Evolution of morphology, crystallinity, and growth modes of thin superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ films on SrTiO_3 and NdGaO_3 substrates

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The evolution of surface morphology, crystallinity, and growth modes of *c*-axis-oriented superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) thin films deposited by pulsed laser deposition on $\text{SrTiO}_3(100)$ and $\text{NdGaO}_3(110)$ substrates have been systematically investigated with scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM), x-ray energy-dispersive spectroscopy, and x-ray diffraction. SEM and AFM images of the YBCO films grown on NdGaO_3 with a sequential thickness revealed that the films were rather smooth and grew in a two-dimensional manner. In contrast, the YBCO films of the same thickness on SrTiO_3 were found to have spiral growth features, even in the thinnest film (10 nm or 8 unit cells thick) indicating that an island growth mechanism took place. A change of the full width at half-maximum of the rocking curves of the (005) reflection with the film thickness showed that the crystallinity of the films on NdGaO_3 was much better than those grown on SrTiO_3 , suggesting that a lattice match is a crucial factor to obtain good crystallinity. In the early stages of growth the preferential *a*-axis orientation of the films on NdGaO_3 , as revealed by TEM, was consistent with the smooth and featureless film surfaces found, in contrast to the *c*-axis growth mode on SrTiO_3 . © 1995 American Institute of Physics.

I. INTRODUCTION

In spite of the rather advanced status of the high- T_c superconducting thin-film technology, intensive research still continues to grow better quality films for future technological applications as well as to understand the growth mechanism(s). The definition of "better quality" is, of course, arbitrary, however, in most cases it is strongly dependent on requirements. Potential superconducting applications, such as superconductor-insulator-superconductor (SIS) structures, Josephson junctions (JJ), and superconducting quantum interference devices (SQUID) all require "real" epitaxial films with abrupt interfaces throughout the layers. For this purpose it is essential to choose insulating materials with a good lattice match with $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) as substrates. Although SrTiO_3 has been one of the widely used substrates for growth of YBCO films, several other substrates are also under serious scrutiny to improve several aspects of YBCO film growth, such as crystallinity, morphology, smoothness, etc. NdGaO_3 is one of the most attractive materials because of its excellent lattice match with YBCO. Moreover, NdGaO_3 , like YBCO, has a perovskite structure and also its thermal-expansion coefficient is close to that of YBCO, which is about $10^{-6}/^\circ\text{C}$.¹

Another important aspect of the superconducting thin-film work that is not yet well understood is the mechanism of nucleation and growth. Studies of the initial growth mechanism, formation, and evolution of the film morphology, microstructure, crystallinity, defects, etc. can provide useful in-

formation to achieve ideal smooth epitaxial films. The morphology, structure, and microstructure of thin films can take on an almost limitless variety of forms while the modes of growth can be categorized into four types: island [Volmer-Weber (VW)], layer by layer [Frank and Van der Merwe (FM)], layer plus island [Stranski-Krastanov (SK)], and columnar.² Both VW and SK growth modes of YBCO films have been reported in the literature.³⁻⁵

In this article we report our findings on the effect of the substrate material on crystallinity, morphology, and growth mode for YBCO films of varying thicknesses (5–260 nm) on $\text{SrTiO}_3(100)$ and $\text{NdGaO}_3(110)$ substrates. The YBCO films were grown by pulsed laser deposition (KrF excimer laser, 248 nm) using a single $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ stoichiometric target. The details of the experimental setup can be found elsewhere.⁶ The films were characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM), x-ray energy-dispersive spectroscopy (XEDS), and x-ray diffraction (XRD).

II. EXPERIMENT

The $\text{SrTiO}_3(100)$ and $\text{NdGaO}_3(110)$ substrates were ultrasonically cleaned first in acetone and then in methanol. Both the YBCO target and the substrate holder were rotated during growth in order to uniformly remove and deposit YBCO, respectively. The laser energy density was $\sim 1.5 \text{ J/cm}^2$ and the repetition rate of the laser was 2 Hz. The deposition rate was defined as the total thickness divided by the deposition time and was around 0.2 nm/s. The substrate temperature and oxygen pressure were 780 °C and 1 mbar,

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respectively, for all the films grown both on SrTiO_3 and NdGaO_3 . The substrate temperature was determined by calibration based on the comparison of the pyrometrically measured temperatures of the substrate surface with readings of a thermocouple located adjacent to the substrate holder. Immediately after deposition the chamber was vented with oxygen and the films were cooled to room temperature. Nine films with increasing thicknesses were deposited under identical conditions with the thicknesses of 5, 10, 20, 40, 60, 80, 100, 130, and 260 nm. Their *ex situ* investigation was done by AFM and SEM to understand the effect of the substrate on morphology, roughness, and microstructure. The 40 nm and thicker films were examined by XRD. The XRD results on both substrates showed that the YBCO films had (00 l) orientation. Rocking curves of the (005) reflection measured by XRD and the full width at the half-maximum was used to assess crystallinity of the YBCO films on SrTiO_3 and NdGaO_3 . In $\Theta-2\Theta$ scans, increase in the widths of the (00 l) reflections with the increasing diffraction angle qualitatively suggested the presence of nonuniform strains perpendicular to the films and along the c axis. The breadth of the (005) reflection for the YBCO/ NdGaO_3 film decreased regularly as the films became thicker. This result was exploited to explain the possible growth mode of the YBCO film. The different stages of the growth were also observed by TEM. The YBCO films were deposited onto prethinned substrates. The thin foil substrates were prepared from the SrTiO_3 and NdGaO_3 single-crystal substrates by cutting disks of 3 mm diameter. The disks were then polished from both sides to a total thickness of 150 μm . The polished disks were dimpled from one side down to 40 μm . After a cleaning process the substrates were used for YBCO deposition and then ion milled from the substrate side at a low voltage of 3.5 kV in a liquid-nitrogen-cooled stage for perforation.

III. RESULTS AND DISCUSSION

The stages of the YBCO film growth for nine different thicknesses were observed by AFM and SEM. Figures 1(a) and 1(b) show images of the nominally 5 nm thick YBCO films on the SrTiO_3 and NdGaO_3 substrates, respectively. The randomly oriented film particles on SrTiO_3 were conical-spherical in shape and their number density was $23 \times 10^{10}/\text{cm}^2$. On the other hand, the film prepared in the same conditions on NdGaO_3 had particles whose sides were perfectly aligned with respect to the substrate. The average particle width varied from 150 to 220 nm and the number density was $7 \times 10^{10}/\text{cm}^2$. The AFM pictures of the thicker YBCO films on SrTiO_3 and NdGaO_3 are shown in Figs. 2 and 3, respectively. From these pictures one can clearly see that the nucleation and growth proceed quite differently on these substrates. In Fig. 2(a), in the 10-nm-thick film, the coalescence of rounded nuclei starts after the sudden burst of various sizes of nuclei [Fig. 1(a)]. In the next stage [Fig. 2(b), the 20-nm-thick film] rounded features of nuclei are replaced by rectangular shapes. These shapes may be characteristic of the nucleation stage. At this thickness also a secondary nucleation starts with continuing deposition, the secondary nuclei grow until they touch their neighbors forming larger islands. They finally get incorporated in large is-

(a)

(b)

FIG. 1. The AFM images of 5-nm-thick YBCO film: (a) on $\text{SrTiO}_3(100)$, and (b) on $\text{NdGaO}_3(110)$.

lands as observed in the 40-nm-thick film shown in Fig. 2(c). As the islands grow they become elongated and join to form a network structure in which the deposited material is separated by long and irregular channels. These channels are bridged at some points due to ongoing nucleation. The thick films are continuous but have several holes and channels and exhibit a liquidlike topography as seen in Fig. 2(f).

Figure 3 shows that the YBCO films grown on NdGaO_3 display quite different features. This difference can be observed throughout the various thicknesses of the films. For example, the 10-nm-thick film, in Fig. 3(a), does not exhibit obvious clustering of islands; the film under the secondary and subsequent nuclei seems to be well connected and smooth. This observation indicates that the initial nucleation and growth is two dimensional in nature. This behavior persists as the films get thicker. The spiral features are first observed in the 60-nm-thick film [Fig. 3(d)]. The layer growth also manifests itself in the thicker films by giving rise

FIG. 2. The AFM images of the YBCO films on SrTiO_3 : (a) 10 nm; (b) 20 nm; (c) 40 nm; (d) 60 nm; (e) 80 nm; and (f) 260 nm.

to more ordered films as one can clearly see in Figs. 3(e) and 3(f). In contrast to the results of Chin, Takahashi, and Morishita⁷ and in spite of the high temperatures employed during deposition,⁸ the TEM observations of the early stages of the growth (5–20 nm) on NdGaO_3 showed that the films appeared to be preferentially oriented with the c axis in the film-substrate plane. Figure 4(a) corresponds to the planar view of the TEM image of a 10-nm-thick film. Most of the interconnected film areas appear to be a -axis oriented (c parallel) and only small regions showing c -axis orientation (c

perpendicular) are distinguishable. The film presents a network structure consisting of small domains. Two different orientations of a -axis-oriented grains with the c axis perpendicular to each other are observed. The electron-diffraction pattern shown in Fig. 4(b) is the superposition of three different sets of reflections: (1) one set corresponding to the $[100]$ zone axis of the YBCO phase where the c axis lies in the two perpendicular directions; (2) one set of maxima corresponding to the $[110]$ zone axis of NdGaO_3 in the two perpendicular directions, and this fact indicates the presence

FIG. 3. The AFM images of the YBCO films on NdGaO_3 : (a) 10 nm; (b) 20 nm; (c) 40 nm; (d) 60 nm; (e) 80 nm; and (f) 260 nm.

(a)

(b)

FIG. 4. (a) Planar-view bright-field image of the 10-nm-thick film on NdGaO₃ along the [110] direction of the substrate. (b) Corresponding electron-diffraction pattern indexed with respect to the film as well as to the substrate (subindexes *f* and *s* refer to the substrate and film, respectively).

of the twins on the substrate; and (3) a minor contribution of the [001] zone axis of the YBCO phase. One possible reason of this preferential *c*-parallel orientation is the good lattice matching between *b'* along the [110] direction of NdGaO₃ and that of *a* (or *b*) of YBCO. NdGaO₃ presents an orthorhombic distorted perovskite structure which contains a pseudocubic sublattice,

$$a' = b' = [(a^2 + b^2)^{1/2}]/2 = 0.3861 \text{ nm}$$

and $c' = c/2 = 0.3865 \text{ nm}$.⁹ The lattice mismatches between *c*/3 of YBCO and *c'*, *b'* of NdGaO₃ are 2.3% and 2.1%, respectively. The lattice mismatches between *b* (or *a*) of YBCO and *c'*, *b'* of NdGaO₃, however, are 0.28% and 0.1%, respectively. Furthermore, because of the layered

structure, the growth of the *c*-axis-oriented films proceeds by a ledge mechanism by addition of chemical species to a surface ledge and the material is most easily added at the *ac* and *bc* faces, that is, the (010) and (100) planes of the unit cell. In this case, the *ab* plane is the low-energy plane and the ledge is normal to the *ab* plane. By contrast, the growth of the *a*-axis-oriented films occurs everywhere along the growth interface because the surface normal is the preferred growth direction. This fact, together with the relatively high deposition rate employed,¹⁰ could be an explanation for the preferential *a*-axis growth. The fact that the YBCO films grow *c* parallel on the NdGaO₃ substrate is consistent with the smooth and featureless surface observed by AFM in the ultrathin films. Although *c*-axis orientation is the thermodynamically preferred growth mode of YBCO, kinetically it is rather inefficient.^{11,12} A large surface diffusion is required to grow a layer which is only one unit cell thick; however, if the film orientation were *c* parallel practically no surface diffusion would be necessary. This fact is reflected also by the surface morphology and roughness which decrease radically because there is no long-range surface diffusion. From a thickness of about 40 nm the orientation changed to *c* axis as revealed by the XRD data. Figure 5(a) shows the planar-view TEM image of a 100-nm-thick film. A high density of twin domain boundaries along the {110} planes is observed. The presence of twin boundaries as well as the electron-diffraction pattern in Fig. 5(b) confirms the film orientation as being normal to the substrate surface. The presence of twins can also be observed in the diffractogram: The amount of splitting corresponds to the ratio of $(b-a)/b$. No *a*-axis-oriented grains are distinguishable.

The films on SrTiO₃ grew *c*-axis oriented from the early stages of growth and showed evenly distributed small particles (10–50 nm) among the thin interconnected film. XEDS analysis, electron diffraction, and high-resolution electron microscopy showed that these particles were [001]-oriented Y₂O₃ and [010]-oriented CuO. A recent report of Locquet *et al.*¹³ shows that the origin of the Cu-rich precipitates is the formation of Cu₂O crystals in spite of the strong oxidizing conditions employed during deposition.

The images in Figs. 6 and 7 correspond to the planar-section TEM micrographs of 10- and 100-nm-thick films on SrTiO₃, respectively. The particles were found to be uniformly distributed over the substrate in the early stages of the film growth as well as over the film surface as inclusions in the thicker deposits. These particles were also observed on NdGaO₃ from a film thickness of about 100 nm. In Fig. 5(a), two Y₂O₃ precipitates can be seen. The corresponding electron-diffraction pattern shows the orientation relationship between the precipitate and the YBCO matrix. Figure 8 corresponds to the high-resolution planar section micrograph of one of these Y₂O₃ particles. Moiré fringes parallel to the [100] direction of the matrix are visible.

The appearance of both Y₂O₃ and CuO particles has been already the subject of numerous studies in films prepared by different procedures and has been found to be an intrinsic behavior of the REBCO (RE: rare-earth elements) films with the best quality in terms of the superconducting properties.^{14–16}

(a)

(b)

FIG. 5. (a) Planar-view transmission electron micrograph (bright-field image) of the 100-nm-thick film on NdGaO₃ in the [110] direction of the substrate. The diagonal lines correspond to the twin boundaries along the {110} planes. The image shows also the presence of two ytria particles. (b) Corresponding electron-diffraction pattern showing the splitting of the diffraction maxima due to the presence of twins. The diffraction maxima have been indexed with respect to the film as well as to the ytria inclusions; y and m refer to Y₂O₃ and the YBCO matrix, respectively.

It is interesting to note the significant difference in the surface roughness of the films on SrTiO₃ and NdGaO₃ when comparing the TEM micrographs on Figs. 5(a) and 7; this difference of the surface roughness was also revealed by the AFM images in Figs. 2(f) and 3(f).

Figures 9 and 10 show the SEM pictures of 10-, 20-, 80-, and 130-nm-thick films on the SrTiO₃ and NdGaO₃ substrates, respectively. The films on SrTiO₃ are rough and have several holes and particles indicating an island growth while the films on NdGaO₃ are smoother and seem to be more ordered, and these features can be associated with a layered two-dimensional growth mechanism. Figure 10(b) shows that needlelike grains appear together with the continuous

FIG. 6. Planar-view transmission electron micrograph (bright-field image) of a 10-nm-thick film on SrTiO₃ along the [100] direction of the substrate. The two types of particles are indicated.

YBCO film on NdGaO₃. They are 800 nm long and 60 nm wide and well oriented along the [110] and [110] directions of the substrate. The density of these needlelike grains decreased as the films became thicker and the grains eventually

FIG. 7. Planar-view transmission electron micrograph (bright-field image) of the 100-nm-thick film on SrTiO₃ along the [100] direction of the substrate. The corresponding electron-diffraction pattern (inset) confirms the *c*-axis orientation of the film.

FIG. 8. High-resolution planar section TEM micrograph tilted slightly around the [001] zone axis, showing an yttria particle in the YBCO matrix. Moiré fringes parallel to the [100] direction are visible.

disappeared. We believe that these needlelike grains on NdGaO_3 are a -axis oriented and their disappearance with the increasing film thickness is consistent with the TEM observations which showed that the thin films were a -axis oriented while the thick films were c -axis oriented.

The XRD patterns of the films (≥ 40 nm) on both substrates showed that the YBCO films were c -axis oriented. The crystallinity of the films was determined from the full width at half-maximum of the rocking curve of the (005) reflection. We first rocked our single-crystal SrTiO_3 and NdGaO_3 substrates and the value of Δw (Δw : full width at half-maximum) for both substrates was 0.04. Therefore, any Δw value measured for the films which is greater than 0.04

is a measure of the film crystallinity. Figure 11 shows Δw values of the YBCO films on SrTiO_3 and NdGaO_3 substrates as a function of the film thickness. The value of Δw for the 40- and 60-nm-thick YBCO films on NdGaO_3 is very small which indicates excellent crystallinity. Δw increases slightly with the film thickness showing that the films maintain their good crystallinity. In contrast, the films of the same thickness grown on the SrTiO_3 substrate exhibit poorer crystallinity; however, their crystallinity improves with the film thickness. It may be that the crystal structure of these films improved after the relief of strains due to the lattice mismatch.

In Θ - 2Θ scans of the YBCO films on SrTiO_3 and NdGaO_3 , the width of the (00 l) reflections increased with increasing diffraction angle, suggesting the presence of non-uniform strains. When the lattice parameters of a unit cell vary, then a nonuniform strain is present, giving rise to line broadening. Similarly, when the thickness of the diffraction domain measured in a direction perpendicular to the reflecting planes is less than 100 nm, the diffraction lines again become broadened.¹⁷ It may be very difficult to separate strain effects from small particle size effects since most of the time both effects coexist; however, it is conceivable to assume that broadening may be due to nonuniform strain for the thick films. Several methods can be employed to analyze diffraction line broadening.¹⁷ In this work we used an integral breadth method in which the integral breadth is defined as

$$\beta = \int J(2\theta) d\theta / J_{\max},$$

where $J(2\theta)$ is the diffracted intensity as a function of scat-

FIG. 9. SEM images of the YBCO films on SrTiO_3 : (a) 10 nm; (b) 20 nm; (c) 80 nm; and (d) 130 nm.

FIG. 10. SEM images of the YBCO films on NdGaO_3 : (a) 10 nm; (b) 20 nm; (c) 80 nm; and (d) 130 nm.

FIG. 11. The full width at half-maximum of the rocking curve of the (005) reflection of the YBCO films on SrTiO_3 and NdGaO_3 . The straight dashed line shows the value of the full width at half-maxima for both single crystals, SrTiO_3 and NdGaO_3 , therefore, it represents the measurement limit of the XRD.

FIG. 12. The breadth of the broadened (005) reflection of the YBCO film on SrTiO_3 and NdGaO_3 vs the film thickness.

tering angle 2θ .¹⁸ In Fig. 12 the change of integral breadth of the (005) reflection was plotted as a function of film thickness. The breadth on NdGaO₃ decreased regularly with increasing film thickness. This regular decrease of the integral breadth of the film on NdGaO₃ may indicate that the film growth was layer by layer. The essential features of the layer-by-layer growth are the following: For a lattice misfit f_0 , the overgrowth is pseudomorphic up to a critical film thickness t_c . This means that the overgrowth film is strained to the lattice parameter and structure of the substrate up to a thickness t_c . Beyond this thickness, misfit dislocations are introduced and the strain decreases approximately as $1/t$, where t is the film thickness.¹⁹ The critical thickness is smaller for a greater misfit, for example, for YBCO/SrTiO₃, the misfit is 0.02; by using $1/t$, we can deduce that the critical thickness would be around 5 nm. The critical thickness is ten times bigger, i.e., 50 nm, for YBCO/NdGaO₃ since the misfit is ten times smaller. This fact is well manifested in the AFM images: Spirals due to screw dislocations on SrTiO₃ were seen even in the 10-nm-thick film [see Fig 2(b)] at the stage of island coalescence. On the other hand, spirals started appearing on NdGaO₃ at a greater thickness, 60 nm as can be seen in Fig. 3(d). These observations suggest that there is a substrate-dependent critical thickness for introduction of screw dislocations. The other possible explanation for the emergence of the screw dislocations can be due to island coalescence in which dislocations are generated because of rotational, translational, or vertical misalignment of the separate islands.²⁰

Based on both our experimental results and the above arguments we believe that the YBCO films with the initial thickness of 5 nm on SrTiO₃ grew in three-dimensional form and as the films became thicker this mode persisted. However, we do not rule out a possibility of a SK mode in which a switch in the growth mode may have taken place from two dimensional to three-dimensional mode. In this case, only the first couple of unit cells could grow layer by layer. On the other hand, the YBCO films on the NdGaO₃ substrate followed a layer-by-layer growth at least up to a thickness of 60 nm. Although the screw dislocations were visible in the 60-nm-thick film, the thicker films were still much more ordered, smoother, and furthermore had the better crystalline quality than those of on SrTiO₃.

IV. CONCLUSIONS

Superconducting YBCO films were deposited by the pulsed laser deposition technique on both SrTiO₃ and NdGaO₃ to see the impact of the substrate material on the structural properties of the films. The crystalline quality of the films on the NdGaO₃ substrate whose lattice mismatch with the YBCO material is very small was much better than that of on SrTiO₃. Comparison of SEM and AFM images showed that the films on NdGaO₃ were rather smooth and

featureless while the films on SrTiO₃ had several holes and/or channels. Appearance of spirals in very thin films together with this morphology on the SrTiO₃ substrate strongly suggested that the growth mode was a three-dimensional island type. On the other hand, the films on NdGaO₃ chose a different growth mechanism, namely, a two-dimensional nucleation and growth type. The film particles at the very early stages of the growth were well aligned with respect to the substrate, coalesced regularly, and covered the entire substrate surface more efficiently. Although the dislocations were introduced in the 60-nm-thick film the crystalline quality, morphology, and smoothness of the thicker films were still much better than the films of the same thickness on SrTiO₃.

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