YBa$_2$Cu$_3$O$_7$ films grown onto SrTiO$_3$ and YSZ substrates by chemical solution deposition of trifluoroacetates

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Abstract

We report the preparation and characterization of YBa$_2$Cu$_3$O$_7$ (YBCO) films grown onto SrTiO$_3$ and YSZ substrates by the trifluoroacetates chemical solution deposition method and following sintering with oxygen atmosphere at 860 °C. The X-ray diffraction (XRD) reveals (00ℓ) – oriented crystallites indicating epitaxial growth of the films in the c-direction. Despite granular morphology and the presence of Y$_2$BaCuO$_5$ and CuO as minor secondary phases, the technique shows the successful formation of the superconducting YBCO and preventing the formation of the unwanted BaCO$_3$ phase. Rocking curve measurements of the (005) reflection for the YBCO/SrTiO$_3$ was fitted with one Gaussian function with full width at the half maximum (FWHM) of 0.44° confirming that it consists of YBCO crystallites with different texture. For the sample grown on YSZ, the rocking curve was fitted with two Gaussian functions, one corresponding to the YBCO layer (FWHM = 0.4°) and another to the substrate (FWHM = 0.3°). The magnetic measurements taken in zero field cooling and field cooling modes confirm the formation of the superconducting YBCO with critical temperatures ($T_c$) 91.8 and 85.7 K for the samples grown onto YSZ and SrTiO$_3$, respectively. The critical current density ($J_C$) curves indirectly calculated by using the Bean’s model from the $M(T)$ loops were $J_C \sim 10^9$ A/cm$^2$ for the sample deposited onto YSZ and $J_C \sim 10^7$ A/cm$^2$ for the YBCO deposited onto SrTiO$_3$. Overall, the difference in $T_c$ and $J_C$ values between both samples could be related to their difference in oxygen content, porosity, hole concentration per Cu ion and the presence of secondary phases.

Keywords YBCO · Chemical solution deposition · Superconductors · Trifluoroacetates

1 Introduction

The trifluoroacetate metal–organic chemical solution deposition (TFA-MO-CSD) method is a low cost route for producing YBCO superconducting films because it does not require vacuum conditions or expensive equipment [1–13]. This technique was introduced by Gupta et al. in 1988 in order to prevent the formation of BaCO$_3$ [14–26] during fabrication of YBCO films by following the chemical solution deposition (CSD) method [27]. In fact, in other different CSD routes, BaCO$_3$ agglomerates at the grain boundaries affecting the critical current density ($J_C$) of the resulting YBCO films up to a factor of around 10^2–10^3 [28, 29]. The TFA-MO-CSD technique includes the preparation of the precursor solution by mixing Y and Ba trifluoro-acetates with Cu acetate (molar ratio 1:2:3) in methanol [30] or water [31]. The organic components are eliminated by pyrolysis at around 400 °C, yielding in Y$_2$O$_3$, BaF$_2$ and CuO. These intermediate compounds are then transformed into YBCO tetragonal (non superconductor) by annealing at around 800 °C. The YBCO orthorhombic (superconductor) phase is then obtained by oxygenating the latest compound at high temperatures. The drawback is that, by this technique, BaF$_2$ forms during the decomposition of the organic compounds...
(pyrolysis) and the YBCO films are then obtained via hydrolysis of BaF2 [32].

About substrates, SrTiO3 and YSZ single crystals are commonly used to grow YBCO films since their lattice constant (0.3905 and 0.512 nm, respectively) are quite similar to that of YBCO orthorhombic. The mismatches with the YBCO lattice constant are: 1.35% for the SrTiO3 and 32.88% for the YSZ. However, these mismatches influence also in the morphology and magnetic properties of the resulting films [33]. We have previously reported the epitaxial growth of the YBCO film on YSZ substrate despite the highest mismatch in lattice constant with respect to YBCO [34].

In this work we grow YBCO onto SrTiO3 and YSZ substrates by following the TFA-MO-CSD method and sintering at 860 °C. We obtained uniaxial granular films showing superconducting behavior below ~90 K. The critical current density \( J_c \) values of around 10^5 A/cm² was indirectly derived from the magnetic measurements using the Bean method. Overall, the difference in critical temperature \( T_c \) and \( J_c \) values between both samples could be related to their difference in oxygen content, porosity, hole concentration per Cu ion and presence of secondary phases.

2 Experimental

The fabrication of the YBCO films was performed by the TFA-MO-CSD method and annealing. In this work, the following salts (99% purity, Alfa Aesar Inc.) were reacted: Y trifluoroacetate \([Y(OOCCF_3)_3]_2 \), 3–0 H₂O; Cu trifluoroacetate (II) \([Cu(OOCCF_3)_2 \), 0–7 H₂O] and Ba trifluoroacetate \([Ba(OOCCF_3)_2 \), 1–9 H₂O]. Stoichiometric amounts of the Y:Ba:Cu trifluoroacetates in 1:2:3 ratio were mixed and completely dissolved in an ethanol \((C_2 H_5OH)\) and bi-distilled water solution of 1:1 ratio using a magnetic stirrer at room temperature. Subsequently, the obtained solution containing the Y³⁺, Cu²⁺ and Ba²⁺ ions was decanted for 12 h in a matrix of oxalic acid and ethanol alcohol to obtain oxo-fluorates. The system was stirrer at 250 rpm thereby to ensure a correct homogeneity. This precursor solution was directly dripped onto YSZ (100) and SrTiO3 (100) substrates using a Fisher pipette. The samples were immediately dried at 40 °C, in order to remove volatile elements. This process was repeated 7 times.

The crystallization and epitaxial growth of the YBCO layers were achieved by heat treatments and sintering at 860 °C in a tubular furnace (LENTON LTF-PTF Model 16/610) in oxygen atmosphere for 12 h. Then, the furnace was set to 600 °C for 4 h in oxygen atmosphere. The furnace was programmed to anneal and to quench the sample at the same ratio (1 °C/min).

The crystallization of the sample was studied by X-ray diffraction (XRD). The data were collected from 8° to 80° (0.02° step) using a universal diffractometer Bruker D8 with Lynx Eye detector and Cu Kα radiation (\( \lambda = 1.54,184 \) Å). The morphology of the samples were inspected by scanning electron microscopy (SEM), cross sectional – transmission electron microscopy (CS-TEM) and high resolution-transmission electron microscopy (HR-TEM). For the TEM measurements the samples were coated with a Pt protective layer, then milled and thinned with a focused ion beam (FIB) in a dual-beam Quanta 3D apparatus (Philips). The interfaces were inspected in a Tecnai 20 (Philips) microscope, with a 200 keV beam generated by a tungsten source.

The superconducting state of the sample was studied from its susceptibility vs. temperature response which was obtained in a DC magnetic property measurement system (DC-MPMS—SQUID) from Quantum Design Inc. The data were collected in zero field cooling (ZFC) and field cooling (FC) modes in the temperature range 8—110 K. The critical current density \( J_c \) for the samples were indirectly obtained from their respective hysteresis loops taken at 10 and 20 K by following the Bean’s equation [35]:

\[
J_c = \frac{20\Delta M}{\pi w^2 (l - \frac{w}{3})}
\]

where \( \Delta M \) is the difference in magnetization values (+M and −M) at a particular magnetic field; and \( t, w, \) and \( l \) are the thickness, width and length of the samples, respectively.

3 Results and discussions

Figure 1 shows the X-ray diffraction, at logarithmical scale, of the annealed YBCO films. The presence of the peaks (003), (005) and (006) reveal epitaxial growth in the c-direction. The presence of the (103) reflection accounts for a minor part of YBCO that has not followed an epitaxial growth that possibly belonging to the most superficial layers of the sample. For the sample grown on YSZ, the two strong reflections at 34.96° and 73.53° correspond to the substrate. The first reflection screens the main (005) peak belonging to the YBCO film. The \( Y_2 BaCuO_5 \) (Y211) and CuO are also detected as secondary phases. The Y211 phase (PDF card 78–1719 [36]) is detected by the reflection around 29.99° corresponding to (311) peak. The CuO phase (PDF card 89–2899 [36]) might be present due to inhomogeneities in the starting precursor solution. This phase is observed in relative low intensity with reflections around 32.65° (110), 38.68° (111) and 65.57° (022).

For the case of the YBCO film grown onto SrTiO3 substrate, the XRD (Fig. 1(b)) shows the presence of the peaks (002), (003), (004), (005), (006) and (007) revealing epitaxial growth in the c-direction or the texture axis. The three
strong reflections at 23.06°, 46.95° and 72.83° correspond to the SrTiO₃ substrate. The Y211 and CuO are also detected as secondary phases. A relative high number of CuO reflections are observed due to the low annealing temperature.

In general, the XRD patterns show a uniaxial texture along c-axis for the YBCO films growth in both substrates with no formation of BaCO₃ as secondary phase, proving the versatility of the TFA-CSD technique. The obtained phases, main reflections and crystalline parameters are listed in Table 1. For the case of the YBCO/YSZ sample, the phase has a relative high crystallite size (27.74 nm) compared to the secondary phases CuO and Y211 (23.84 and 22.28 nm respectively), while for the YBCO film grown onto SrTiO₃ substrate the highest crystallite size detected belongs to the CuO secondary phase (49.62 nm) and the minor size (35.23 nm) belongs to the YBCO superconductor phase. These differences might be related to the granularity of the films as discussed next in the microscopy analysis. Overall, these values confirm the versatility of the TFA-MO-CSD method for growing YBCO films [37, 38].

Figure 2 shows the surface morphology, cross sectional view and high resolution TEM images of the YBCO samples. As shown, the morphology of the films is granular, especially in the sample grown onto SrTiO₃, in which agglomeration of grains is observed. In contrast, the sample grown onto YSZ substrate is more compact. The cross-sectional views indicate cumulus of YBCO grains together with unreacted phases. As mentioned in the experimental section, the top Pt layers were used for protecting the samples during FIB milling and do not influence in the morphology of the samples. Note that the grain components in the sample grown on SrTiO₃ are not uniform and produce accumulation of around 1 µm size while for the sample grown on YSZ the film is more uniform.

Table 1 Phase formation, Miller indexes, peak positions, interplanar distances and crystallite sizes of YBCO thin films grown onto YSZ and SrTiO₃ substrates by following the TFA-MO-CSD technique and annealing at 860 °C.

| Substrate | Phase | Miller Index | 2θ (°) | Interplanar Distance (nm) | Crystallite Sizes (nm) |
|-----------|-------|--------------|-------|--------------------------|------------------------|
| YSZ       | YBCO  | (003)        | 22.93 | 3.9                      | 27.74                  |
|           |       | (003)        | 32.91 | 2.7                      |                        |
|           |       | (005)        | 38.71 | 2.3                      |                        |
|           |       | (006)        | 46.86 | 1.9                      |                        |
|           | CuO   | (110)        | 32.6  | 2.7                      | 23.84                  |
|           |       | (111)        | 38.68 | 2.3                      |                        |
|           |       | (022)        | 65.57 | 1.4                      |                        |
|           | Y211  | (311)        | 29.99 | 2.9                      | 22.28                  |
| SrTiO₃    | YBCO  | (002)        | 15.46 | 5.8                      | 35.23                  |
|           |       | (003)        | 23.11 | 3.9                      |                        |
|           |       | (004)        | 30.79 | 2.9                      |                        |
|           |       | (013)        | 33.03 | 2.7                      |                        |
|           |       | (005)        | 38.91 | 2.3                      |                        |
|           |       | (006)        | 46.91 | 1.9                      |                        |
|           |       | (007)        | 55.39 | 1.6                      |                        |
|           | Y211  | (112)        | 30.81 | 2.9                      | 47.55                  |
|           |       | (203)        | 40.61 | 2.2                      |                        |
|           | CuO   | (111)        | 35.73 | 2.5                      | 49.62                  |
|           |       | (202)        | 48.91 | 1.7                      |                        |
|           |       | (202)        | 58.43 | 1.6                      |                        |
|           |       | (113)        | 61.71 | 1.5                      |                        |
|           |       | (022)        | 65.99 | 1.5                      |                        |
|           |       | (220)        | 68.29 | 1.4                      |                        |
|           |       | (221)        | 69.01 | 1.4                      |                        |
and compact. The HR-TEM image of the YBCO grains indicates crystallites of YBCO with interplanar distance 2.3 Å belonging to the (005) reflection, in agreement to the XRD and Table 1. For the YBCO/SrTiO$_3$ sample, the interplanar distances 2.9 and 1.7 nm are also observed corresponding to the Y211 (311) and CuO (202) reflections of the secondary phase. Thus, the YBCO film is highly granular and composed mainly by secondary phases, as it was also detected by the XRD above. Note that the sample grown onto YSZ is more compact and uniform showing an interplanar distance around 3.8 Å corresponding to the (003) reflection of the YBCO phase. These results suggest that 860 °C is not enough to form uniform YBCO film and leave unreacted phases Y211 and CuO (as also observed in the XRD plot).

In order to study the out-of-plane texture quality produced by the difference in substrates, Rocking Curve (RC) measurements were performed around the (005) reflection (Fig. 3). This reflection was chosen because its intensity is higher than the other (00 l) reflections and also because usually its position in the XRD plot (around 2θ = 38.72°) is far away from any other reflection from the substrate [33, 34]. However, as it is observed in Fig. 1(b), the YSZ reflection (around 34.96°) is close to the (005) YBCO reflection. Thus, in order to maintain the same peak for the later comparison, we add the YSZ reflection’s texture to the final fitting. The full width at the half maximum (FWHM) value of the RC provides information about the degree of inclination of the c-axes of the superconducting grains respecting to the normal axis to the substrate [39]. The RC measurement allows us to observe one region with out-of-plane texture for the YBCO/SrTiO$_3$ sample (see Fig. 3(a)), with FWHM (Δω) = 0.44°. Similarly, Fig. 3(b) shows one region of texture with Δω = 0.40° for de YBCO/YSZ sample. The observed substrate contribution suggests that, in contrast

![Fig. 2](image1.jpg)  
**Fig. 2** Surface, cross sectional TEM and high resolution TEM images of the YBCO films grown onto SrTiO$_3$ and YSZ substrates following the TFA-MO-CSD technique

![Fig. 3](image2.jpg)  
**Fig. 3** Rocking curve measurement of the (005) YBCO reflection for (a) the YBCO/SrTiO$_3$ fitted with a Gaussian function with FWHM = 0.44° and (b) the YBCO/YSZ film where the contribution of the YSZ substrate is observed
to the YBCO/SrTiO₃, this film is in more contact with the substrate as confirmed by the cross sectional TEM images above; this in spite the greater lattice constant mismatch of the crystalline structure (32.88%) compared with the SrTiO₃ perovskite structure (1.35%) mentioned above. This effect might be also related to the differences in roughness in both substrate surfaces. To name, the YSZ surface is less rough than that for SrTiO₃. The (005) YBCO peak is close to the YSZ (002) reflection (around 34.86°).

Figure 4 shows the temperature dependence of the susceptibility ($\chi$) of the samples in the superconducting and normal states obtained under an applied magnetic field of 1 kOe. For the sample grown onto SrTiO₃, it becomes diamagnetic (indicating the Meissner effect, typical from superconductors) from the onset temperature $T_{C_{\text{onset}}}$ ≈ 85.7 K (see Fig. 4(a)). The point from which the ZFC and FC loops diverge define the irreversibility temperature ($T_{irr}$) and it depends on the magnitude of the external magnetic field [38, 40]. Thus, under the applied magnetic field of 1 kOe, $T_{irr}$ ≈ 73.9 K for the YBCO/SrTiO₃ sample. The corresponding $\chi$ (T) loops in the normal state show weak ferromagnetic interaction between moments with Curie–Weiss temperature of $\theta_C = 66.70$ K. Similarly, the susceptibility vs temperature plot for the sample grown on YSZ substrate reveals transition temperature values of $T_{C_{\text{onset}}}$ ≈ 91.8 K and $T_{irr}$ ≈ 84.7 K. Note that in this case, the inset 1/$\chi$ vs. T plot is fitted with two lines suggesting the presence of two kind of magnetic interactions between moments: One weak ferromagnetic interaction $\theta_C = 95.17$ K and another antiferromagnetic interaction with $\theta_C = -100$ K, being this last signal possibly caused due to other phases which are present in the sample.

Figure 5(a), (b) show the $J_C(H)$ curves indirectly obtained by using the Bean’s equation (Eq. 1) and the hysteresis loops taken at 10 and 20 K. For the sample YBCO/SrTiO₃, the value of $J_C$ (at $H = 0$ Oe and $T = 20$ K) is estimated as 0.9 MA/cm². Whereas for the YBCO/YSZ, $J_C$ (at $H = 0$ Oe and $T = 10$ K) is estimated as 0.36 MA/cm². Table 2 compares the $J_C$ values obtained in this work and those reported in the literature. According to it, the values of $J_C$ seem to be influenced by the amount of different types of formed phases [7], the substrate and the growth temperature. High $J_C$ values are obtained for the samples with less secondary phases and substrates with less lattice mismatch compared to that of YBCO [33]. Since the growth temperature mainly influences in the phase formation, density and epitaxy of the film, it should be adjusted depending on the used ingredients and type of substrates to obtain the highest $J_C$ values. In addition to the secondary phases, the granularity and pores should also influence in the $J_C$ values since they are obstacles for the transport of current [29]. This is observed in our results, the samples grown on SrTiO₃ presents greater $J_C$ than over YSZ substrates. A lower degree of inclination also allows obtaining higher $J_C$ values because the CuO₂ planes are better ordered along ab-plane. In the present work, a relative lower degree of
inclination is observed in the YBCO/YSZ sample. Finally, considering that Y211 phase acts as flux pinning centers [33], the quantity of green phase also influences on the $J_c$ values and according to the XRD patterns, a major quantity of Y211 reflections are observed in the (203) reflection around 40.61°.
4 Conclusions

YBCO films were successfully deposited on SrTiO₃ and YSZ single-crystal substrates via the trifluoroacetates metal–organic chemical solution deposition route and annealing at 860 °C. XRD revealed that most of the YBCO is c-axis oriented and without the formation of unwanted BaCO₃. Rocking curves measurements showed only one out-of-plane textured region in both samples. The critical temperature values were 91.8 K and 85.7 K, corresponding to the YBCO samples grown on YSZ and SrTiO₃ respectively. The difference in Tₛ values should be related to the oxygen content, the hole concentration per Cu ion and the presence of secondary phases. The Jₛ curves indirectly calculated by using the Bean’s model from the H(J) loops were Jₛ~10⁹ A/cm² for the sample deposited on YSZ and Jₛ~10⁸ A/cm² for the YBCO/SrTiO₃ film being this difference product of the quantity of Y211 phase observed in XRD and also due to porosity and growth texture in the samples.

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Data availability Data will be made available on reasonable request.

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Table 2 Critical current density Jₛ values obtained at Hₛ=0 in this work and compared with those reported in the literature

| Fabrication technique | Jₛ (A/cm²) | Tₛ (K) | substrate | Secondary phases | Ref |
|-----------------------|-----------|-------|----------|------------------|----|
| CSD                   | 1 x 10⁹   | 77    | LaAlO₃   | BaCu₂O₂, CuO     | [7]|
| TFA-MOD-CSD           | 1 x 10⁹   | 77    | LaAlO₃   | BaCu₂O₂, CuO     | [7]|
| TFA-MOD-CSD           | 0.3 x 10⁹ | 77    | LaAlO₃   | Cu₂O, BaF₂, Y₂O₃| [29]|
| TFA-MOD-CSD           | 2.5 x 10⁹ | 77    | LaAlO₃   | Cu₂O, BaF₂, Y₂O₃| [29]|
| TFA-MOD               | 2.5 x 10⁶ | 75    | CeO₂/YSZ (IBAD) | BaCeO₂ | [41]|
| TFA-MOD-CSD           | 1.5 x 10⁹ | 10    | SrTiO₃   | Cu₂O, Y211       | This work |
| TFA-MOD-CSD           | 0.9 x 10⁹ | 20    | SrTiO₃   | Cu₂O, Y211, YBa₂Cu₃O₆ | This work |
| TFA-MOD-CSD           | 0.4 x 10⁹ | 10    | YSZ      | Cu₂O, Y211, YBa₂Cu₃O₆ | This work |
| TFA-MOD-CSD           | 0.3 x 10⁹ | 20    | YSZ      | Cu₂O, Y211, YBa₂Cu₃O₆ | This work |
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