Embedded polytypes in $Bi_2Sr_{2-x}La_xCuO_6$ thin films grown by laser ablation

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We investigate the presence of secondary phases in La-doped Bi-2201 thin films grown by laser ablation. The cation ratios in the target material, the oxygen pressure, and the substrate temperature during the deposition are the main parameters determining the presence of diluted intergrowth and/or polytype aggregates. A statistical model of random intergrowth is used to analyze the x-ray diffraction (XRD) anomalies caused by hidden defects and to characterize the latter. A detailed structural XRD refinement on oriented aggregates allows us to identify the guest phase as a Bi deficient phase, Bi-1201. The occurrence of this particular embedded polytype is accompanied by a global Bi deficiency introduced in the films by the growing process and/or by the annealing treatment. The presence of La favors the Bi-1201 formation mostly as La-rich *c*-axis oriented aggregates. Bi excess in the target material improves considerably the crystallographic structure of Bi-2201, avoids intergrowth formation, but does not prevent the phase separation of Bi-1201 in La-doped thin films. We also investigate the influence of the deposition parameters on the type of intergrowth as well as their variation with La doping. This work introduces a specific methodology for optimizing the growth of thin films grown by laser ablation, which applies to layered oxides that admit polytypes with close formation enthalpies in their phase diagram.

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I. INTRODUCTION

Pulsed laser deposition (PLD) is a widely used technique to grow high critical temperature superconducting cuprates for applications as well as for fundamental research. In both cases a precise knowledge of the film structure is necessary. One of the main advantages of using PLD is the possibility to grow complex materials from their vapor phase using a single stoichiometric target. Indeed, laser ablation transfers the exact stoichiometry of the target to the formed plasma. However, as for other vapor deposition techniques, the correct stoichiometry of the vapor phase alone does not guarantee the quality of the condensed phase. Furthermore, PLD is an out-of-equilibrium process, which can favor the irreversible formation of otherwise metastable secondary phases.[1,2] In the particular context of our work, i.e., the growth of $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ $(n=1,2,3)$ (BSCCO) systems, a large number of different polytypes with close formation enthalpy can occur. Earlier work on BSCCO grown by sputtering[3–5] shows the presence of intergrowth (intercalated and oriented similar structures) embedded in the majority-phase matrix. Such intergrowth can be achieved if, e.g., $CaCu_2O_y$ layers of Bi-2212 or $Ca_2Cu_3O_y$ layers of Bi-2223 structures are substituted by CuO_y layers of the Bi-2201 structure.[6] Intergrowth of different phases in thin films of Bi-based compounds is a general issue of out-of-equilibrium techniques such as PLD and sputtering. Molecular beam epitaxy (MBE) is more close to thermodynamic equilibrium thus favoring single-phase formation[7,8] but, even there, a kind of intergrowth formation was also reported.[9] However, regardless of the deposition technique, it is possible to produce single-phase films by adjusting properly the deposition parameters.

In the present work, we focus on the growth of the lowest *n* La-doped compound: $Bi_2Sr_{2-x}La_xCuO_6$ (Bi-2201). The La^{3+} substitution for Sr^{2+} reduces the hole doping and reduces the out-of-plane disorder, thus increasing the critical temperature (T_c).[10–12] La-doped Bi-2201 thin films grown by PLD[4] or other deposition techniques[13,14] have been already studied by other groups. However, more studies are needed on the structural properties and our work is inserted in this context. We surprisingly encountered a type of intergrowth (IG) in Bi-2201 films, despite the absence of Ca. This polytype has been identified earlier as $BiSr_2CuO_5$ (Bi-1201)[15] in the form of an oriented aggregate within the Bi-2201 host lattice. This Bi-deficient phase was already reported,[16,17] as a stacking fault in the Bi-2212 structure. While the information about Bi-1201 compound in literature is sparse, the crystallographic structures of Hg- and Tl-based isostructural compounds are well known.[18,19] We report on the investigation of the occurrence of polytypes in Bi-2201 films grown by laser ablation as a function of the La substitution, the Bi content in the target, the deposition parameters, and the annealing treatment. The presence of La favors the Bi-1201 formation mostly as *c*-axis oriented aggregates hereafter phase separation (PS). For PS we intend "visible" defects that appear as extra peaks in the x-ray diffraction spectrum. IG, on the contrary, are "hidden" defects that can be revealed only by a more subtle diffraction analysis as in Ref. 21.

In contrast, the undoped compound can be grown as a single phase or containing Bi-1201 random intergrowth but never showing PS. The presence of Bi-1201 inclusions implies a global Bi deficiency in the films. Bi excess in the target is observed to improve the Bi-2201 crystal structure, to avoid IG formation, but it does not prevent PS to occur. Concerning the effect of the deposition parameters, the intergrowth is favored when the substrate temperature is reduced and/or the sample-to-target distance and oxygen pressure are increased. Post-annealing treatments on La-doped films sup-

press intergrowth in favor of phase separation and have no noticeable effect on undoped samples. From our results we propose an interpretation based on structural rearrangement driven by La in-plane mobility, charge balance, and local chemical stability.

In Sec. II we describe the sample fabrication and the characterization techniques. Section III is devoted to results and discussion. Finally, we summarize the main points and draw conclusions in Sec. IV.

II. EXPERIMENT

Stoichiometric $Bi_2Sr_{2-x}La_xCuO_6$ ($x=0,0.2,0.3$) and nonstoichiometric $Bi_{2.2}Sr_{2-x}La_xCuO_6$ ($x=0.05,0.1,0.2,0.3$) targets are made by firing appropriate amounts of $SrCO_3$, La_2O_3, CuO, and Bi_2O_3 in an Al_2O_3 crucible. The reaction mixtures were sintered in pellet form at 800–810 °C with frequent regrinding. The target composition was analyzed by powder diffraction, showing only the Bi-2201 phase. Thin films are grown by PLD on oriented (100) $SrTiO_3$ substrates with a pulsed yttrium aluminum garnet laser at 266 nm, 100 mJ pulses, and 3 Hz repetition rate. The films are grown under different conditions; the oxygen pressures, the target-substrate distance, and the temperature of the substrate are changed. The films are annealed *in situ* at 700 °C for 30 min in 1 atm of O_2. Some of them are also *ex situ* annealed at 700 °C in 1 atm of oxygen for 1 h. All the films were characterized by x-ray diffraction (XRD) and resistivity measurements. XRD was carried out in a four-axis diffractometer using Cu Kα radiation (1.5406 Å). The crystal structures of the film are studied in depth by XRD using conventional θ-2θ scans and rocking curves (RCs). Resistance measurements are carried out using a standard dc four-probe system.

III. RESULTS AND DISCUSSION

Analysis of the XRD data in our films shows different kinds of defects. The most visible ones appear as extra peaks superimposed on the expected 2201 peaks, denoting a separated secondary phase. Additionally, we encounter more subtle defects, harder to detect: random intergrowth. Indeed, at a first glance, the θ-2θ analysis shows only the presence of the (00l) oriented Bi-2201 phase. However, a more accurate analysis reveals nonmonotonous deviations of the Bragg peak positions (Fig. 1) and anomalous intensity ratios in the θ-2θ spectrum (Fig. 2). Figure 1 shows the *c*-axis parameter, determined from each peak, as a function $\cos^2\theta/\sin\theta$ in order to correct for potential displacement errors. The film containing intergrowth shows large oscillations instead of the expected linear dependence shown by the single crystal substrate or the intergrowth-free sample. Figure 2 shows the XRD patterns of two samples, with and without intergrowth, in comparison with the calculated pattern of pure Bi-2201 based on the fractional atomic coordinates extracted from Ref. 20. It is clear from this picture that the intensity ratios for the sample with intergrowth are radically different from the standard pure Bi-2201 diffraction pattern. Furthermore, the RC shows anomalous features as it is shown in Fig. 3. There are two contributions to the in-plane correlation function giving rise to the two components of the RC: one broader Lorentzian function associated with the standard in-plane correlation within finite-size coherent domains; and a "δ peak" from the long-range contribution of equidisplaced domains.[21]

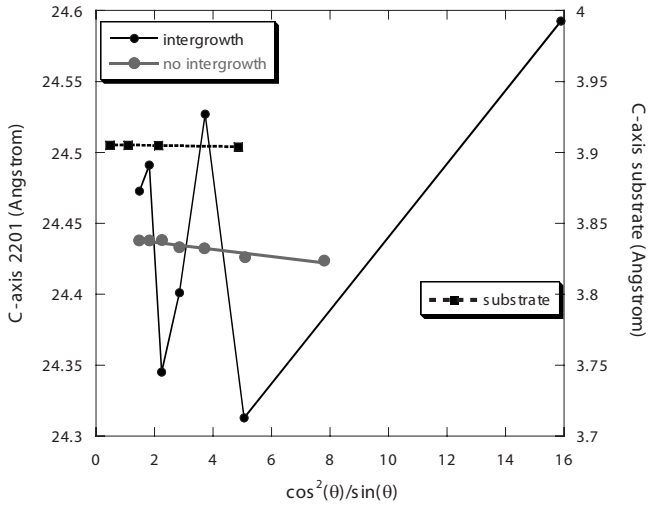

FIG. 1. Comparison between the *c*-axis deviation of a film with and without intergrowth and of an $SrTiO_3$ substrate. The *c*-axis oscillation is strongly pronounced for samples with intergrowth.

Such features combined with oscillations in the full width at half maximum (FWHM) of the peaks are unambiguous signatures of random intercalation. To analyze the intergrowth presence, we use a statistical model, introduced in earlier publications.[21,22] We extract the fraction of the guest phase, α, and the difference in *c*-axis lattice constants, Δc, between the host and the guest unit cells (UCs). We observe two different types of intergrowth (Fig. 4): (1) the *c* axis of the guest phase is ~3.7 Å larger than the one of the host lattice (24.4 Å), hereafter "+3"; and (2) the *c* axis of the guest phase is ~3.1 Å shorter than the host lattice, hereafter "−3."

The first kind of intergrowth is observed only in 20% La-doped samples. The 30% La-doped samples never show intergrowth. We observe, however, phase separation and we identify the guest phase as a Bi-deficient phase: $Bi(Sr,La)_2CuO_5$, Bi-1201.[15,16] This helps us to understand the two kinds of intergrowth observed in our films: both are due to the intercalation of a Bi-1201 phase. Namely, the *c* axis of Bi-1201 is 9.45 Å; the presence of three consecutive UCs of this phase in the +3 IG could explain the +3.7 Å difference in *c* axis (Fig. 5). Similarly, the −3 IG, mainly observed in undoped samples, is formed by half UC of the Bi-2201 and one UC of Bi-1201 giving a total *c* axis of 21.2 Å. The occurrence of intergrowth depends on the La doping and on the growing conditions as it will be explained in the next two subsections.

A. Undoped samples

The presence of Bi-1201 in the films denotes a global Bi deficiency. Since PLD is assumed to transfer the exact target stoichiometry to the vapor phase,[23] the Bi loss must occur

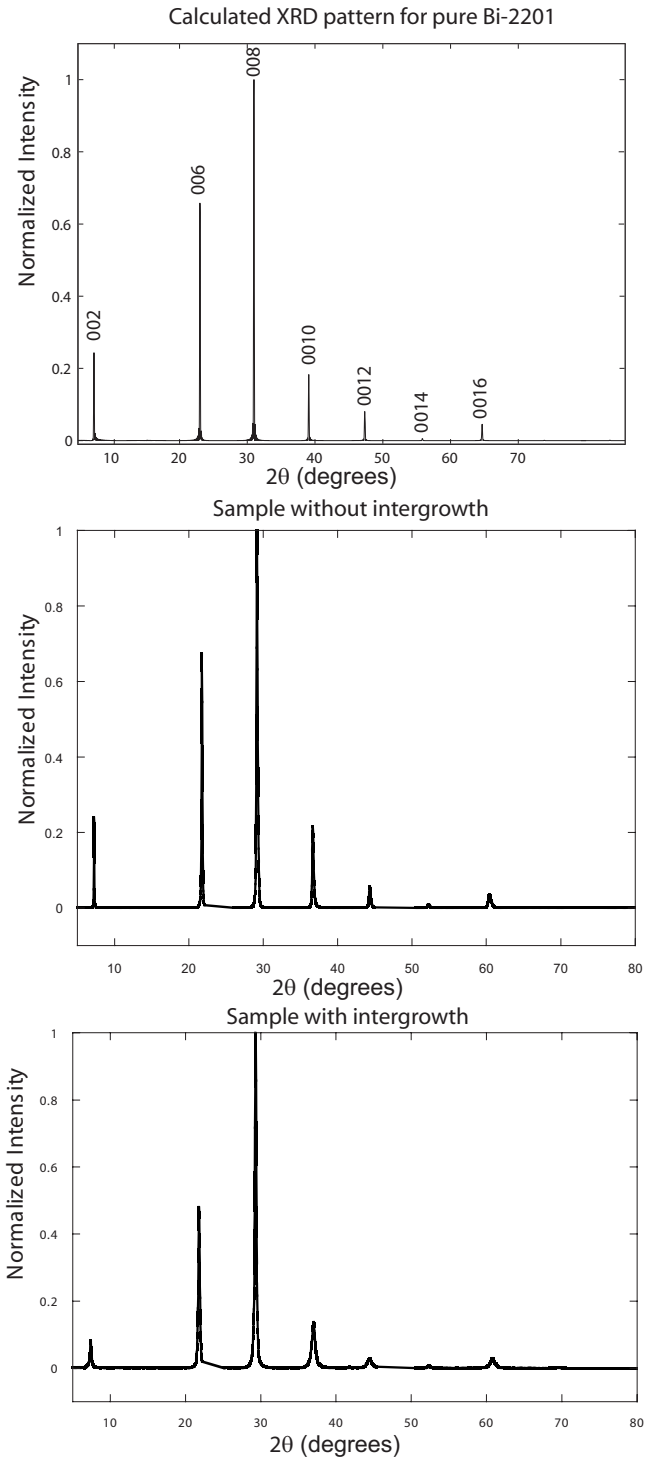

FIG. 2. Diffraction patterns for a film with and without intergrowth compared with a calculated XRD pattern for undoped Bi-2201.

during the condensation process. Bi volatility is known to affect the crystal composition when growing BSCCO from the melt or from the vapor phase in some techniques close to equilibrium, such as MBE[25] or sputtering.[2,24] Using PLD, an out-of-equilibrium technique, the sensitivity to Bi loss is enhanced. As discussed in an earlier publication,[21] PLD can be viewed as a repeated quenching process. Indeed, after each

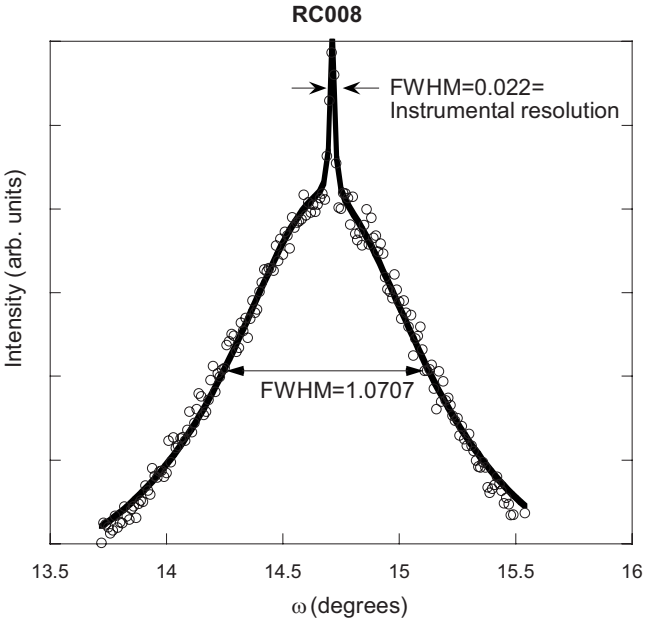

FIG. 3. Typical RC of a film with intergrowth. Two contributions are evidenced: the broader Lorentzian corresponds to the exponential correlation within finite size coherent domains while the narrow peak comes from the space independent correlations.

laser pulse of approximately 20 ns, the adiabatic expansion of the high-energy plasma supplies the growing film on the heated substrate with an impressive amount of kinetic energy, in addition to the latent heat liberated by the condensation process itself, contributing to the surface mobility of the present species, and producing a short-time annealing of the underlying atomic layers. This external heating enhances the effective substrate temperature for about 1 μs, after which the film stays at the nominal substrate temperature until the next laser pulse, i.e., 300 ms later. In the random intergrowth model proposed in Ref. 21, it is assumed that local composition fluctuations, namely, Bi deficiency, produce a Bi-poor oriented polytype that can be trapped in the Bi-2201 matrix as a stacking fault. The unreacted Bi is easily removed by the continuous O_2 flow in the chamber during the deposition. This local defect, in turn, favors the formation of additional stacking faults that locally reduce the elastic energy. The final result is a random distribution of intergrowth along the growth direction, responsible for the aforementioned XRD anomalies, with a tendency to form in-plane flakelike clusters denoted by the Lorentzian contribution to the RC. The above simple scenario seems to apply to undoped Bi-2201 films, where the intergrowth structure can be reduced or even suppressed using specific growing conditions (as indicated in Table I): small target-to-substrate distance, low O_2 pressure, and high substrate temperature. In other words, reducing the thermalization of the plasma in the plume by bringing the substrate nearer to the target or by reducing the O_2 pressure in the chamber contributes to the increase of the effective growth temperature, which means growing the film close to equilibrium conditions. However, post-annealing treatments of undoped films containing intergrowth do not noticeably alter either their as-grown structure or their transport proper-

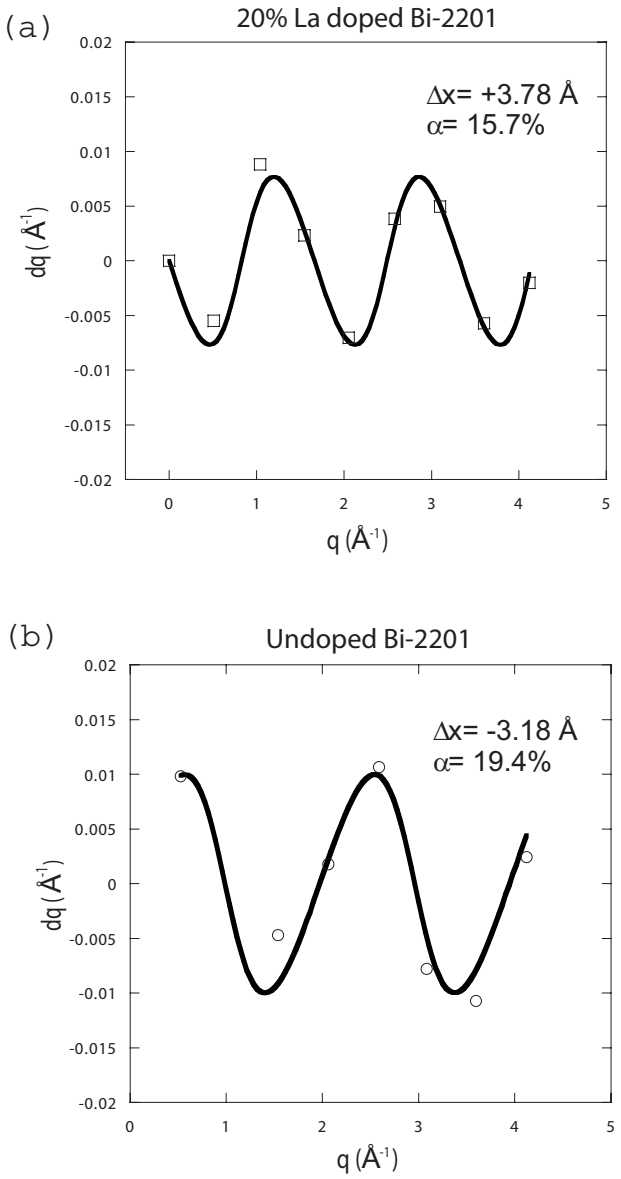

FIG. 4. Momentum deviation for (a) a 20% La-doped Bi-2201 film and for (b) an undoped one fitted with the statistical model of Ref. 21.

ties. The annealing temperature used in our experiments is presumably too low to allow the system to overcome the energy barriers associated with the local diffusion of Bi atoms in the matrix.

B. La-doped samples

In contrast to what is observed for undoped samples, La-doped samples are highly sensitive to both growing conditions and post-annealing treatments. In general, under the same growing conditions, La-doped films contain more polytype defects and have a marked tendency to undergo phase separation in the form of oriented La-rich Bi-1201 aggregates, which seems to be the ultimate equilibrium configuration after annealing. In addition, no pure 2201 phase can be achieved in the presence of La (Table I). Thus, the simple

TABLE I. Growth conditions.

	$Bi_2Sr_2CuO_6$	
	Intergrowth	No intergrowth, single phase
T (°C)	600	⩾600
O_2 pressure (mbar)	0.4	0.4
Distance (cm)	5	4
	$Bi_2Sr_{1.8}La_{0.2}CuO_6$	
	Intergrowth	No intergrowth but phase separation
T (°C)	⩽600	>610
O_2 pressure (mbar)	0.4	0.1
Distance (cm)	5, 4	4
	$Bi_2Sr_{1.7}La_{0.3}CuO_6$	
	Intergrowth	Phase separation
T (°C)		>610
O_2 pressure (mbar)		0.1, 0.4
Distance (cm)		4,5

model invoked for undoped samples has to be complemented in order to explain the trends observed in La-doped films. One ingredient to be added to the scenario is the interplay between charge balance and elastic energy. Indeed, La^{3+} ions are essential to accomplish the charge balance in Bi-1201,[26] even favoring the formation of this phase, while they are largely unbalanced in the 2201 structure. Furthermore, local chemical and elastic relaxation can be improved by La diffusion from 2201 cells to neighboring 1201 intergrowth. The Bi-deficient polytype will then be more stable with La. Apart from the local composition fluctuations, the nucleation of the Bi-deficient structure is now dominated by the charge balance mechanism related to the La presence. That explains why we observe 1201 PS only in La-doped samples. During the annealing process, further rearrangements yield a phase-separated final configuration, where the 1201 aggregates capture most of the La^{3+} ions. Such rearrangements need also some Bi mobility that seems to be triggered by the larger La diffusivity at the considered annealing temperatures. A last feature to be considered here is the difference between the type of intergrowth appearing in undoped and La-doped films. While the main defects in undoped films (−3 IG) consist of diluted single Bi-1201 unit cells, the so-called +3

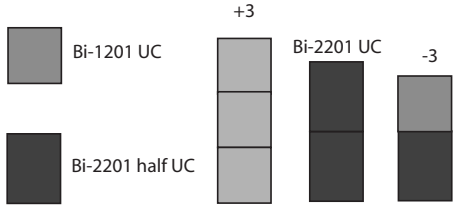

FIG. 5. Schematic representation of the two kinds of intergrowth discussed in the text.

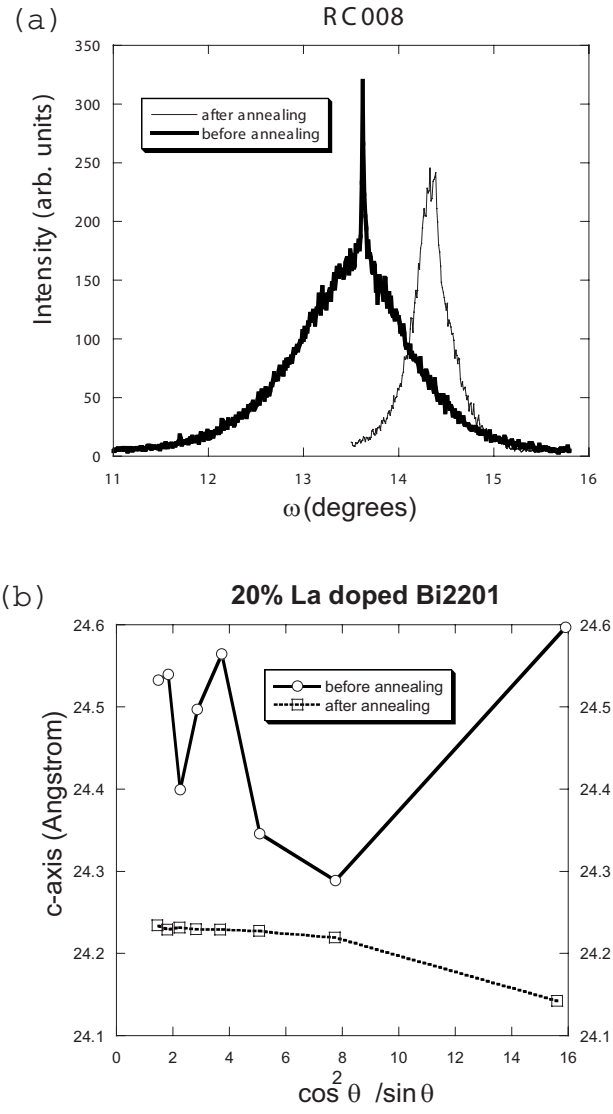

FIG. 6. Comparison of the c-axis behavior and the RC for a film before and after annealing.

intergrowth, formed by local piling up of three consecutive Bi-1201 unit cells, in the La-doped films explained in Sec. III A, imply a higher density of defect nucleation during the growth as well as a good mobility of La^{3+} and Bi^{3+} ions within the host matrix. Thus, the mechanisms invoked for the evolution during the post-annealing are already active during the film formation and explain the PS observed in La heavily doped as-grown films. The absence of piling up of just two 1201-UC is due to the higher cost in elastic energy of the resulting step in the 2201 matrix: while the displacement step between a complete unit cell of 2201 and a sequence (half-2212)-(1201) or $3\times(1201)$ are about −3 or +3 Å, respectively, the difference with a sequence of $2\times(1201)$ would be of about −6 Å, a mismatch that makes this event statistically improbable.

We can observe −3 intergrowth for 20% La-doped samples when no *in situ* annealing treatment is performed. An *ex situ* annealing of these samples is suppressing the −3 intergrowth and causing phase separation. The same result is obtained if we anneal *ex situ*, +3 IG samples. Indeed, as it is

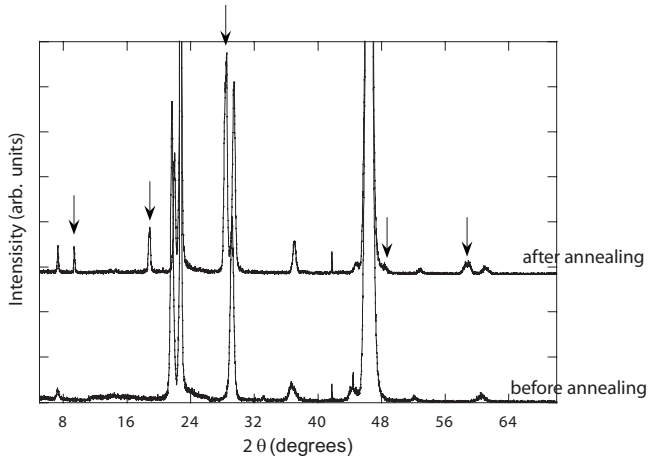

FIG. 7. Comparison of the θ-2θ spectrum of the film before and after annealing for a film with IG; the peaks of the 1201 phase appear after annealing (indicated by arrows).

shown in Figs. 6(a) and 6(b), the anomalies in the c-axis oscillations and in the RC disappear. However, the Bi-deficient phase, Bi-1201, appears as an oriented aggregate (cf. Fig. 7). Concomitantly, we notice that the c axis after annealing is 1% smaller than the average value before the annealing (cf. Fig. 6). This is presumably due to Bi losses in the 2201 phase during the annealing. As we shall see in the next subsection, this reduction of the c axis is not observed in Bi-compensated samples. These results are confirming the picture described previously: the annealing is favoring the aggregation of Bi-1201 first as +3 IG and second, for longer annealing time, as PS.

C. Bi-compensated samples

As previously discussed, the presence of intergrowth and phase separation implies a global Bi deficiency in the films. To compensate the Bi deficiency in films, we grow the films starting from a nonstoichiometric target. We use different La-doped targets ($x=0.05,0.1,0.2,0.3$) with Bi excess of 0.2 ($Bi_{2.2}Sr_{2-x}La_xCuO_6$). However, even reducing the La content to $x=0.05$, x-ray diffraction still reveals phase separation of Bi-1201, underlying the fact that the Bi excess in the target is favoring rather than preventing the phase separation. Nevertheless, the excess Bi contributes to improve the Bi-2201 phase: the in-plane crystallinity and along the c axis are increased. In Fig. 8, the RC comparison shows that the FWHM is reduced by 53% in the Bi-compensated samples, implying an improved in-plane crystallinity. At the same time, θ-2θ data show higher absolute intensities and the $K\alpha$ doublet is well resolved denoting an improved structural coherence along the c axis. Bi excess in the target reduces the disorder in Bi-2201 phase: comparatively, films made from a stoichiometric target contain more Bi deficiencies and consequently more defects than the ones made from Bi-compensated targets. Furthermore, a fraction of La^{3+} ions are released toward the 1201 phase and, at the same time, the structure of 2201 becomes more complete, i.e., better RC. As a consequence, we have a better 2201 structure but a higher amount of 1201

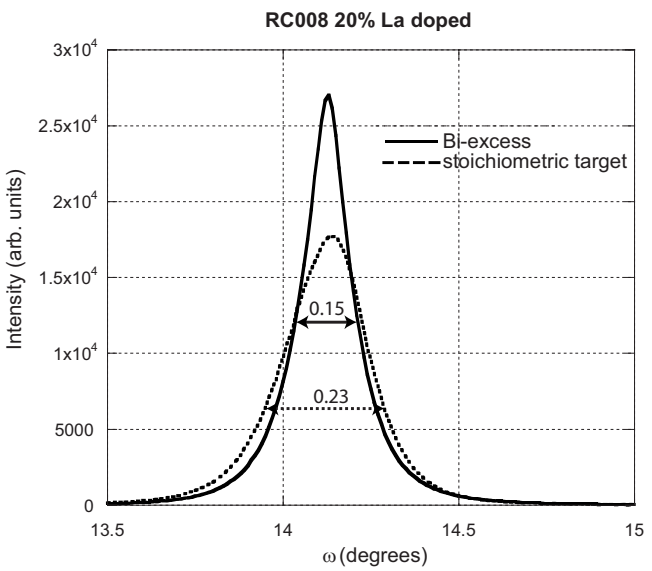

FIG. 8. Comparison of the RCs around the (008) for a film produced by a stoichiometric and a Bi excess target.

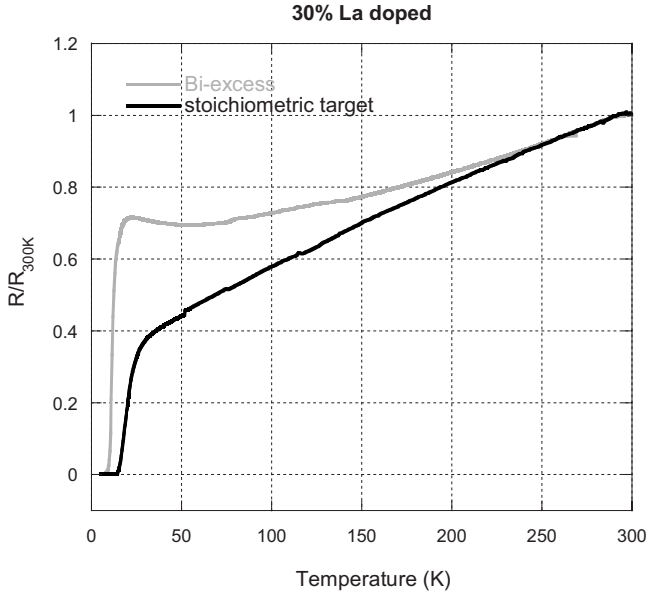

FIG. 9. Comparison of the normalized resistance for stoichiometric and Bi-compensated 30% La-doped samples.

aggregates. Moreover, the resistivity measurements show that for the same La doping, the T_c is lower for a Bi-compensated sample (cf. Fig. 9). This can be accounted for by the higher amount of defects and by the fact that the effective La doping is lower than in a stoichiometric sample: while the presence of defects is detrimental from the crystal-structure point of view, it is favoring the inclusion of La^{3+} in the 2201 phase more than for Bi-compensated samples with less defects.

IV. CONCLUSIONS

We show in this work that the presence of embedded polytypes in La-doped Bi-2201 thin films grown by PLD is a critical issue and should be investigated in order to grow high quality samples. We discover the presence of a particular embedded polytype: a Bi-deficient phase, Bi-1201, which can appear as random intergrowth or as an oriented separated aggregate (phase separation). The growth process with PLD is performed out of thermodynamic equilibrium: local composition fluctuations, namely, Bi deficiency, are quenched and trapped in the Bi-2201 matrix as stacking faults (intergrowth). Their presence must be carefully investigated by detailed structural XRD studies, since a preliminary inspection of the θ-2θ diffraction patterns reveals only one set of lines as in single-phase samples. Only a statistical analysis of the Bragg peak deviations provides us with the correct characterization of such diluted epitaxial polytypes. Their formation as a *c*-axis oriented separated phase is only observed for La-doped samples, emphasizing the fundamental role played by La in the nucleation of the 1201 phase. Indeed, local strain and charge balance are the keys to explain the mobility of La^{3+} ions which cluster in the 1201 phase. In any case, this aggregation seems to be the most stable configuration obtained systematically after further annealing.

The quantitative analysis of the XRD data reveals two kinds of intergrowth. Single-phase Bi-2201 samples are obtained only without La. A departure from the ideal deposition conditions in pure samples only produces random intergrowth which is not noticeably affected by further annealing treatments. The presence of such polytypes implies a global Bi deficiency introduced in the films by the growth process and/or by the annealing treatment. The Bi-compensated samples show a better crystallographic order of the Bi-2201 but they always undergo phase separation of Bi-1201. The reduced amount of defects in the Bi-compensated 2201 structure drives the La^{3+} ions toward the 1201 unit cells, making the 2201 structure more stable but reduces the effective doping and consequently the T_c. The specific methodology introduced in this work can easily be extended to other types of systems, namely, high-T_c layered cuprates that admit polytypes with close formation enthalpies. Further studies are needed in order to be able to grow pure single-phase La-doped samples for spectroscopy analysis such as angle-resolved photoelectron spectroscopy.

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