Structural and morphological characterization of \( \text{YBa}_2\text{Cu}_3\text{O}_{7-x} \) films deposited by screen printing from \( \text{YBa}_2\text{Cu}_3\text{O}_{6.962} \) superconductor in bulk

J M Juárez-Lopez 1, A Guillén-Cervantes 2, J G Quiñones-Galván 3, K E Nieto-Zepeda 1, O Zelaya-Angel 1, J Santos-Cruz 1, E Díaz-Valdés 1, G Contreras-Puente 4 and F de Moure-Flores 5

1 Facultad de Química, Materiales-Energía, Universidad Autónoma de Querétaro, Querétaro, C.P. 76010, México
2 Departamento de Física, CINVESTAV-IPN, Apartado Postal 14-740 D. F. C.P. 07360, México
3 Departamento de Física, Universidad de Guadalajara, Guadalajara, Jalisco, C.P. 44430, México
4 Facultad de Ingeniería, Universidad Autónoma de Querétaro, Querétaro C.P. 76010, México
5 Escuela Superior de Física y Matemáticas del IPN, México D.F. 07738, México

E-mail: fcomoure@hotmail.com
Keywords: \( \text{YBa}_2\text{Cu}_3\text{O}_{7-x} \) superconductor, \( \text{YBa}_2\text{Cu}_3\text{O}_{6.962} \) thin films, screen printing

Abstract

\( \text{YBa}_2\text{Cu}_3\text{O}_{7-x} \) films were deposited onto flexible copper substrates by screen printing technique. \( \text{YBa}_2\text{Cu}_3\text{O}_{6.962} \) films were prepared from a \( \text{YBa}_2\text{Cu}_3\text{O}_{7-x} \) superconductor powder with ethylene glycol. The mixtures were screen printed and then sintered in air at different temperatures: 373, 473, and 573 K. The structural characterization showed the presence of different phases; the proportion of phases in films depends on sintering temperature. Scanning electron microscope images showed that an annealing temperature increase leads to an increase in grain size due to a coalescence process, which promotes the growth of superconductor phases with higher oxygen content.

1. Introduction

The superconductivity effect has its fundamentals in the phonon–electron interactions that take place in a crystalline lattice at low temperatures. The low temperature reduces the atomic vibrations in the material, which produces a drastic reduction in the superconductor electrical resistance \( (0 \Omega) \), this results in the free transport of charge carriers, generating a diamagnetic state \([1, 2]\). The superconductor state occurs when the material is at a temperature known as critical temperature \( (T_c) \) or below. Since its discovery by Onnes \([3]\), phonon–electron interaction has been observed in different superconductor materials through extensive analysis, allowing innovations in superconductor compounds based on copper oxides, and the development of microscopic theories as that of Bardeen, Cooper, and Schrieffer (BCS), that explain the quantum behavior of materials when they are at a critical temperature \([4–6]\).

The development of these superconductor materials derives, mainly, from the crystalline structure understanding and the elements that conform them, as well as their behavior below \( T_c \), this later intended as close to room temperature as possible \([7]\). Materials with a critical temperature above 30 K are considered high critical temperature superconductors (HCTS). Among the most studied and with full applications are those that contain copper and oxygen atoms in the unitary cell since they yield \( \text{Cu}_2\text{O}_2 \) crystalline planes formation \([8]\), these particular crystalline planes facilitate the movement of electric charge at low temperature. However, these materials present some drawbacks in their physicochemical properties, such as the notable oxygen deficiencies in the unit cell. On the other hand, superconductor unit cell dimensions present discrepancies due to the high quantity of elements involved, which produces different crystalline arrangements with variations in the number of \( \text{Cu}_2\text{O}_2 \) crystalline planes formed. This behavior can help to obtain superconductor materials with a \( T_c \) close to 273 K \([8]\).

Among superconductors, \( \text{YBa}_2\text{Cu}_3\text{O}_{7-x} \) presents superconductivity at 90 K, can tolerate critical fields up to 674 T \([8]\). This compound can be found at different superconductor phases, depending on the oxygen content \( (0 \leq x \leq 0.65) \) \([9]\). This superconductor has applications in several fields. Some examples are the fabrication of...
more efficient electrical and electronic devices with a smaller size, including the development of electrical generators and its use in eolic turbines [10–12]; energy ripening and power transmission systems [13–19]; instruments and components for microwave transmission [20]; electro-mechanics systems for assistance in aircraft take-off and landing [21]; development of devices for transport vehicles [22–24] and magnetic refrigeration systems [25]. Most of these technological innovations use YBa2Cu3O7-x superconductor coatings in tapes and cables to reduce the weight and size of electrical devices, increasing their energy efficiency. Different deposition techniques have been used to obtain superconductor films, such as pulsed laser deposition, sputtering, sol-gel, evaporation, epitaxy growth [26–35], among others. However, YBa2Cu3O7-x superconductor coatings require special growth conditions, including substrate preparation with buffer layers deposition from different materials. Besides, in deposition techniques mentioned before, relatively large processes with elevate energy consumption are required due to electro-mechanics devices they use. In this article, we report the deposition of YBa2Cu3O7-x superconductor films by screen printing technique on flexible copper substrates, the as-grown samples were annealed at 373.15, 473.15, and 573.15 K. YBa2Cu3O7-x superconductor films were deposited on flexible copper substrates because the research group is interested in using these films in the development of electric generators.

2. Experiment

2.1. YBa2Cu3O7-x superconductor bulk fabrication
YBa2Cu3O7-x superconductor bulk was obtained from solid-state reaction with Y2O3, BaCO3, CuO powder precursors with 99.99% purity. Powders were mixed in a ball mill and subjected to different thermal treatments. The first thermal treatment was carried out at 1188 K for 11 h; second thermal treatment at 1208 K for 9 h. Immediately after this, an oxygenation process was performed at 638 K for 4 h. The third treatment was at 1183 K for 10 h, followed by oxygenation treatment at 723 K for 4 h. The fourth thermal treatment was realized at 1188 for 10 h. The fifth thermal treatment was at 1218 K for 9 h. Sixth thermal treatment at 1218 K lasted 3 h.

2.2. Films fabrication from YBa2Cu3O7-x superconductor
A mix of YBa2Cu3O7-x superconductor powder with ethylene glycol was obtained with a molarity of 4.32 mol kg⁻¹, which was prepared in an ultrasonic bath for 2 h. Mix was deposited on 1 × 1 in² Cu substrates with a 32 wires/cm mesh with an aperture of ~0.19 mm. Six tests were performed. Samples YBCO-1D, YBCO-1D-373, YBCO-1D-473, and YBCO-1D-573 were obtained with the application of one deposit on each Cu substrate. The film YBCO-1D did not receive the thermal treatment to compare with thermally treated samples. Samples YBCO-1D-373, YBCO-1D-473, and YBCO-1D-573 were annealed at 373, 473, and 573 K. YBa2Cu3O7-x superconductor films were deposited on flexible copper substrates because the research group is interested in using these films in the development of electric generators.

2.3. Characterization
Samples were characterized by X-ray diffraction in a Siemens D5000 equipment with the Cu Kα line to identify crystalline phases. The surface morphology of samples was imaged with a Jeol JSM-7401F field emission scanning electron microscope (FE-SEM) operating at 5 kV. Atomic quantification was performed at 20 kV with a Noran energy dispersive spectrometer installed in the electron microscope. Characteristic vibrational modes were identified with Raman spectroscopy in a Thermo Scientific DXR2 system using the 633 nm line from a 6 mW laser with an aperture of 50 μm.

3. Results and discussion

3.1. Characterization of YBa2Cu3O7-x superconductor in bulk
The X-ray diffraction pattern shown in figure 1, corresponds to the bulk sample obtained by solid-state reaction, in the pattern, the characteristic peaks of the YBa2Cu3O692 superconductor phase can be identified and were
indexed according to powder diffraction file 50-1886. YBa$_2$Cu$_3$O$_{6.962}$ superconductor in bulk belongs to spatial group Pmmm (No. 47) with orthorhombic structure with lattice parameters: $\alpha = \beta = \gamma = 90^\circ$, $a = 3.819$ Å, $b = 3.884$ Å, and $c = 11.683$ Å.

The crystallite size ($D$) and dislocation density ($\delta$) were calculated using the following equations:

$$D = \frac{\lambda \cdot k}{\beta \cdot \cos \theta}$$  \hspace{1cm} (1)

$$\delta = D^{-2}$$  \hspace{1cm} (2)

Where $\beta$ is the FWHM of the diffraction peaks, $\theta$ is the Bragg angle, $\lambda$ is the wavelength of the X-rays emitted by the Cu filament with value 1.5406 Å, $D$ corresponds to crystallite size and $k$ is a constant with value 0.93 [36]. The crystallite size of the YBa$_2$Cu$_3$O$_{6.962}$ superconductor in bulk was 32 nm, and the dislocation density was $9.76 \times 10^{14}$ lines/m$^2$.

Figure 2 shows the resistance ($R$) as a function of the temperature ($T$) for the YBa$_2$Cu$_3$O$_{6.962}$ superconductor obtained by solid-state reaction technique. The inset of figure 2 exhibits a graphic of $dR/dT$ versus temperature. A maximum can be observed, which is associated with the critical temperature of the YBa$_2$Cu$_3$O$_{6.962}$ compound. The superconductor obtained by solid-state reaction technique has a critical temperature of 91.20 K.
3.2. Structural characterization of YBa$_2$Cu$_3$O$_{7-x}$ films

In Figure 3, the X-ray diffraction patterns of the samples obtained by screen printing are observed; crystalline planes corresponding to YBa$_2$Cu$_3$O$_{7-x}$ superconductor phases can be identified. The sample obtained with one deposition step (YBCO-1D) presents the YBa$_2$Cu$_3$O$_{6.9}$ phase, which is a phase with less content of oxygen in comparison with stoichiometry in bulk. Sample with one deposition step and thermal treatment at 373 K (YBCO-1D-373), has a combination of superconductor phases, each with different oxygen content ranging from YBa$_2$Cu$_3$O$_{6.83}$ to YBa$_2$Cu$_3$O$_{6.91}$. This fact could be attributed to the thermal treatment at 373 K. Sample YBCO-1D-473 (annealed at 473 K) exhibited a combination of two superconductor phases identified as YBa$_2$Cu$_3$O$_{6.83}$ and YBa$_2$Cu$_3$O$_{6.948}$, the later with more contributions in diffractogram due, probably, to sintering temperature increase in an open atmosphere. For the sample with thermal treatment at 573 K (YBCO-1D-573 sample), an increase in the superconductor phases, YBa$_2$Cu$_3$O$_{6.94}$, YBa$_2$Cu$_3$O$_{6.948}$, and YBa$_2$Cu$_3$O$_{6.98}$, can be appreciated. On the other hand, the sample with two deposition steps, YBCO-2D-473, shows characteristic signals of superconductor phases YBa$_2$Cu$_3$O$_{6.86}$ and YBa$_2$Cu$_3$O$_{6.967}$, can be appreciated. On the other hand, the sample with two deposition steps, YBCO-2D-473, shows characteristic signals of superconductor phases YBa$_2$Cu$_3$O$_{6.86}$, YBa$_2$Cu$_3$O$_{6.948}$, and YBa$_2$Cu$_3$O$_{6.98}$. Sample YBCO-2D-573 with thermal treatment at 573 K, showed phases recombination; the phases obtained were YBa$_2$Cu$_3$O$_{6.948}$, YBa$_2$Cu$_3$O$_{6.967}$, and YBa$_2$Cu$_3$O$_{6.98}$. Oxygen incorporation in samples is due to thermal treatments performed in an open atmosphere. Note that when the temperature increases, the oxygen incorporation in samples also increases. Figure 4 shows a magnification of the x-ray patterns around 33°, and a slight shift can be appreciated. The YBa$_2$Cu$_3$O$_{7-x}$ superconductor in bulk present diffraction peaks at 32.53° and 32.82°, which correspond to the planes (013) and (103), respectively, of the YBa$_2$Cu$_3$O$_{6.9}$ phase. For the sample obtained with one deposition step (YBCO-1D), these planes are present at 32.77° and 33.04°. When the YBCO-1D sample was annealed at 373 K, the diffraction planes have a shift to lower angles. As the annealing temperature increases, the diffraction
**Figure 4.** X-ray diffraction pattern magnification showing crystalline planes (013) and (103) of bulk and films obtained by screen printing with YBa$_2$Cu$_3$O$_{7-x}$ superconductor phases.

**Table 2.** Crystallite size and dislocation density of YBa$_2$Cu$_3$O$_{7-x}$ in bulk and films calculated with Scherrer-Debye.

| Sample         | Crystallite size (nm) | $\delta \times 10^{15}$ (lines/m$^2$) |
|----------------|-----------------------|----------------------------------------|
| Bulk           | 32                    | 0.976                                  |
| YBCO-1D        | 22                    | 2.07                                   |
| YBCO-1D-373    | 15                    | 4.44                                   |
| YBCO-1D-473    | 19                    | 2.77                                   |
| YBCO-1D-573    | 18                    | 3.09                                   |
| YBCO-2D-473    | 19                    | 2.77                                   |
| YBCO-2D-573    | 20                    | 2.50                                   |
peaks shift continues until returning to the positions corresponding to the YBa2Cu3O692 phase. These results indicate that the annealing temperature promotes the formation of the YBa2Cu3O692 phase in films obtained by screen printing. This phase variation is responsible for the observed shifts.

From X-ray patterns in figure 3 and equations (1) and (2), average crystallite size and dislocations density were calculated. The results are displayed in table 2. The crystallite size of the as-grown film (YBCO-1D sample) was 22 nm, while the YBCO-373 sample (annealing at 373 K) has a crystallite size of 15 nm. For annealing temperatures of 473 and 573 K, the crystallite size decreases to 19 and 18 nm, respectively. It can be appreciated that the variation in crystallite size for the screen printed films can be considered as negligible. However, the lowest size for the sample annealed at 373 K indicates that the crystalline quality is lower for this film, which can be observed by the higher value of the dislocation density for the YBCO-373 film. Note that the crystallite size of the YBa2Cu3O692 superconductor in bulk is higher than for films obtained by screen printing, which may be due to the high oxygenation treatment at 723 K.

In order to understand the terminology used in the characterization by Raman spectroscopy in figure 5, an arrangement as well as typical positions of the elements conforming the unit cell for YBa2Cu3O7-x superconductor, are shown (figure 5(a)). The image defines Cu2, O2, and O3 atoms bonds to form CuO2 planes. In figure 5(b), it can be observed how O1 and Cu1 atoms form CuO parallel oriented chains with respect to the c axis, and CuO and BaO planes. Oxygen atom O4 bonds to CuO2 planes with CuO chains.

YBa2Cu3O7-x superconductor presents active Raman modes 5A_g + 5B_2g + 5B_3g in the orthorhombic arrangement [37]. Therefore, this high-temperature superconductor has variations in the oxygen content [38]. Thus, it presents very slight shifts in the vibrational modes due to lattice stress attributable to a mix of superconductor phases in different proportions. This behavior is observed in the Raman spectra in figure 6, corresponding to YBa2Cu3O7-x in bulk and the samples obtained by screen printing. A_g Raman modes belong to Ba bonds in ~115 cm^{-1}, Cu2 in ~145 cm^{-1}. Vibrational modes in ~335 cm^{-1} are asymmetric vibrations due to O2 and O3 bonds; also note O2 and O3 bonds formation with symmetric vibrations in ~446 cm^{-1}, these last vibrational modes are associated to CuO2 planes where superconductivity takes place [37, 39–41]. The vibrational mode in ~500 cm^{-1} is inherent to O4, a vibrational tension in the c axis attributed to Cu-O bonds [42, 43]. Samples in this work, in addition to characteristic vibrational modes from YBa2Cu3O7-x, present the existence of other vibrational modes in ~174–191, ~203–233, ~245–253, ~275–289, ~386–392, ~408–417, ~529, and ~584–608 cm^{-1}. These modes are characteristic of this high-temperature superconductor and are attributed to defects and/or partial oxygen vacancies [44–53].
Figure 6. Raman spectra of YBa$_2$Cu$_3$O$_{6.962}$ superconductor in bulk and films deposited by screen printing on flexible Cu substrates.

| Sample       | Y (At%) | Ba (At%) | Cu (At%) | O (At%) | Y:Ba:Cu:O    |
|--------------|---------|----------|----------|---------|--------------|
| Bulk         | 7.02    | 14.33    | 20.55    | 58.10   | 1:2.04:2.93:8.27 |
| YBCO-1D      | 7.19    | 14.60    | 22.40    | 55.81   | 1:2.03:3.11:7.76 |
| YBCO-1D-373  | 7.16    | 15.27    | 22.42    | 55.15   | 1:2.13:3.13:7.70 |
| YBCO-1D-473  | 7.24    | 15.57    | 21.25    | 56.94   | 1:2.01:3.93:7.86 |
| YBCO-1D-573  | 7.27    | 14.87    | 23.25    | 54.61   | 1:2.04:3.20:7.51 |
| YBCO-2D-473  | 7.21    | 14.47    | 22.33    | 55.79   | 1:2.01:3.12:7.74 |
| YBCO-2D-573  | 7.05    | 14.22    | 22.11    | 56.62   | 1:2.02:3.14:8.03 |
Bulk and films with the YBa$_2$Cu$_3$O$_{7-x}$ superconductor phases were analyzed through energy dispersive spectroscopy (EDS). Measurement results are shown in Table 3. In general, Y, Ba, and Cu atomic percentages (at%) remain constant, while nominal oxygen concentration has a random variation. Y and Ba at% have values around 7 and 14%, respectively. Cu at% has values between 20 and 23%, while the oxygen at% has values around 55%–58%; similar results can be found in the literature [54]. Note that the proportions of elements with respect to Y are close to expected stoichiometry, see Table 3.

3.3. Morphological characterization of YBa$_2$Cu$_3$O$_{7-x}$ films
In order to analyze the surface morphology of films deposited by screen printing, Scanning Electron Microscopy (SEM) measurements were carried out. Figure 7(a) shows the SEM image of the as-grown YBa$_2$Cu$_3$O$_{7-x}$ film, while figures 7(b)–(d) shows the SEM images of YBa$_2$Cu$_3$O$_{7-x}$ films annealed at 373, 473 and 573 K, respectively. Note that apparently, the sample without heat treatment as well as sample annealed at 373 K have
pinholes. It can be seen that when the annealing temperature increases, there is a coalescence of the grains, which reduces the presence of pinholes in the surface of the YBa$_2$Cu$_3$O$_{7-x}$ films deposited by screen printing. This coalescence of grains results in obtaining larger grains, figure 7(d) shows the morphology of the sample with annealing at 573 K, in which grains of up to 30 μm can be observed. The existence of a high density of bigger grains in comparison with pores and grain boundaries density favors electrical conductivity properties according to reports in the literature [55]. The samples obtained with two deposits and annealed at 473 K and 573 K are shown in figure 7(e) and figure 7(f), respectively. Observe that that the YBa$_2$Cu$_3$O$_{7-x}$ sample processed with two deposits and annealed at 573 K shows better compaction and the absence of pinholes, in agreement with XRD analysis; increasing the annealing temperature promotes the formation of superconductor phases.

4. Conclusions

YBa$_2$Cu$_3$O$_{7-x}$ films, with different superconductor phases, were obtained by screen printing technique on flexible Cu substrates using a YBa$_2$Cu$_3$O$_{6.962}$ superconductor as source. Structural characterization with X-ray diffraction showed the presence of different phases, which depends on annealing temperature and the number of layers. Higher annealing temperature promotes phases richer in oxygen. Raman analysis allowed the identification of characteristic vibrational modes associated with YBa$_2$Cu$_3$O$_{7-x}$. This analysis, together with EDS results, indicated that no other elements were incorporated into the compound. SEM images showed that thermal treatment temperature increase leads to an increase in grain size due to a coalescence process, which promotes the formation of superconductor phases with higher content in oxygen. This work reveals the importance that both thermal treatment conditions and the number of deposits have to obtain YBa$_2$Cu$_3$O$_{7-x}$ superconductor phases on flexible Cu substrates that could have applications in electronic and electric devices fabrication.

Acknowledgments

We acknowledge the technical support of Marcela Guerrero and A. Garcia-Sotelo from the Physics Department, CINVESTAV-IPN. Also, acknowledge the technical support of Bruno Flores-Hernandez. The authors are thankful for the financial support from FONDO SECTORIAL CONACYT-SENER-SUSTENTABILIDAD ENERGÉTICA through CeMIE-sol, in the strategic project 37 'Development of new photovoltaic devices and semi-superconductor materials'.

ORCID iDs

A Guillén-Cervantes @ https://orcid.org/0000-0001-6467-2939
J G Quiñones-Galván @ https://orcid.org/0000-0001-7931-3955
F de Moure-Flores @ https://orcid.org/0000-0002-8010-3573

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