# Fabrication and Microstructural Development of Ag–Clad $TlBa_2Ca_2Cu_3O_x$ Tapes*

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Argonne National Laboratory, Argonne, IL 60439–4838 USA

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# FABRICATION AND MICROSTRUCTURAL DEVELOPMENT OF Ag-CLAD $TlBa_2Ca_2Cu_3O_x$ TAPES


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Argonne National Laboratory
Energy Technology Division
Argonne, IL 60439


## INTRODUCTION:

One of the most successful fabrication techniques for high-Tc superconductors is the powder-in-tube method, which consists of packing superconductor powder into a silver tube and mechanically deforming the composite into a tape. Microstructural development and critical current density have been explored extensively for Y-, Bi-, and Tl- based superconductors. Early studies on $YBa_2Cu_3O_7$ have shown that critical current density, $J_c$, is limited by weak links that restrict current transport across grain boundaries.[1] Low $J_c$, 600 $A/cm^2$ at 77 K, has been attributed to the absence of grain alignment which causes weak-link behavior.[2]

The effect of processing on microstructure and $J_c$ has been explored extensively for Bi-based superconductors. Large critical current densities, $5.4x10^4$ $A/cm^2$ at 77 K, have been reported for Ag-clad $(BiPb)_2Sr_2Ca_2Cu_3O_{10}$ tapes fabricated by the powder-in-tube method.[3] The $J_c$ values for $(BiPb)_2Sr_2Ca_2Cu_3O_{10}$ are significantly higher than $YBa_2Cu_3O_7$ owing to better texture in Bi-based superconductors. However, a significant decrease in $J_c$ is observed in $(BiPb)_2Sr_2Ca_2Cu_3O_{10}$ for high magnetic fields and is an intrinsic limitation caused by thermally activated motion of magnetic flux lines.

Recent work on thermally activated flux motion of high-Tc superconductors has shown that $Tl_1Ba_2Ca_2Cu_3O_9$ , designated 1223, is intrinsically superior to Bi-based superconductors at 77 K.[4] It is anticipated that if microstructures in 1223 are similar to Bi-based superconductors, then high $J_c$ in high magnetic field are expected. Analogous to $Bi_2Sr_2CaCu_2O_8$ superconductors, the effect of partial melting on grain growth and alignment was studied for Ag-clad 1223 tapes. A significant amount of secondary phase was found in the microstructure and critical current densities of $4x10^3$ A/cm$^2$ were achieved by this method.[5] Solid state sintering has produced $J_c$ values of $6.2x10^3$ A/cm$^2$ for 1223 tapes. These tapes exhibited weak link behavior owing to poor texturing and little grain growth.[6]

A very promising approach in $(BiPb)_2Sr_2Ca_2Cu_3O_{10}$ tape is a two-powder process in which precursor powder is polyphase.[7] Reactive sintering takes place which promotes grain growth and a high degree of phase purity. A similar approach is taken in this study to determine if a polyphase powder will enhance crystal growth. The focus of this work is to develop a processing scheme to develop highly aligned microstructures of 1223 tapes while maintaining superior intrinsic properties.

PROCEDURE:

Three separate powders of the $TlBa_2Ca_2Cu_{3.0}O_9$ stoichiometry were synthesized under different conditions. Powder 1 was phase-pure 1223. Powder 2 was an assemblage of $Ca_2CuO_3$, $BaCuO_2$ and $Tl_2O_3$ phases. Powder 3 was a mixture of 80% powder 1 and 20% powder 2. Separate tapes were made from these powders by the powder-in-tube process.[8]

Powder was packed into a silver tube having an outside diameter of 6.35 mm and a wall thickness of 1 mm. The tubes were sealed on each end and were drawn to 2 mm diameter and rolled into tapes 0.25 mm thick and 6 mm wide. Short sections, approximately 3 cm long, were cut for heat-treatment, $J_c$ measurement, and microstructural analysis.

The annealing temperatures have been selected on the basis of the differential thermal analysis results. Previous work has shown that 1223 incongruently melts at 870°C in the presence of silver.[5] Tapes were heat-treated between 800°C and 870°C for up to 80 h. After each heat treatment step, the critical current density was measured at 77 K with a voltage criterion of 1 µv/cm. Phase development and microstructures were studied by x-ray analysis and scanning electron microscopy.

RESULTS AND DISCUSSION:

The amount of superconducting phase was monitored by x-ray diffraction. Tape 2, which started as a polyphase mixture, transformed to $Tl_2Ba_2CaCu_2O_9$ when heated to 800 °C for 0.5 h. Heating to higher temperatures produced the 1223 phase (Figure 1). Tapes 1 and 3 consisted of the 1223 phase for all heat treatment conditions. No significant decomposition of the 1223 phase was observed from x-ray diffraction between 800 and 870°C.

Microstructural data revealed the presence of secondary phases not detected by x-ray diffraction. Tapes 1 and 3 exhibited similar microstructures, and tape 2 had a significantly larger number of secondary phases (Figure 2). Examination of tape 3 revealed the dark phases to be CaO and $BaCuO_2$. (Figure 3). The secondary phases were not removed upon further heat treatment and coarsened to 5 µm after 1 h anneal at 865 °C. A prolonged heat treatment of 80 h resulted in additional phases that had a plate-like morphology and compositional analysis by energy-dispersive spectroscopy shows that this plate-like phase contains Tl,Ba, Ca, and Cu. This plate-like phase is Ca rich with respect to the 1223 compound. In addition to observing the superconducting core, the silver-superconductor interface was examined as a function of heat treatment time. A significant amount of reaction was found at the interface above 865°C.

Critical current densities were measured for all tapes as a function of annealing temperature and time. Tape 2 had inferior properties to tapes 1 and 3 for all heat treatment conditions, owing to greater number of impurity phases. The critical current density of tape 3 was monitored as a function of soak temperature for times less than and 1 hr, (Figure 4). It was observed that the maximum $J_c$ was for a heat treatment temperature of 865°C which was slightly below the incongruent melting temperature of the 1223/Ag system. $J_c$ decreased as temperature increased from 800°C to 865°C, and was attributed to faster sintering kinetics. The decrease in Jc at 870°C was attributed to reaction with the silver sheath. In addition, $J_c$ was monitored as a function of soak time at 865°C (Figure 5). Microstructural data in Figure 3 suggest that Jc is decreased as the amount of secondary phase increased.

CONCLUSION:

Three different 1223 powders with varied phase distributions were used to fabricated tapes by the powder-in-tube process. It was found that powders consisting primarily of phase pure 1223 were superior to a phase assemblage of $Ca_2CuO_3$, $BaCuO_2$ and $Tl_2O_3$ powders. No texturing in the microstructure was observed for any of the annealing times or temperatures. Critical current densities over 5,000 $A/cm^2$ were measured at 77 K, and $J_c$ degraded as the number of impurity phases increased. Further work will focus on other phase assemblages in the starting powder to determine if grain growth and alignment can be achieved in 1223.


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Figure Captions:

Figure 1. X-ray diffraction patterns for Tape 2. a) heat treated for 0.5 h at 800°C, and b) heat treated for 0.5 h at 850°C.

Figure 2. Microstructures of a) tape 1 and b) tape 2 heat treated for 2 h at 865°C.

Figure 3 Microstructures of tape 3 heat treated at 865 for a) 0, b) 1 and c) 80 h.

Figure 4 Critical current density of tape 3 heat treated for less than 1 h.

Figure 5 Critical current density of tape 3 heat treated at 865°C.

Figure 4

☆ = 1223

O = 2212

□ = $BaCuO_2$

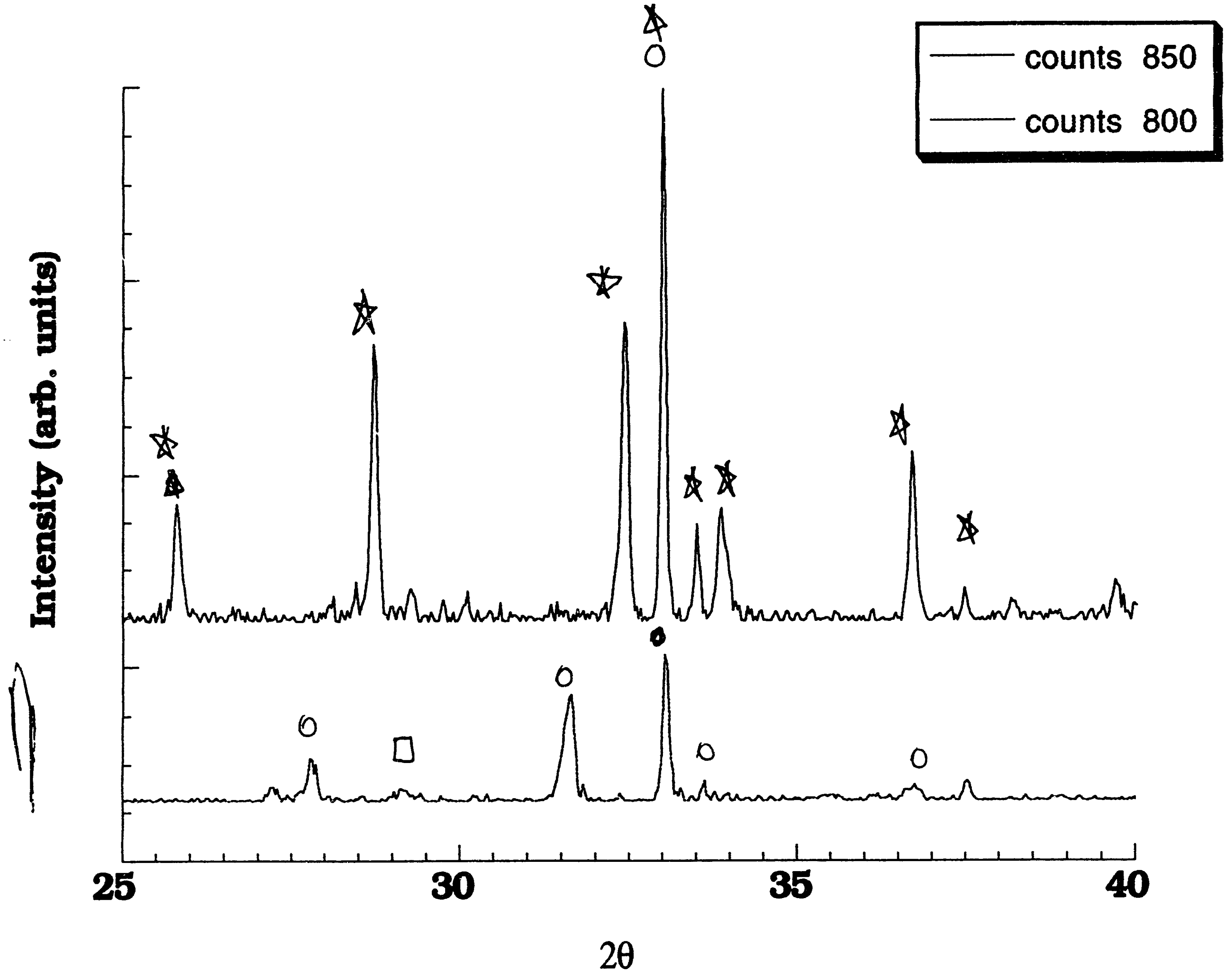
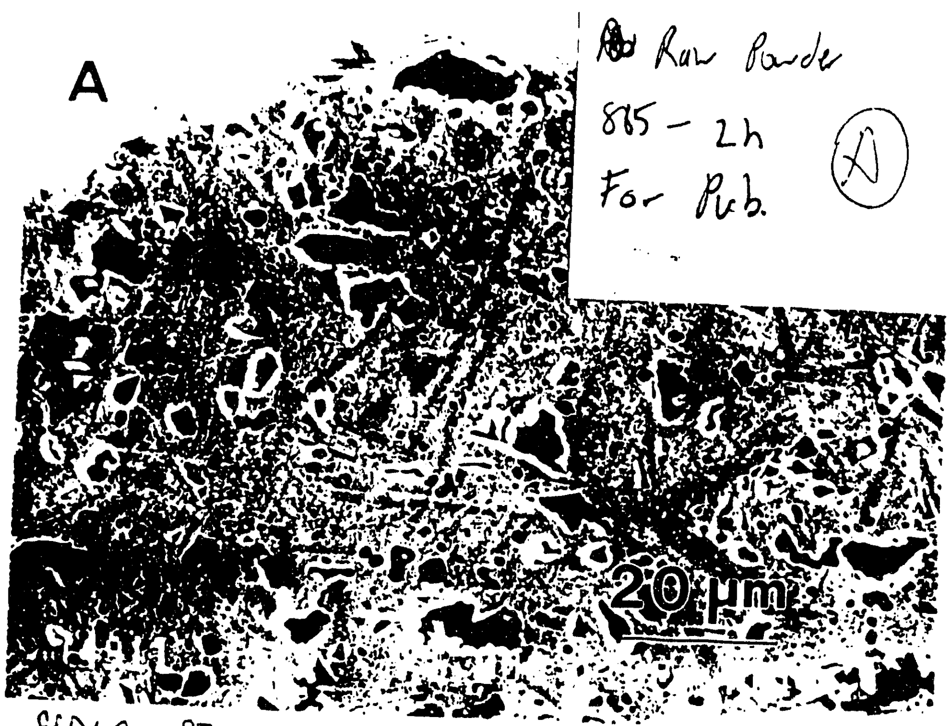


Figure 2

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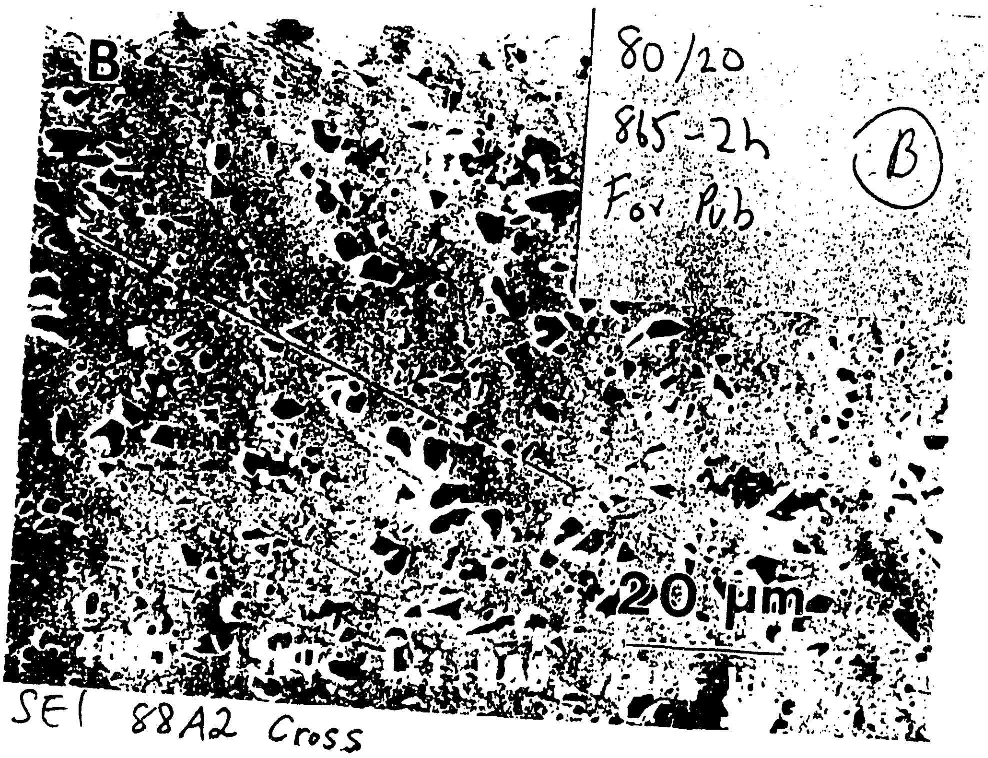


SEI 88A2 Cross


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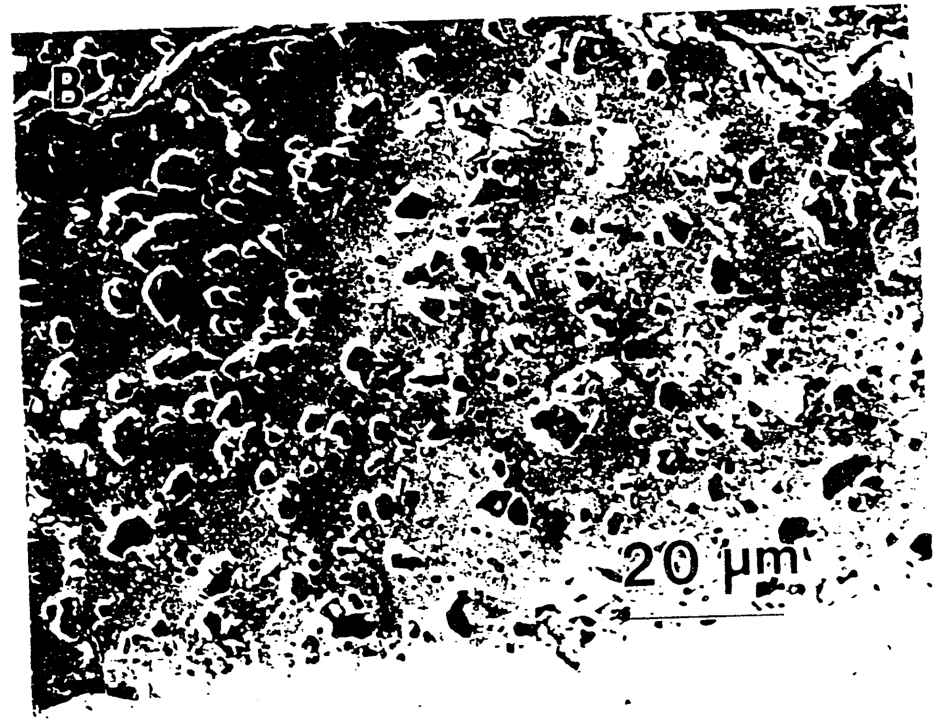



13 mil cross

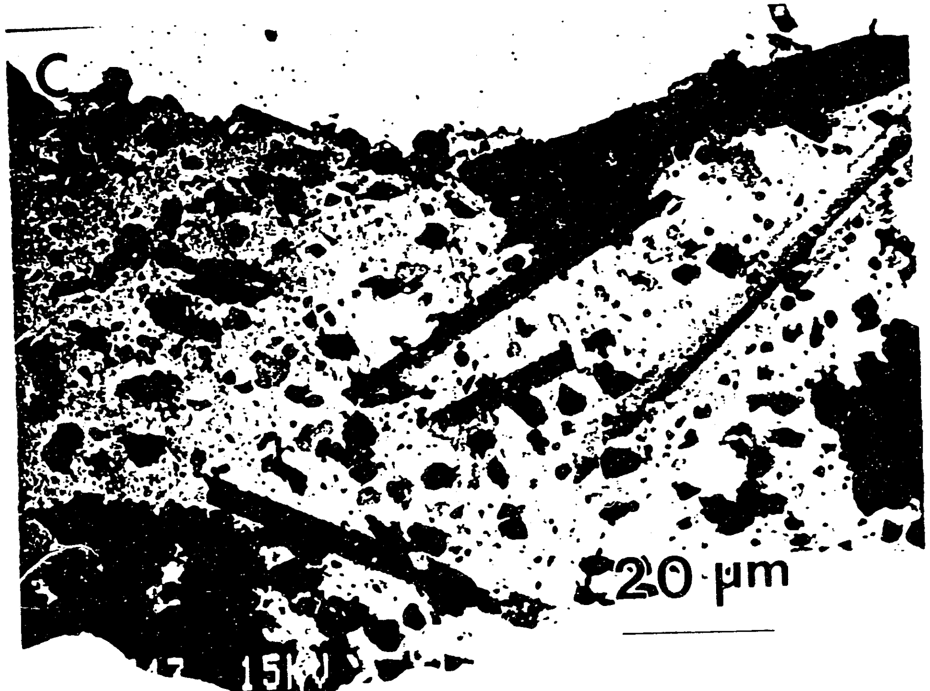



Figure 3

Figure 4

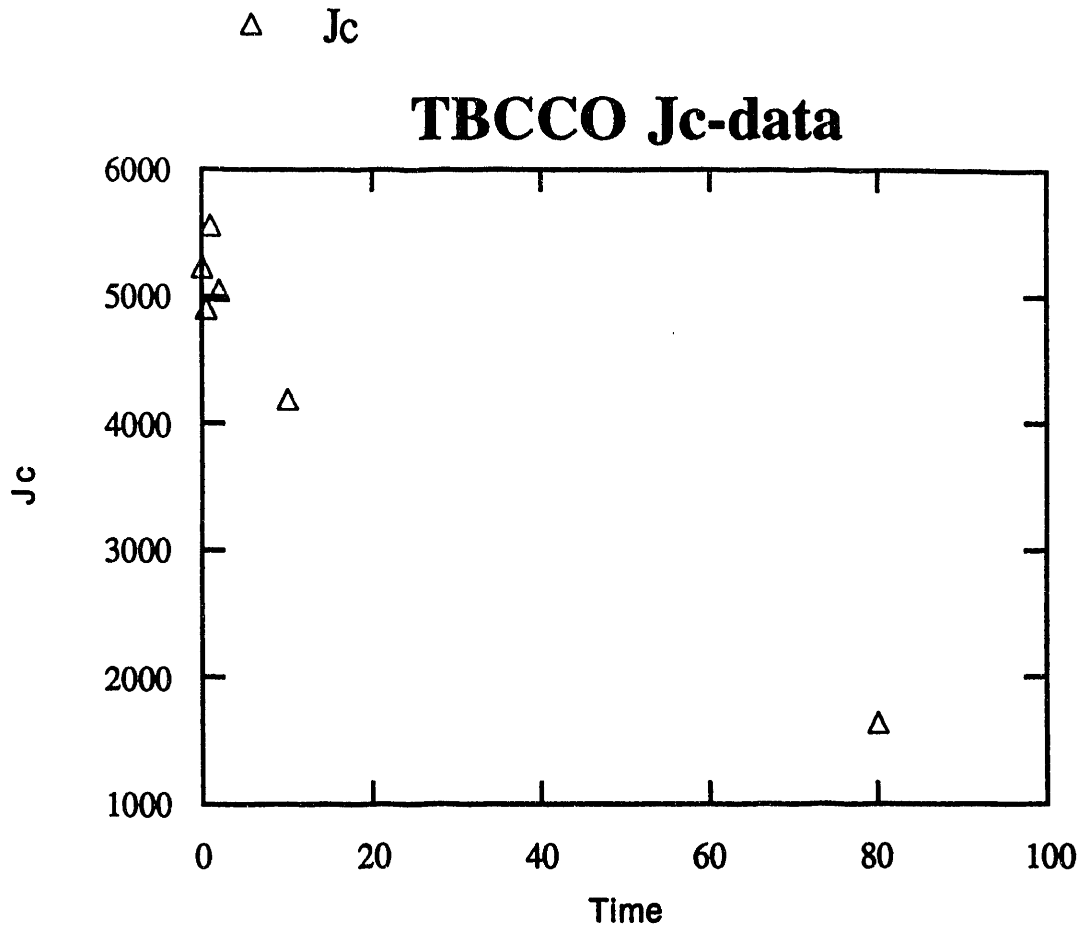
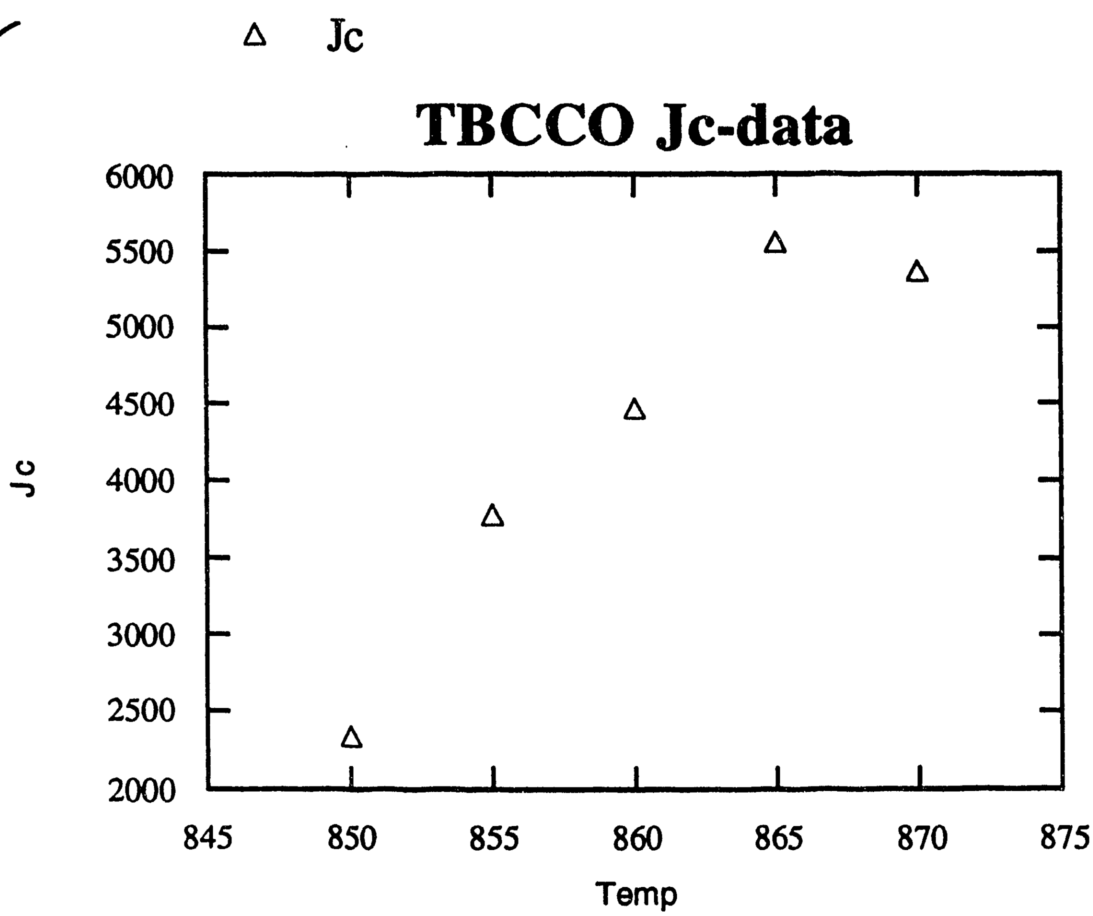



Figure 5



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