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**MICROSTRUCTURE OF ARTIFICIAL 45° [001] TILT GRAIN BOUNDARIES
IN YBCO FILMS GROWN ON (001) MgO***

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Microstructure of artificial 45° [001] tilt grain boundaries in YBCO films grown on (001) MgO

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It is well known that high-angle grain boundaries in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) show weak-link effects and behave as Josephson junctions. This kind of grain boundary junction (GBJ) has potential applications in magnetic field measurement and electronic devices. This work studies the microstructure of artificially made GBJs in YBCO films on (001) MgO and the mechanism of the boundary formation, with the goal to improve the GBJ quality and obtain a better understanding of the junctions' transport properties.

Ion-sputter-induced epitaxy¹ is used to form YBCO films with isolated 45° [001] tilt grain boundaries. Prior to YBCO film growth the (001) MgO substrate is selectively sputtered by a low energy Ar ion beam. A portion of the substrate remains non-sputtered by protecting the surface with a patterned photoresist mask. After removing the mask, a YBCO film is grown on the substrate using pulsed organometallic molecular beam epitaxy (POMBE)². Under suitable conditions single crystal YBCO c-axis films can be reproducibly obtained in both the sputtered and non-sputtered regions. The orientation between the films and the substrate has been examined by both x-ray diffraction and electron diffraction. The in-plane orientation relation is $[110]_{\text{YBCO}}//[100]_{\text{MgO}}$ on the non-sputtered epitaxially polished MgO and $[100]_{\text{YBCO}}//[100]_{\text{MgO}}$ on the sputtered MgO. Thus, 45° tilt boundaries are formed in the film at the boundary between the sputtered and non-sputtered substrate regions.

TEM samples are prepared by mechanical polishing and Ar ion milling. Care is taken to keep them from exposure to moisture and elevated temperature. The samples are examined in a JEOL-4000EXII and a Hitachi HF2000. Fig. 1-2 are plan-view images of the boundary. There is no second phase on the boundary except for a few Y_2O_3 particles often observed in YBCO films. One of the most obvious feature of the boundary is that it meanders across the straight line defined by the photoresist. It is easy to see in dark field TEM image (fig. 1) that the interpenetration between the two sides can be over 100 nm. Another important feature is the preference for asymmetric facets at which the (100) plane on one side is parallel to the (110) plane of the other side. About 70% of the boundary is formed by this kind of facet. A cross-section view of a typical boundary is shown in fig. 3. At the edge of the sputtered region there is a small step formed by the ion sputtering (indicated by an arrow). The 45° boundary, indicated by the curved black line, starts about 40 nm from the edge of the sputtered region. The boundary is initially inclined but becomes practically pure tilt at a distance of 20 nm from the substrate. The vertical features at the boundary are Moiré fringes which is believed to be caused by the crystal overlap in the beam direction due to the meandering of the boundary. The fact that the GBs are free of secondary phases and formed by well connected asymmetric facets explains the relatively high critical current supported by the sputter-induced GBJs. The boundary meandering and inclination may in part be responsible for the complex behavior in the magnetic field dependence of the junction critical current³.

The microstructural difference of the YBCO/MgO interfaces in the sputtered and non-sputtered regions is shown in fig. 4 and 5. The interface in the non-sputtered region is flat, sharp and free of precipitates. The periodical features indicated by arrows are caused by interfacial dislocations. Except in the immediate vicinity of the interface no distortions are observed in either sides of the interface, presumably because most of the strain caused by the lattice mismatch is relieved by the interfacial dislocations. The interface in the sputtered region, however, is relatively rough. Between YBCO film and MgO substrate there is an intermediate layer of 2-5 nm thick. Rutherford backscattering spectroscopy of the as-sputtered MgO surface indicates that the ion sputter process implants an amount of W and Ar corresponding to 0.8 and 1 monolayer, respectively, into the top 3 nm of the surface. The source of the W is the hot W filament in the Ar ion gun. High-resolution EDX confirms that W is present in the interface layer of the sputtered region. The detection of W in the interface region is significant in that it may play an important role in the mechanism of the sputter-induced rotation discussed by Buchholz et al⁴. The initial growth of the film may be affected by the W present in the surface layer of the MgO substrate. This is still being investigated.⁵

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