

Twin boundaries and critical current densities of $\text{YBa}_2\text{Cu}_3\text{O}_7$ thick films fabricated by the BaF_2 process

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Abstract

2 μm thick epitaxial $\text{YBa}_2\text{Cu}_3\text{O}_7$, YBCO, films on SrTiO_3 substrates were prepared at the reaction temperatures of 725, 750 and 800 $^\circ\text{C}$ by the BaF_2 process. The dc critical current densities of these films were found to vary by a factor of 2 at 77 K in perpendicular dc magnetic fields measured up to 1.5 T. The twin boundary spacing was essentially unchanged among these films while the size of Y_2O_3 precipitates in the films varied by a factor of ~ 5 . Also, the measured ratios of the lattice parameters, a/b , of the films were surprisingly larger than those found in standard fully oxygenated YBCO. This unusual property of the films appeared to be caused by the presence of the Y_2O_3 precipitates which provided a strain constraint in limiting the full tetragonal-to-orthorhombic phase transformation due to the oxygenation. Differences in the degree of the constraint possibly led to the observed difference in J_c among these films.

1. Introduction

Since the discovery of superconducting $\text{YBa}_2\text{Cu}_3\text{O}_7$ (YBCO), with critical temperature T_c well above 77 K in 1989, critical current densities J_c of epitaxial films, prepared by various methods, have been extensively studied [1]. Interestingly, it was shown that while thin films ($<1 \mu\text{m}$) of YBCO deposited on single crystalline substrates can sustain very high values of J_c , over several times 10^4 A mm^{-2} in self field at 77 K, the value of J_c decreases to $\sim 1 \times 10^4 \text{ A mm}^{-2}$ for the films a few μm thick [2]. Although this systematic study of the J_c -dependence on the thickness of the YBCO films was carried out for films formed *in situ* by pulsed laser deposition, PLD, a nearly identical thickness dependence was also observed for epitaxial films prepared by the so-called BaF_2 process, an *ex situ* reaction process [3]. At present, neither the nature of the crystalline defects which pin flux lines in these high J_c films, the mechanism(s) for flux pinning, nor the above thickness dependence are known despite the importance of such understanding for the planned applications of YBCO coated conductors for future electric systems.

A number of possibilities for the relevant crystalline structural defects in these films were proposed as flux-line

pinning centres to sustain these high values of critical current densities. Earlier, Hylton and Beasley [4] suggested that a high density ($\sim 10^{25} \text{ m}^{-3}$) of nanometre-sized pinning centres might be responsible for the high current densities in very thin films. Recently a detailed study by van der Beek *et al* [5] of the J_c of thin ($<500 \text{ nm}$) PLD films was carried out as a function of magnetic field amplitude angle, temperature and film thickness. Contrary to the Hylton and Beasley estimate for the pinning centres, van der Beek *et al* concluded that the primary pinning centres in these films were sparsely distributed non-superconducting particles, such as Y_2O_3 , of a few tens nm in diameter and 10^{21} – 10^{22} m^{-3} in density. On the other hand, it is well known that there is a substantial number of grown-in dislocations in these films [6]. Thus, based on the measurement of the densities of the threading dislocations in PLD films and of J_c at low temperatures, it was claimed that these dislocations were the primary factor contributing to the high current densities in these films at low temperatures [7]. However, in general, there is no peak in the angular dependence of $J_c(H)$ at low temperature in similarly processed films when applied magnetic fields are aligned along the dislocations [1, 5], and thus it is questionable whether the relatively low density dislocations (10^{13} – 10^{14} m^{-2}) in these films are the major pinning centres, particularly at low temperatures.

Twin boundaries are the most commonly observed structural defects in YBCO. They are formed as the result of a tetragonal-to-orthorhombic structural phase transformation during the oxygenation process of YBCO, and the formation of twins in bulk and films of YBCO is well documented [8, 9]. Also, the twin boundaries are shown to consist of thin (~ 1 nm) structurally disordered and oxygen-hole depleted regions [10]. Either way, these regions could be either strong flux pinning centres or weakly coupled planes. Thus, the boundaries are likely to influence the values of J_c in these films. However, it was reported earlier that changes in the twin boundary density in YBCO films which were prepared *in situ* by electron beam evaporation did not result in a systematic change in critical current densities at low temperatures [11]. This somewhat surprising observation is due to the fact that other pinning centres, e.g., high densities of very fine structural defects which are present in these films deposited *in situ*, become more dominant pinning centres than the twin boundaries at the low temperatures (~ 5 K) where J_c was measured in this study. The possible dominance of the pinning by small defects rather than planar ones at low temperatures can be seen from, for example, the absence of the peak in the angular dependence in $J_c(\theta)$ at $H \parallel c$, i.e. $\theta = 90^\circ$, at low temperatures, while it was observable at high temperatures, e.g., 75 K [1].

In order to clarify the effect of twin boundaries on critical current densities of YBCO at high temperatures, the motion of flux lines due to the Lorentz force was investigated in detail by Kwok *et al* [12] using single crystals in which the twin boundaries were uni-directionally oriented. These experiments showed that planar boundaries could be strong pinning sites when the flux lines were pushed against the boundaries, while they could act as ‘channels’ for easy flux motion when the Lorentz force is directed along the boundary planes. More recently, a very interesting experiment was performed by Safar *et al* [13] demonstrating that the direction of the Lorentz force on the flux lines with respect to the orientation of the twin planes is indeed important in determining the values of J_c in YBCO films which were deposited on buffered metallic substrates by PLD. The YBCO grains and the misorientation angles between adjacent grains were very small in these films. In addition, very closely spaced twin boundaries were unidirectional within each grain, but many of them were perpendicular to those in adjacent grains across the low angle grain boundaries. It was found that J_c in these films was greater when the Lorentz force was directed at $\sim 45^\circ$ to the twin boundary planes compared to when it was perpendicular and parallel to the planes. The difference was nearly by a factor of 2 at 75 K with an applied magnetic field of 5.2 T along the c -axis of the film. It was argued that those boundaries which are parallel to the Lorentz force acted as easy flux flow channels in reducing J_c . On the other hand, when the Lorentz force was directed at 45° to the boundary planes, J_c was greater since the effective force along the channel was reduced by a factor of $\sqrt{2}$ in this case relative to the other orientation. These studies clearly demonstrated that twin boundaries could play an important role in providing locally weak or strong regions in YBCO films at high temperatures, e.g., 77 K. Since the majority of applications of thick YBCO films would be utilized at high temperatures, it is important to develop a better understanding

of the role of the twin boundaries in determining J_c in thick YBCO films ($> 1 \mu\text{m}$).

The so-called BaF₂ process [14, 15] for the synthesis of YBCO films is considered to be one of the primary candidates for large scale production of YBCO conductors for electric utility applications. In spite of this, detailed studies have not yet been carried out on the relationship between the defect structures and J_c in thick YBCO films made by this process. Thus, in order to improve upon this situation, we have carried out a study characterizing critical current densities and microstructures of such films in order to investigate the possible role of the twin boundaries in determining J_c . In this paper, we primarily report the results obtained from $2 \mu\text{m}$ thick films, but also include some results from $5 \mu\text{m}$ thick YBCO films [3] to facilitate the discussion of the possible role of the twin boundaries on J_c of these thick films.

2. Experimental procedures

In this study we utilized the so-called BaF₂ process for synthesis of YBCO films. This process consisted of the co-deposition of the precursor films, made up of fine grained Y, Cu and BaF₂, by electron beam evaporation on [100] oriented SrTiO₃ single crystals ($3 \times 10 \text{ mm}^2$) and of heating them in a quartz tube at 725–800 °C in a flowing process gas at atmospheric pressure. The gas was a mixture of O₂, H₂O and N₂, and its composition was $p(\text{O}_2) = 100 \text{ mTorr}$, $p(\text{H}_2\text{O}) = 60 \text{ Torr}$ and the balance being N₂. For this study, films with a thickness of $2 \mu\text{m}$ were heated at 725, 750 and 800 °C, and the times to fully react the precursors into YBCO were 6, 5 and 2 h, respectively. In order to ensure that the compositions of these films were identical, all of the films were deposited during a single evaporation run with constant deposition parameters. This was accomplished by rotating each substrate in sequence over the evaporation sources with the use of a carousel, which held all of the substrates. Also, the depositions were made such that the amount of Y in the films was somewhat in excess ($\sim 5\%$) of the stoichiometric composition of YBCO. $5 \mu\text{m}$ thick films were prepared separately and were heated at high water vapour pressures to avoid the a -axis oriented grains [3]. Details of the deposition and synthesis methods for YBCO films [3, 16] and of the kinetics of YBCO formation in this *ex situ* process were previously discussed [17].

Critical temperatures T_c of these films were resistively determined and were all ~ 90 K. dc critical currents I_c were measured using a standard four-probe method in liquid nitrogen and with applied magnetic fields, perpendicular to the film, (\parallel to the c axis of the film), up to 1.5 T. The fields were provided by a home-made electromagnet operating in liquid nitrogen. The electric field criterion for I_c was 0.1 mV m^{-1} . Specimens for transmission electron microscopy were prepared for planar views of the films after the superconducting properties of the films were determined. The transmission electron microscopy was carried out using a JEOL 3000 FX to determine the twin boundary spacing and the Y₂O₃ particle size. Also, the ratios of the a - and b -axis lattice parameters were determined from diffraction patterns taken with the electron beam directed along the c -axis of the films. Standard θ - 2θ x-ray diffraction scans were performed to ensure that the films were c -axis oriented and to determine the c -axis lattice parameters of the films.

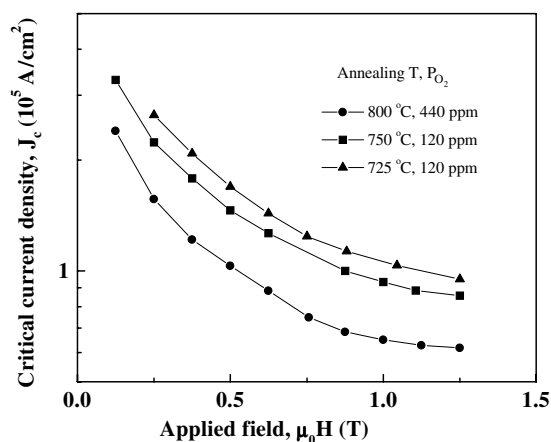


Figure 1. Critical current densities, measured at 77 K in perpendicular dc magnetic fields, are compared for three films which were heated at 725, 750 and 800 °C.

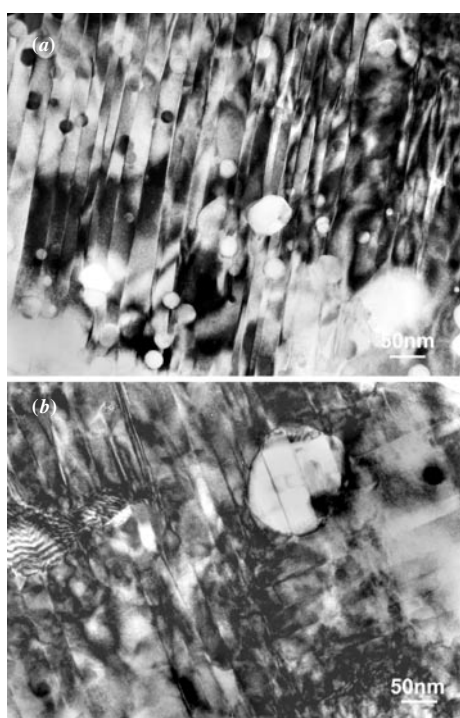


Figure 2. A planar view of YBCO films by transmission electron microscopy showing the twin boundaries and Y₂O₃ precipitates. These were reacted at (a) 725 and (b) 800 °C, respectively.

3. Results and discussions

As shown in figure 1, the critical current densities of the three films varied approximately by a factor of 2 from the highest to the lowest. In order to see whether this variation is correlated with the twin boundary spacing, the average spacing for each film was measured from the TEM images. Examples of the images are shown in figures 2(a) and (b). These were taken from the films which were heat treated at 725 and 800 °C, respectively. They also illustrate the presence of Y₂O₃ precipitates in the film in addition to the twin boundaries. Although other precipitates such as Y₂Cu₂O₅ were occasionally seen, twin boundaries and Y₂O₃ precipitates

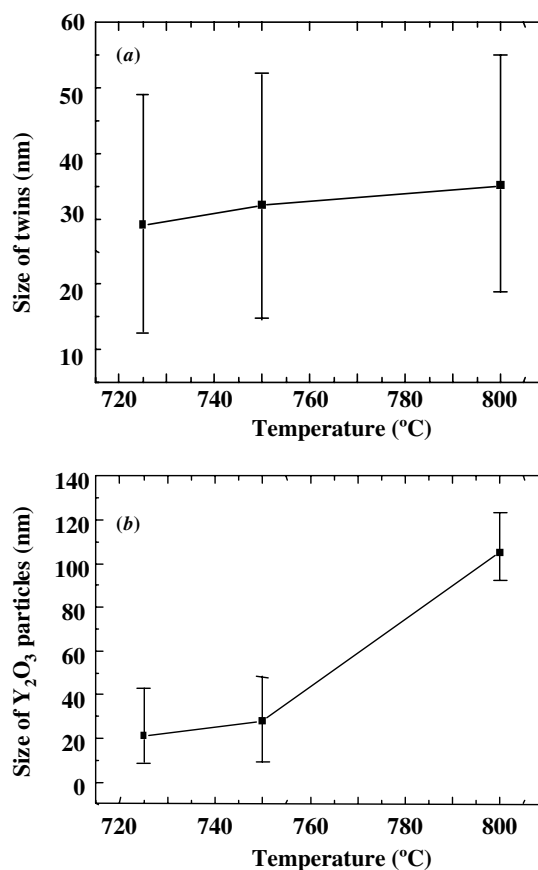


Figure 3. (a) The twin boundary spacing as a function of temperatures for the YBCO formation, and (b) the diameters of Y₂O₃ precipitates as a function of temperatures for the YBCO formation.

were the primary structural defects seen at these magnifications in these films. As shown in figure 3(a), the average twin boundary spacing was essentially unchanged with the reaction temperatures while it had a large variation within a given film. The observed nearly constant twin boundary spacing among these films is consistent with the idea that the twin boundary spacing is primarily controlled by the smallest dimension of the YBCO grains [18–20]. In this case, it is the thickness of the films, and they are the same. Thus, the boundaries do not appear to be directly controlling the values of J_c of the films since the spacing hardly changed among these films while the values of J_c varied by a factor of 2 between the films reacted at 725 and 800 °C. However, as shown in figure 4 and observed by others [1, 21, 22], at these elevated temperatures there is a small enhancement of J_c when magnetic field direction is parallel to the c -axis. Also, the nucleation and growth mechanisms for YBCO films in this process [17] are very different from those in PLD processed YBCO films [7], and there aren't sufficient threading dislocations parallel to the c -axis in the interior of the grains to produce such an enhancement of J_c at these fields. Furthermore, the grains in these epitaxial films are so large that the grain boundaries were hardly encountered in TEM studies. Thus, we can attribute the peaks observed in the data such as those shown in figure 4 as a consequence of having twin boundaries in the films. Possible mechanisms for it will be discussed below.

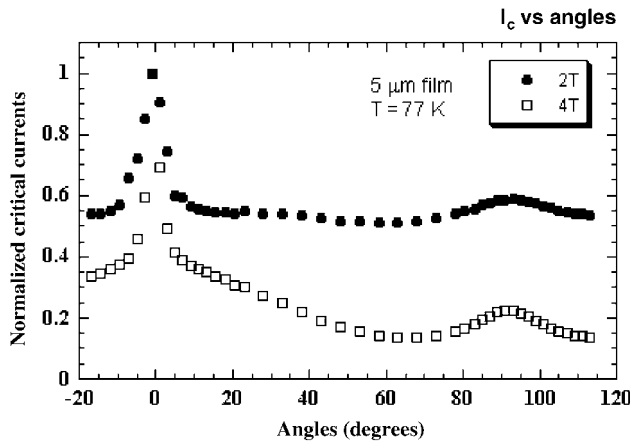


Figure 4. Normalized critical currents as a function of the angles between the direction of applied magnetic fields and the plane of the film for a $5\ \mu\text{m}$ thick YBCO film reacted at $720\ ^\circ\text{C}$. At the angle of 90° the field is oriented parallel to the twin boundaries.

Significant changes with the reaction temperatures were observed in the size of Y_2O_3 in these films. This is shown in figures 2 and 3(b). Interestingly, the size of the precipitates varied by a factor of 5 between the films which were heated at 725 and $800\ ^\circ\text{C}$. This implies that the particle densities of Y_2O_3 differ by a factor of over 100 since the excess Y content over the stoichiometric composition was kept identical in these films. Interestingly, the size and the estimated densities of the particles in the films heated at 725 and $750\ ^\circ\text{C}$ are in the range of those which were suggested to be the primary pinning centres in the films by PLD of [5] in order to describe the observed increases in J_c with increasing thickness of the films from 100 to ~ 300 nm. Thus, this change is a possible candidate for the difference in J_c among these films. However, it is difficult to argue for a difference in J_c of only a factor of 2 when the density of the particles decreased by over 100 while the volume of the average particles also increased by over 100. In addition, these circular (presumably spherical) precipitates will not lead to an orientational anisotropy of J_c with respect to the direction of applied fields as seen in figure 4. Hence, it is difficult to attribute the variations in J_c of these films simply to the enhanced flux pinning due to the increased density of Y_2O_3 particles in the films heated at lower temperatures.

Even though we cannot directly relate the difference in J_c in the present films to the twin boundary spacing, one could consider the following possibility which involves both the twin boundaries and the Y_2O_3 particles in determining the values of J_c in the films. We recall that the twin boundaries can be channels for the easy flow of flux lines, and the critical current densities of YBCO films produced by PLD were increased by reducing the component of the Lorentz force on the flux lines resolved along the twin boundary planes by orienting the force at 45° to the planes [12, 13]. Although the orientations of the boundaries with respect to Lorentz force are identical in the present films, the observed differences in J_c could be achieved by placing pinning centres on the planes to reduce the flux-line flow. (Note that our substrates were cut such that the (100) planes of SrTiO_3 were parallel to the edges of the substrates. Hence, the Lorentz forces on the flux lines in these films were oriented at 45° to the twin boundary planes.) The increased

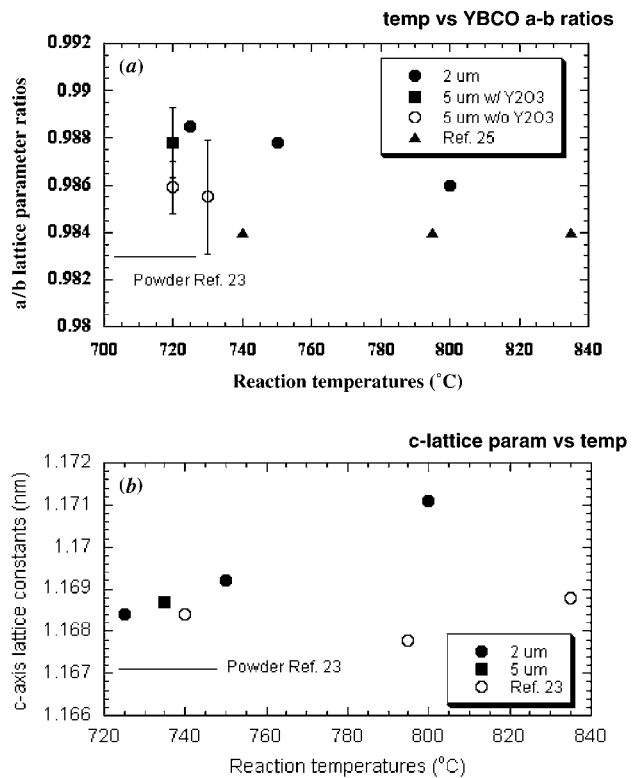


Figure 5. (a) The ratios of the a - and b -axis lattice parameters as a function of YBCO formation temperatures. The ratios for $5\ \mu\text{m}$ thick YBCO films and that for a fully transformed YBCO [25] are also shown. (b) The c -axis of the YBCO films as a function of the YBCO formation temperatures.

density of Y_2O_3 particles in the films which were heated at lower temperatures could cause more of the precipitates to be situated on the boundaries and thus reduce the flow of flux lines along the boundaries. Yet depending on the flux-line flexibility, the density of the precipitates may not be sufficient even for such a mechanism to account for the difference of a factor of 2 in J_c among these films.

Another interesting observation derived from TEM results is that the ratios of the a - and b -axis lattice parameters of YBCO in these films are surprisingly large compared with the fully oxidized sintered powder of YBCO as shown in figure 5(a). It is difficult to determine the precise lattice parameters from electron diffraction patterns, but the ratios can be determined quite accurately from the splitting of the diffraction spots at $(h\ h\ 0)$ due to twinning. Note that this ratio for fully orthorhombic YBCO powder is 0.982 – 0.983 [23]. However, the ratios ranged from ~ 0.9885 to ~ 0.9857 for the films heated at 725 and $800\ ^\circ\text{C}$, respectively. The latter value is nearly identical to the ratio for the so-called ‘ortho II’ phase of YBCO, with an oxygen content of ~ 6.7 and a T_c of ~ 60 K if we attribute this large a/b ratio totally to oxygen deficiency. However, the values of the resistively determined T_c for these films were not reduced appreciably, but were what one expects for the fully oxygenated YBCO, ~ 90 K. Also, the values of J_c for these films are sufficiently high that it is reasonable to expect that the values of T_c measured are representative of the entire area of the films. (Magneto-optical imaging of similar films also confirms this view [24].) One

possible source of these surprisingly large ratios is the epitaxial growth of YBCO on a cubic substrate, SrTiO_3 , which could restrain the orthorhombic transformation. When the YBCO nucleates and grows on a cubic SrTiO_3 in this BaF_2 process, the oxygen stoichiometry of the films is thought to be ~ 6.1 [15] and hence its structure of YBCO is tetragonal at this stage, i.e. $a = b$. When they are oxygenated at a lower temperature, ~ 500 °C, they become orthorhombic. However, because YBCO is hetero-epitaxially bonded to the substrate, the film, particularly the portion adjacent to the substrate, cannot be easily transformed to a structure with the full orthorhombicity which is found in an unconstrained YBCO crystal. However, this effect does not appear to be sufficient to explain these very large ratios. For example, for similarly processed thin (~ 200 – 400 nm) films of YBCO at a temperature range of 740 – 845 °C, this ratio was approximately 0.984 [25] which is only slightly larger than the ‘standard’ values noted above as shown in figure 5(a). Thus, the epitaxial strain from the substrate cannot account for these large a/b ratios found in these films.

Other possible sources for the differences in the a/b ratio between the present films and those in [25] are the presence of Y_2O_3 precipitates in our films, but not in theirs, or possibly the large difference in film thickness between ours and theirs. In order to pursue this possibility, we also used TEM to examine the a/b ratios in a couple of very thick films, 5 μm , which were processed similarly to the present films and studied for superconducting properties [3]. What is revealing is the fact that the variations in the a/b ratios in these films were very large among the number of the measurements as indicated by the error bars in the figure 5(a). In one film heated at 725 °C, we were able to correlate this variation to the location of the films across its thickness. It turned out that the bottom portions of these films are nearly free of the Y_2O_3 precipitates and the a/b ratios are smaller and closer to the values of the standard and those from [23]. At the same time, the top portion had a high density of Y_2O_3 and exhibited larger a/b ratios that were approximately the same as those in 2 μm thick films which were heated at 730 and 750 °C. This variation in the density of Y_2O_3 across the films’ thickness originates in the particular nature of the epitaxial growth of YBCO in the BaF_2 process which involves a liquid layer between the YBCO growth front and the precursor [17]. This liquid plays a role similar to that of the liquid in single-crystal growth by zone refining methods, i.e. the impurity, in this case the excess Y, becomes more concentrated in the liquid as the growth proceeds and thus Y_2O_3 precipitates more at the top portion, and the bottom portion, where the growth begins, is nearly free of Y_2O_3 . Although this phenomenon exists in all of the YBCO films which are fabricated by this process, the variation of Y_2O_3 densities is greater for thick films, particularly for ~ 5 μm , than for thin ones. Thus, in the 2 μm thick films, the gradation in the density of Y_2O_3 could be less drastic or the films are sufficiently thin that the effect of Y_2O_3 apparently extends across the entire thickness in restraining the full reduction of the a/b ratio. This is consistent with the observation of substantially less scatter in the measured ratios for 2 μm films than for the 5 μm films, as shown in figure 5(a). Thus, the observation of unexpectedly large values of the a/b ratios in these films appears to be a consequence of the presence of Y_2O_3

precipitates. In addition, decreased a/b ratios were observed with increasing reaction temperatures as shown in figure 5(a). This could also be attributed to the fact that the number of the precipitates was much less ($\sim 1/100$) in the film reacted at 800 °C, and they were further apart in this film than in the other two films. Thus, these precipitates seem to provide less constraint to the transformation in this case than those for the other two films.

Since the a/b ratios of these films are substantially greater than that for the standard fully-oxygenated YBCO powder, one then might expect significantly larger c -axis lattice parameters for these films than for the standard one if these large ratios in the present films are due to incomplete oxygenation. In figure 5(b), the c parameters as a function of heat treatment temperatures are shown for these films. Also, one from the 5 μm thick films reacted at 725 °C is included in the figure. As shown, the lattice parameter for the film reacted at 725 °C is quite close to the value for the standard one, but it increased monotonically with the increasing reaction treatment temperatures. (The c -axis parameter decreases with the decreasing a/b ratio for the unconstrained YBCO due to increased oxygen uptake.) Thus, this reconfirms that the observed large a/b ratios are not due to the incomplete oxygenation of these films. From the above discussion, it appears that the state of the YBCO is essentially a fully-oxygenated state but the observed large a/b ratios and the c -lattice constants indicate the presence of large mechanical constraints in the films, i.e. the YBCO is in a state of elastic deformation. Yet, the observed values of T_c of the films are without substantial degradation, which is surprising since mechanical strains are known to affect the T_c of cuprates. However, as discussed below the net effect of the multi-axial strain on the values of T_c is small.

It has been shown that uni-axial stresses in the a - or b -axis direction increase and decrease T_c , respectively, in a twin-free single crystalline YBCO [26]. These changes in T_c with opposite signs are approximately the same in magnitude. But the effect of strains along the c -axis on T_c is smaller by an order of magnitude in comparison with those along the a - and b -directions. One can estimate the amount of T_c changes in these films due to strains derived from the observed a/b ratios and c -axis lattice parameters using the linear strain coefficients of T_c of YBCO [27, 28]. In calculating the T_c deviations, we assumed that the films are fully oxygenated and that the deviations in the lattice parameters are due to elastic strains on YBCO crystals in the films arising from the constraints exerted by Y_2O_3 particles. It turns out that the estimated reduction in T_c from the multi-axial strain is of the order of 0.5 K, and this level of T_c reduction in these films would be unnoticed. Thus, the values of the resistive T_c in these films are representative of the entire films and are reasonable in spite of very large values of the a/b ratios and the c -lattice parameters.

The main question here then is whether one can relate the difference in the values of J_c to the observed variations in the a/b ratios in the films which were heat treated at 725 , 750 and 800 °C. In this regard, one can speculate the following scenario. When the a/b ratio is large (i.e. closer to unity) in a strained, but fully oxygenated YBCO, the structural distortion and consequent depletion of the holes at the twin boundaries are expected to be less than would be in the case when the

a/b ratio is fully developed to its full orthorhombicity. This implies that the depression of T_c at the boundaries in the films with larger a/b ratios would be less than for the boundaries with full orthorhombicity. Thus, the flux flow along the boundaries with larger a/b ratios would be less favourable than that in the case of fully developed orthorhombicity. Furthermore, the degrees of the suppressed superconductivity along the boundaries are expected to vary since the Y_2O_3 particles, which provided the constraint to the full transformation, are randomly distributed. Furthermore, the higher density of fine Y_2O_3 particles in the case of low temperature heat treatments adds additional flux pinning within the twin boundaries. Then, the ease of the flux-line flow along the boundaries in these films, heat treated at 725 and 750 °C, might be reduced from that for the boundaries with a smaller a/b ratio and lower densities of Y_2O_3 , e.g., in the film heated at 800 °C. Thus, the twin boundaries are effective flux pinning sites when the flux lines are pushed against the boundary planes, but more importantly the critical currents in these films are likely to be determined by the boundaries' ability to limit flux flow along them.

As described above, the twin boundaries in these films are important in determining J_c , but the enhancements of J_c , when applied fields are parallel to the boundaries, are relatively small, e.g., ~10% or less at 2 T and 77 K (see figure 4). Thus, this implies that there are other small pinning sites in these films which are not obviously observed by standard TEM imaging, but which are quite effective in retaining high values of J_c even at elevated temperatures. What these possible pinning sites are is presently unknown, and the identification of such sites is important in furthering the understanding of the flux pinning mechanisms, as well as for the improvements of J_c of the films for their applications in electric devices.

4. Summary

It was found that the critical current densities of YBCO films fabricated by the BaF_2 process varied by a factor of 2 when the films are heated to form YBCO from the precursor at 725–800 °C. While the twin boundary spacing was unchanged among these films, the size of Y_2O_3 precipitates varied by a factor of 5 (the film heated at 800 °C having the greatest size). Also, the ratios of the lattice parameters, a/b , for YBCO were much greater than that for full orthorhombicity and decreased with increasing heat treatment temperatures. The presence of small Y_2O_3 particles in these films appeared to cause the observed large a/b ratios by constraining the orthorhombic transformation, while not interfering with full oxygenation, during the oxygenation process of the films. It was speculated that this reduced degree of the transformation resulted in a smaller degree of hole density suppression at the twin boundaries and that Y_2O_3 provided non-uniform constraints along the boundaries. These effects possibly resulted in reduced ease of flux-line flow along twin boundaries in the films with larger a/b ratios, and hence in higher J_c in the films which were heated at low temperatures. Also, it appears that there are other small defects in these films which provide background isotropic flux pinning in these films, but which cannot easily be identified in TEM.

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