

The program is aimed at the development of long lengths of silver-clad BSCCO. The material of choice is BSCCO-2223 with 20K the operating temperature goal. Such a tape conductor ultimately could be used in a coil for a magnet, motor or generator.

The program is designed to tackle several key problems with parallel tasks. A large variety of BSCCO powders are under investigation. Silver tubes are packed with superconductor powder and deformed to form tape conductors. An alternate method of making silver-encapsulated superconducting tape has also been pursued. Variations in the powders and processing parameters are used to optimize the tape J_c .

Emphasis during the last year has focused on improvements in the processes for tape made by the "conventional" powder in tube (PIT) process. Progress continues on several fronts as we clarify the conditions required to obtain high J_c tapes. In this year:

- 1) An internally oxidized silver magnesium alloy was shown to harden (instead of soften) during 830°C air heat treatments. PIT tapes have been made using this material for cladding. The hardened material is much easier to handle after the final tape heat treatment operation.
- 2) Improved J_c values were obtained by special, low oxygen pressure treatments during cooling from the final heat treating temperature. Unanticipated increases in J_c were seen for samples heat treated in a 2% O_2 atmosphere at 780°C . The reason for the J_c increase is now under study.
- 3) Much more rapid conversion to the 2223 phase was attained by sintering in an atmosphere of 8% O_2 instead of air. The sintering time to attain high J_c values still requires the final heat treatment to be tens of hours regardless of atmosphere.
- 4) Using a larger roll and/or higher reductions per pass during the final tape forming gave shorter, but wider tapes. The difference in deformation mode should change the orientation of deformation induced flaws and result in higher J_c values.
- 5) A technique was developed for joining prefired tapes. High J_c values could be attained with superconducting joints made by removing the silver from one side of the ends of two tapes and pressing them together followed by standard heat treatment and deformation processing.

HARDENED SILVER

Silver is the material of choice for the sheath of PIT processed tapes containing bismuth based superconductors. It is very ductile and can be extensively cold worked. It would be desirable, however, if the silver hardness could be increased. After the final tape anneals at temperatures over 800°C any work hardening present in the silver clad is annealed out. The resulting soft silver sheath allows for a more easily damaged tape. In addition, there is a possibility that a harder silver sheath will transmit less tensile strain to the superconductor core during the final pressing or rolling operations. These tensile strains in the plane of the tape can lead to cracking of the core. Such cracks are particularly detrimental if the cracks are transverse to the tape.

We have shown that the addition of a small amount of magnesium to silver results in an alloy which can be internally oxidized during the final tape anneals and results in a hardening of the silver which persists during the very long high temperature anneals. Figure 1 shows hardness data for a magnesium doped silver alloy in comparison with two silver standards. It can be seen that the alloy as-processed shows similar properties to pure silver. When heated in an oxidizing atmosphere, however, the alloy hardens while the pure silver softens. We have made magnesium-doped silver tubing which was used to make superconductor tape. The expected hardening did result in a much more easily handled tape after the final heat treatment. Unfortunately some reaction occurred between the tape and cladding. We are now looking at cures to this problem.

ATMOSPHERE EFFECT DURING COOLING

We have discovered that the cooling atmosphere after the final heat treatment is an important source of variability in J_c . Cooling in high oxygen pressure atmospheres, including air, can degrade the superconductor core of a tape as is shown in Figure 2. On the other hand cooling in reduced oxygen pressures can enhance the J_c , Figure 3. We are now looking for the reason for this improvement. Decomposition of some of the 2223 phase during cooling seems to be the reason for degradation during oxidative cooling. This decomposition would be expected to occur at grain boundaries so that a small amount of decomposition could have a major effect on current transport. The formation of phases containing lead in the +4 state seems to be the major culprit. If this is the case, cooling in a less oxidizing atmosphere reduces the stability of the plumbate such that it is stable only at lower temperatures. If the temperature is low enough, slower kinetics will allow retention of the metastable 2223 during cooling.

A study of literature data on 2223 formation, calcium plumbate formation, and grain boundary losses from ac susceptibility seems to give a consistent picture. It is argued that the best cooling procedure for 2223 tapes is to avoid the lead 4+ plumbate stability region until the temperature is below about 700°C. The way to do this is to use an oxygen pressure of about 2 % during the cooling cycle.

Examples of critical current data from one of our tapes which was fabricated including the low oxygen pressure final cooling cycle are shown in Figures 4 and 5. Figure 4 shows critical current density data for the tape which had a cross section of the superconductor core of 0.163 mm². The magnetic field was in the unfavorable direction with the field perpendicular to the tape. Since a coil made from a tape will inevitably have some region of the coil with a component of the field perpendicular to the tape, the J_c in this configuration may control the coil properties. Figure 5, on the other hand, is for the field in the favorable direction, perpendicular to the tape length but in the plane of the tape. In this case I_c is shown instead of J_c to show the high current possible in a single conductor - important for economical coil winding. The figure also shows the high current measuring capability at GE CRD for temperatures between the boiling points of helium and nitrogen and in fields up to 9T.

ATMOSPHERE EFFECT DURING HEAT TREATMENT

We have conclusively shown that the best J_c values in PIT tapes are obtained starting with 2212 powders and reacting to 2223 after fabrication of the tape configuration. The initial powder can either be a partially reacted mix which has not yet been totally reacted to Bi-2223 or it can be a mixture of pre-reacted Bi-2212 and other phases. The Bi-2212 in the tape is aligned during the tape rolling operation.

The final processing of PIT tapes involves a series of heat treatment and deformation cycles. The optimum heat treatment temperatures (in air) for our compositions are in the range 825 - 840°C. Higher temperatures drastically degrade the samples by a partial melt involving the silver sheath. Lower temperatures do not develop the necessary well-sintered 2223 phase. We have typically used three final heat treatments with two intermediate pressing operations. We found that it is important to have some unreacted 2212 phase present after the two initial heat treatments. It is thought that the reaction from 2212 to the desired 2223 phase helps to heal defects from the pressing operation.

Studies of the effect of oxygen pressure on the kinetics of the 2212 to 2223 reaction showed that lower oxygen pressures greatly speed up the reaction. We found that 24 and 48 hour

times in air for the first two heat treatments could be replaced by 8 and 12 hour heat treatments in an 8% oxygen environment. The final heat treatment times, however, need to be just as long in either atmosphere, 72 hours. Even though most of the reaction to the 2223 phase occurred in less time than this for the low oxygen environment, some other parameter requires a longer time to get high J_c values. It seems that the initial anneals can be done in shorter times in reduced oxygen pressures, but that the time for the final anneal is relatively independent of oxygen pressure, at least for 8 and 20 % oxygen.

MECHANICAL DEFORMATION PROCESSING

A process for fabricating tapes as thin as 0.1 mm is in-hand. Typically, 0.11 m long powder columns packed into 6.35 mm diameter silver tubes result in 2.7 m long tapes 0.25 mm thick and about 3 mm wide. Further rolling can reduce the tape thickness to 0.1 mm if desired. We have made 14 meter long 0.25 mm thick tapes using commercially available 0.5 m silver tubes. Longer lengths can be made depending on the availability of longer and/or larger OD starting tubes.

Swaging is done to densify the powder column, thicken the silver wall and work harden the silver. Most of the diameter reduction and fiber lengthening is done by drawing. A more uniform superconductor thickness across the final tape is obtained if the final operation before tape rolling converts the cross section from round to rectangular using a turks head roll set. (A turks head roll consists of two sets of rolls at 90° so that a rectangular cross section can be rolled.) It also has been found that turks head rolling to form a rectangular cross section wire before the tape rolling operation consistently gives higher J_c values compared to rolling a round wire. The diameter at the transition from drawing to rolling controls the final tape width. We have typically drawn to 1.3 - 1.5 mm which gives 2.3 - 2.8 mm wide tapes at 0.25 mm thickness. During the rolling operation the Bi-2212 in the superconductor core is aligned with the basal planes perpendicular to the thickness of the tape.

Recent work has shown that the roll diameter and the thickness reduction per roll pass affect the tape deformation. Large thickness reductions per pass and/or larger rolls both tend to give wider and shorter tapes. This should tend to cause tensile deformation cracks in the superconductor core to be parallel to the tape axis. Parallel cracks are obviously less detrimental to current transfer than the transverse cracks which occur during tape lengthening.

Rolling or pressing operations are needed to densify the superconducting core. We have shown that pressing is much

superior to rolling to obtain high J_c values. We have built a special pressing jig to allow pressing overlapping segments. There was no degradation of the critical current at the overlap region. In this way we can press tapes of any desired length.

SUPERCONDUCTING JOINTS

A method for joining tapes of Ag clad, oxide superconductors has been developed. Joints can be made to connect two tapes which have not gone through the final heat treatment process. The Ag sheath is removed from one side of each of two tapes without significantly disturbing the superconducting core. The exposed superconducting core of the two tapes are brought into contact and pressed so as to again seal the superconductor in a Ag sheath. Finally a reaction anneal is performed to join the two cores together and repair damage associated with the removal of Ag and the pressing operation. Proper joining during the anneal requires the presence of unreacted material which is precursor to the superconductor. The reaction of this precursor material is thought to assist in the healing of cracks. It is expected that joining of fully reacted tape will require the addition of precursor material when forming a joint.

Measurements of the current-voltage relationship at a joint region were made with several configurations of the voltage probes as indicated in Figure 6. The critical current across the entire joint region (a-e) was typically about 1/2 that of the tapes themselves. Comparison of the voltage drop across different regions in the joint indicated that the degradation occurs where the overlapped region ends (i.e. between b and c or between d and e). In the overlapped region itself (i.e. between c and d), the critical current is approximately twice that of the tapes. The higher J_c in this region results from the larger cross-section of the superconductor.

TALKS AND PAPERS THIS PERIOD

KW Lay, RH Arendt, JE Tkaczyk, and MF Garbauskas, "Silver-Clad Bi-2223 Processing", Talk given at New York State Institute on Superconductivity Conference, Sept. 26, 1991, Buffalo, NY, Published in conference proceedings.

JE Tkaczyk, RH Arendt, HR Hart, KW Lay, and FE Luborsky, "Electric Field Versus Current Density Relations for Bi(2223) Tapes", Talk given at DARPA Meeting in Seattle, WA, Sept. 30, 1991

KW Lay, RH Arendt, JE Tkaczyk, and MF Garbauskas, "Factors Affecting Jc of Silver Clad Bi-2223 Tapes", Talk given at DARPA Meeting in Seattle, WA, Oct. 1, 1991

KW Lay, RH Arendt, JE Tkaczyk, and MF Garbauskas, "Factors Affecting Jc of Silver Clad Bi-2223 Tapes", Poster presented at MRS Fall Meeting in Boston, MA, Dec., 1991

KW Lay, "Post Sintering Oxygen Pressure Effects on the Jc of BPSCCO-Silver Clad Tapes", invited talk at Spring MRS Meeting, San Francisco, CA, May, 1992. To be published in conference proceedings.

GOALS FOR NEXT PERIOD

Further studies of hardened silver for cladding.

Preparation of final report.

FINANCIAL STATUS

All values are cost plus fixed fee total costs.

TOTAL FUNDING REQUIRED FOR EFFORT \$2,424,530
01Sept88 through 31Dec91 (52 months)

CURRENT AUTHORIZATION 2,424,530
01Sept88 through 31Dec92 (52 months)

FUNDING EXPENDED TO-DATE 2,378,812
01Sept88 through 30 June92 (46 months)

Accession For	
NTIS GRA&I	☒
DTIC TAB	☐
Unannounced	☐
Justification	
By <i>per ADA242479</i>	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
<i>A-1</i>	

Increased Hardness of GE CRD Mg-Alloyed Silver After Air Annealing

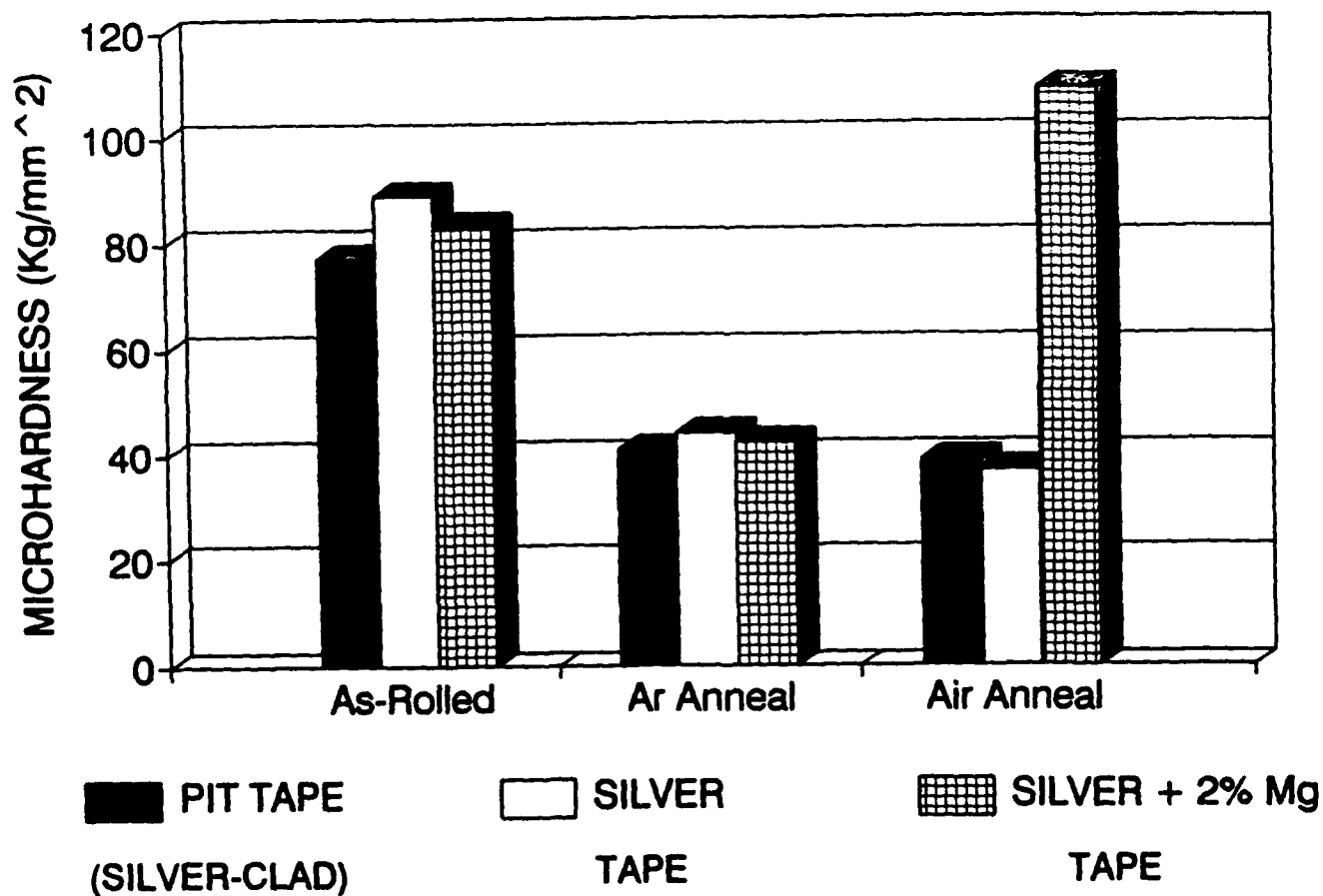


Figure 1 Microhardness of silver cladding on PIT tape, silver tape, and silver + 2 atomic % magnesium tape. Hardness was measured on the surface of tapes as-rolled, after a 30 minute 650°C anneal in argon, or after a 48 hour 830°C anneal in air. The magnesium doped silver is similar to silver during rolling and inert atmosphere annealing so it should be as easily processed into tapes by cold working. It can then be hardened during the final heat treatment of the tapes which are done in oxidizing atmospheres.

Ic versus cooling rate or cooling in oxygen

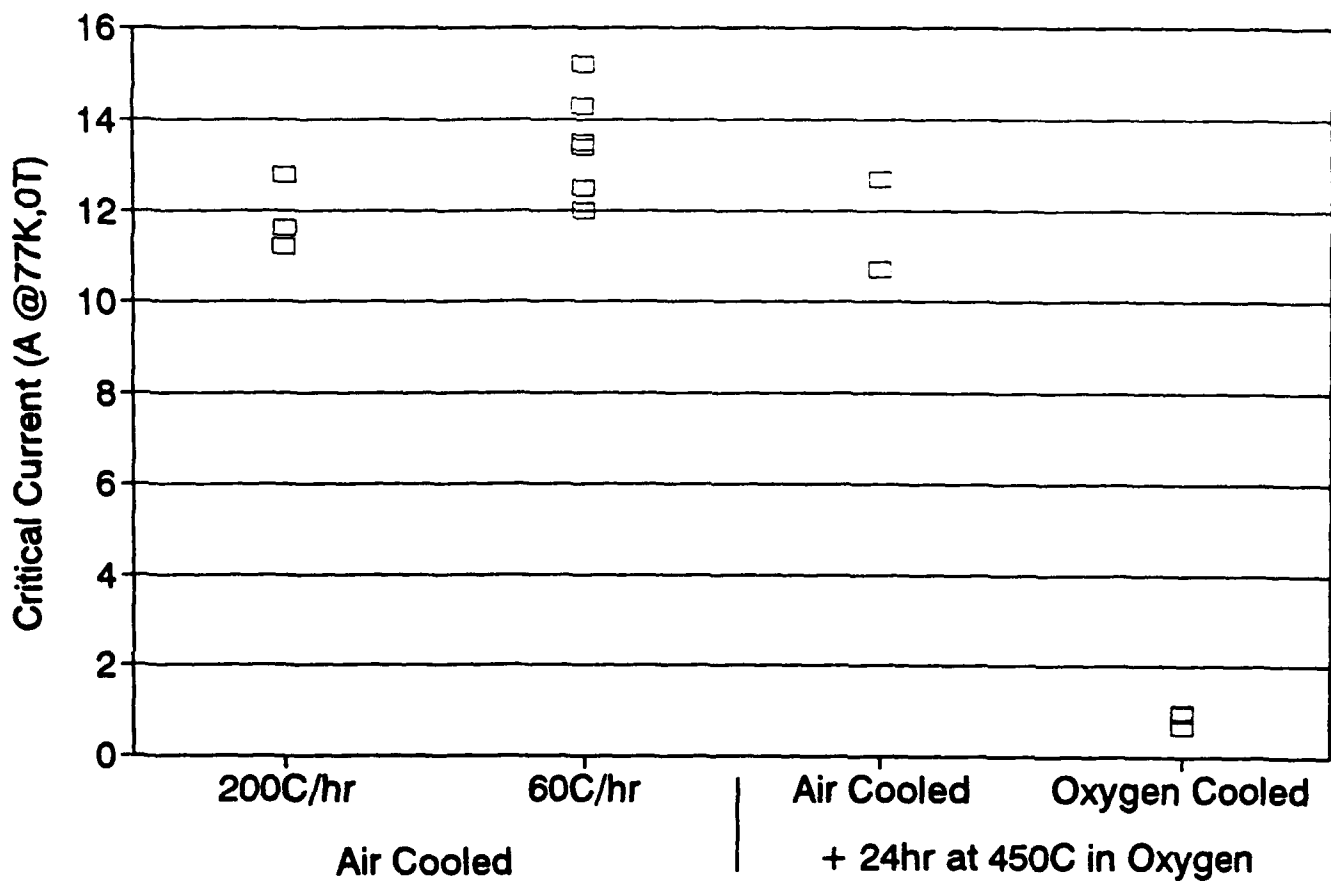


Figure 2 Critical current of a silver clad BSCCO tape as a function of cooling conditions from the third sintering treatment. First two sets cooled in air. Last two cooled in air or oxygen at 60 °C/hr then held at 450 °C in oxygen. Oxygen cooling drastically lowers Ic.

Ic for cooling in either air or low oxygen pressure

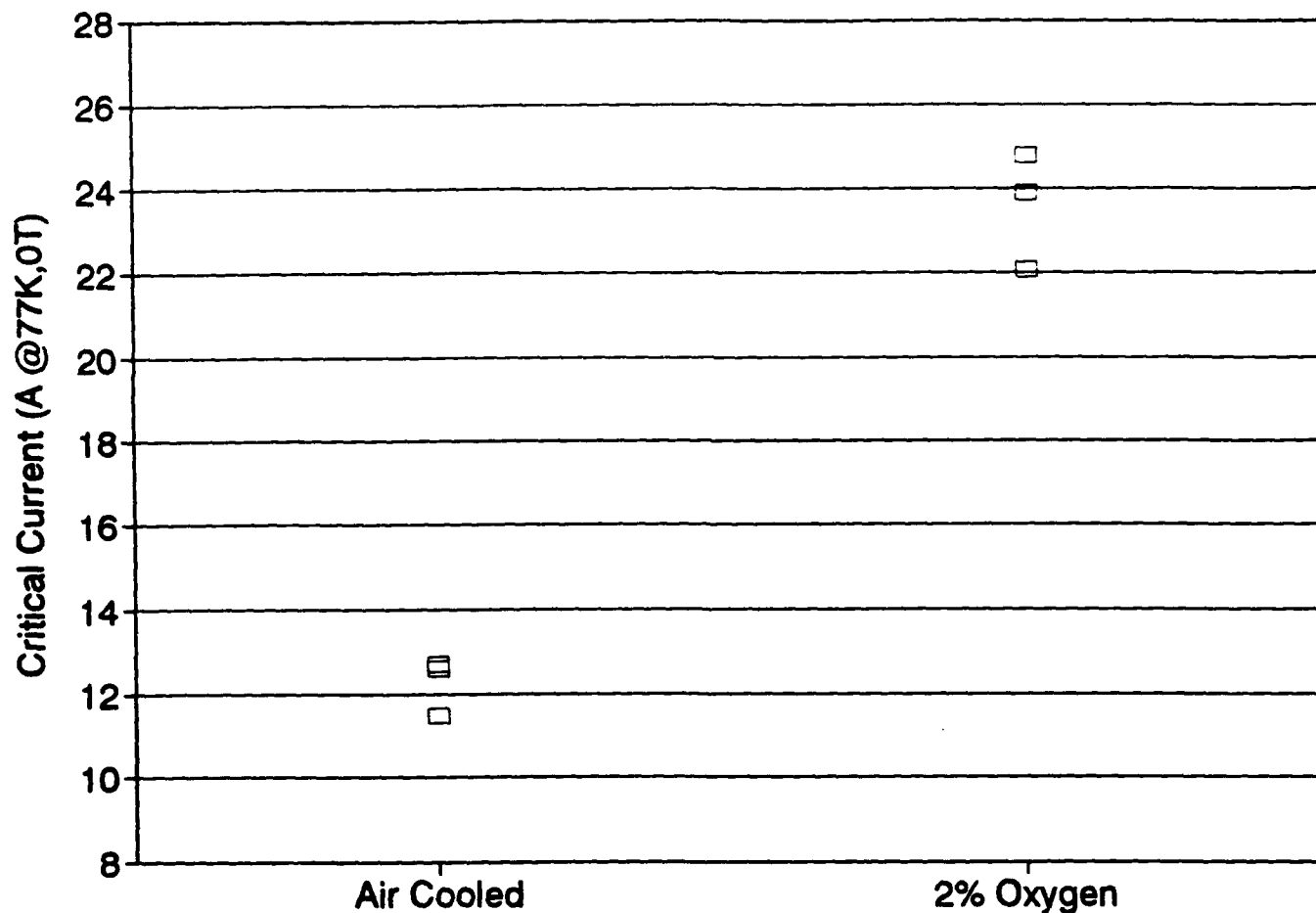


Figure 3 Critical current of a silver clad BSCCO tape as a function of atmosphere during cooling from the third sintering treatment. The first set was cooled at 60 °C/hr in air. The second was cooled to 775 in air then held in 2 % oxygen for 12 hours at 775 °C before cooling in 2 % oxygen. The low oxygen cooling nearly doubled the critical current.

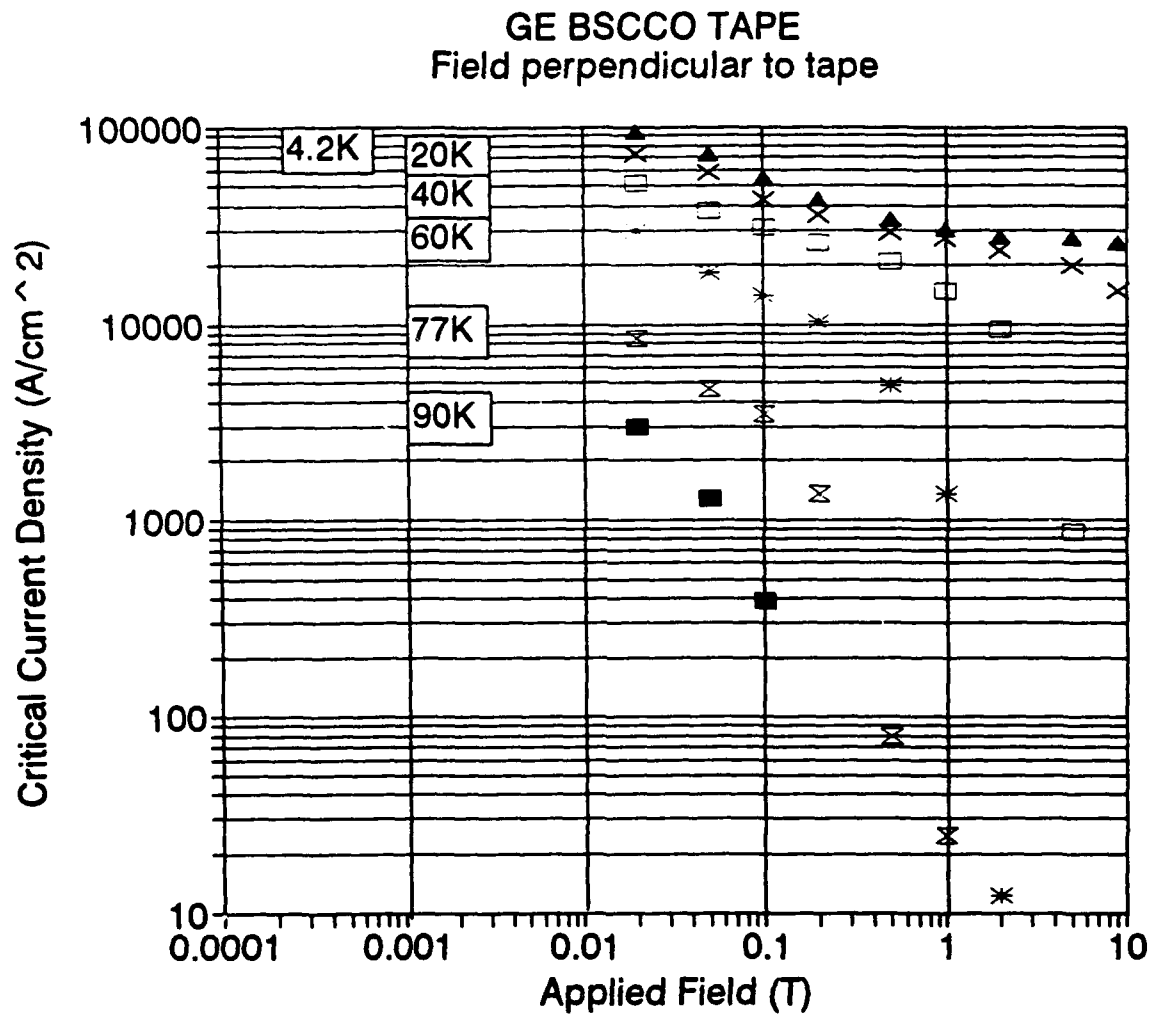


Figure 4 Critical current density of a BSCCO tape as a function of applied field and temperature. The field is applied perpendicular to the tape, the most unfavorable direction. For practical applications a J_c of greater than at least 10^4 will be probably required.

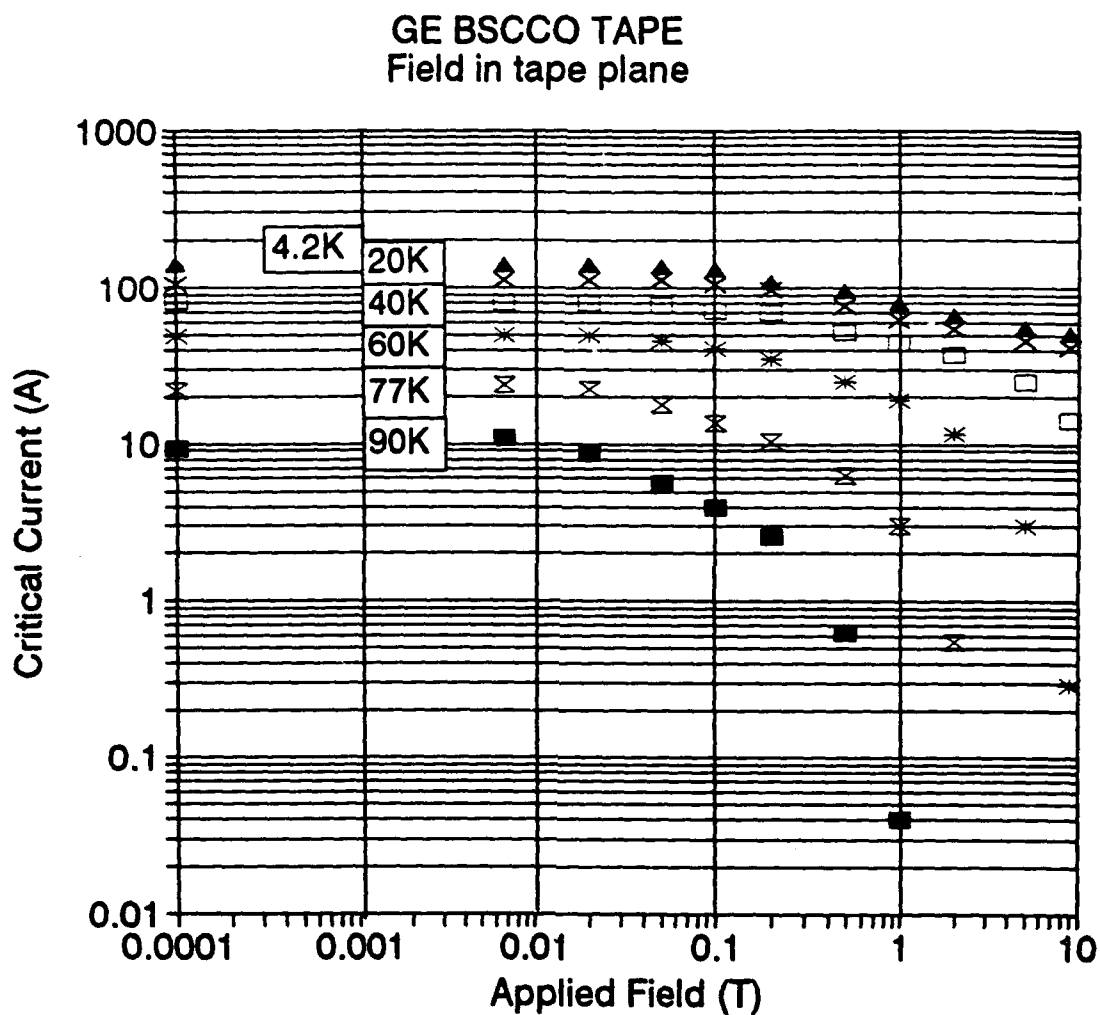


Figure 5 Critical current of a BSCCO tape as a function of applied field and temperature. The field is parallel to the tape, the most favorable direction. Note the drop in I_c for high fields when the temperature is above 20K.

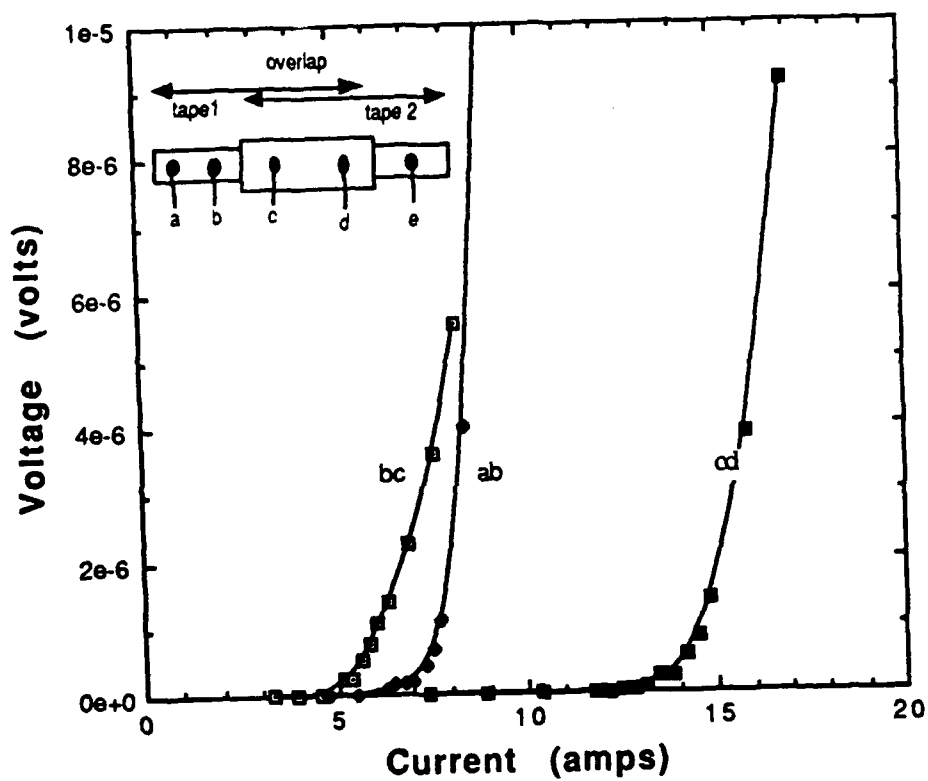


Figure 6 Current voltage curves measured at 77K in the region of a join between two BSCCO silver clad tapes. The I_c at one end of the overlap, bc, is lowered from the tape value, ab. The I_c in the center of the overlap where the core area is doubled is greater than for the original tape.