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EFFECT OF THERMAL ANNEALING ON GRAIN BOUNDARY CHEMISTRY OF POLYCRYSTALLINE $\text{TiBa}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ FILMS

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Recent studies have shown that spray-pyrolyzed films of the Ti-1223 compound ($\text{Ti}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_y$, with $0.7 < x < 0.95$) on polycrystalline yttrium stabilized zirconia substrates can be prepared which have critical current density J_c near 10^5 A/cm^2 at 77 K, in zero field [1]. The films are polycrystalline, have excellent c-axis alignment, and show little evidence of weak-link behavior. Transmission electron microscopy (TEM) studies have shown that most grain boundaries have small misorientation angles. It has been found that the films have a high degree of local texture indicative of colonies of similarly oriented grains [2]. It is believed that inter-colony conduction is enhanced by a percolative network of small angle boundaries at colony interfaces. It has also been found that J_c is increased by a factor of 4 - 5 after the films were annealed at 600°C in oxygen. This study is thus carried out to determine the effect on grain boundary chemistry of the heat treatment.

TEM studies were performed at 100 kV in a Philips EM400 TEM/STEM equipped with a field emission gun (FEG), which generates an electron probe smaller than 2 nm in diameter. Grain boundary (GB) chemistry was determined by nano-probe energy dispersive X-ray spectroscopy (EDS) and high-resolution electron microscopy (HREM).

Both twist and tilt boundaries have been observed. The twist boundary is the flat (001) basal plane and is parallel to the substrate surface. It is possible to orient the twist boundary edge-on, permitting nano-probe microanalysis across the boundary. The tilt boundary, however, is normally rough. The grain aspect ratios appear to be ~ 10 . The cation compositions were determined when the electron probe was pinned on the boundary, the left-hand grain or the right-hand grain. Before thermal annealing, the twist grain boundaries are slightly Ti-rich and Ca-deficient (figure 1). After thermal annealing at 600°C in oxygen, the boundaries show significant Ti-enrichment and Ca-deficiency (figure 2). The Ti content can be as high as 40% more than that in the bulk. The Ba and Cu contents, however, are almost unaffected by thermal annealing. In general, it is believed that the conductivity of a grain boundary decreases if the Ti content increases. Since J_c increases dramatically after thermal annealing, it is unlikely that the twist boundaries are the effective part of the path for the conduction of supercurrent. These results do not support the "brick-wall" model [4] for long-range conduction in the Ti-1223 films, in which the current transport from grain to grain is accommodated by the large-area twist boundaries between grains regardless of their misorientations.

Large planar stacking faults, lying in the (001) plane and running across the entire grain, have been observed. But the density of stacking faults was almost unaffected by the thermal annealing. The stacking faults are sometimes equally spaced at a separation of $\sim 9 \text{ nm}$. EDS shows that the stacking faults involve Ti enrichment. Besides dislocations associated with the secondary phases (Ca-O and Ca-Cu-O), no other major defects have been observed. HREM shows that the grains tend to terminate with Ti-Ba layers at twist boundaries.

In conclusion, the enhancement of J_c in Ti-1223 films does not result from improvement of the properties of twist grain boundaries. The result does not support the "brick-wall" model [3]. The microstructure has been shown from X-ray diffraction and electron back-scattering diffraction studies to consist of colonies of grains with similar but not identical basal plane orientations, and it has been suggested that long-range conduction occurs through a percolative network of small-angle tilt boundaries at colony intersections [2,4].

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3. A.P. Malozemoff in High Transition Temperature Compounds II, S.H. Whang, A. Dasgupta and R.B. Laibowitz (TMS, Warandale, PA, 1990) P.3.
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FIG. 1 -- Chemical composition across grain boundaries determined by nano-probe EDS before sample was treated at 600 °C in O₂.

FIG. 2 -- Chemical composition across grain boundaries determined by nano-probe EDS after sample was treated at 600 °C in O₂.

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