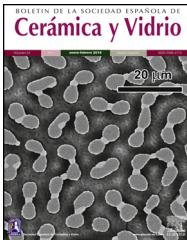




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## Effect of yttrium doping on structural and electrical properties of $\text{Bi}_2\text{Sr}_{1.9}\text{Ca}_{0.1-x}\text{Y}_x\text{Cu}_2\text{O}_{7+\delta}$ (Bi-2202) cuprate ceramics

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### ABSTRACT

In this work, we report on the effect of  $\text{Y}^{3+}$  doping on structural, mechanical and electrical properties of Bi-2202 phase. Samples of  $\text{Bi}_2\text{Sr}_{1.9}\text{Ca}_{0.1-x}\text{Y}_x\text{Cu}_2\text{O}_{7+\delta}$  with  $x=0, 0.025, 0.05, 0.075$  and  $0.10$  are elaborated in air by conventional solid state reaction and characterized by X-ray diffraction (XRD), scanning electronic microscopy (SEM) combined with EDS spectroscopy, density, Vickers microhardness and resistivity measurements. A good correlation between the variations of the bulk density and the Vickers microhardness with doping is obtained. The SEM photograph shows that the samples are composed of grains with a flat shape that characterizes the Bi-based cuprates. Quantitative EDS analysis confirms the reduction of Ca content and the increase of Y content when  $x$  is increased. The variation of resistivity with temperature shows that only samples with  $x=0, 0.025$  and  $0.05$  present an onset transition to the superconducting state. The higher onset transition temperature is obtained for  $x=0.025$  and is about 93.62 K. The transition is wide and is realized in two steps confirming then the presence of the low  $T_c$  Bi-2201 phase in the samples. For  $x=0.075$  and  $0.10$ , a transition to a semiconducting state is seen at low temperatures. Some physical parameters are extracted from these curves and discussed.

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### Efecto del dopado con itrio en la estructura y las propiedades eléctricas de $\text{Bi}_2\text{Sr}_{1.9}\text{Ca}_{0.1-x}\text{Y}_x\text{Cu}_2\text{O}_{7+\delta}$ (Bi-2202) en cerámicas basadas en CuO

### RESUMEN

#### Palabras clave:

Fase Bi-2202

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Dopado

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Superconductores

En este trabajo se describe el efecto del dopado con  $\text{Y}^{3+}$  en la estructura y las propiedades mecánicas y eléctricas de la fase Bi-2202. Las muestras de  $\text{Bi}_2\text{Sr}_{1.9}\text{Ca}_{0.1-x}\text{Y}_x\text{Cu}_2\text{O}_{7+\delta}$ , con  $x=0, 0.025, 0.05, 0.075$  y  $0.10$ , se prepararon en aire por reacción en estado sólido y fueron caracterizadas por medio de difracción de rayos X (XRD), microscopía electrónica de barrido (SEM) combinada con espectroscopía EDS, densidad, microdureza Vickers y resistividad eléctrica. Se ha obtenido una buena correlación entre las modificaciones en densidad y microdureza

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inducidas por el dopado. Las micrografías han mostrado que las muestras están formadas por granos laminares, lo que es típico en los superconductores basados en Bi. El análisis cuantitativo realizado con el EDS ha confirmado la disminución en el contenido de Ca y el aumento de Y, cuando se aumenta el valor de  $x$ . Las variaciones de la resistividad eléctrica en función de la temperatura mostraron que solo las muestras con  $x=0,025$  y  $0,05$  Y presentaban una transición al estado superconductor. La mayor temperatura de transición se ha obtenido en las muestras con  $x=0,025$ , alcanzando 93,62 K. La transición es ancha y se realiza en 2 pasos, lo que confirma la presencia de la fase de baja  $T_c$ , Bi-2201, en las muestras. Para  $x=0,075$  y  $0,10$  se observa una transición a un estado semiconductor a bajas temperaturas. Se han extraído y discutido algunos parámetros físicos relevantes de esas curvas.

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## Introduction

Intensification of synthesis and study of Bi-based superconductors are still of interest for researchers [1–3]. Superconductivity at 9–20 K in ternary bismuth cuprates was first reported by Michel et al. [4] for the BiO–SrO–CuO system. Addition of calcium oxide to this system led Meada et al. [5] to discover bulk superconductivity in  $(\text{Bi},\text{Pb})_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4+\delta}$  ( $n=1-3$ ) and their critical temperatures are approximately 20 K, 85 K and 110 K for the Bi-2201, Bi-2212, and Bi-2223 phases, respectively [4–6]. The only difference between these two consecutive phases is the addition of a double CaCuO block in the perovskite subunit resulting in an increase of the  $c$ -axis parameter. In addition, the Pb-doped Bi–Sr–Ca–Cu–O system has higher thermodynamic stability than the Pb-free one. This is because lead substitutes at Bi sites and reduces the incommensurate modulation [7]. Chemical doping in high  $T_c$  superconducting cuprates (HTSC) has generated a great interest because it represents an easily controlled, non-destructive and efficient tool for improving the mechanical, structural and superconducting properties of these compounds [8–10]. The doping element nature and its ionic radii, preparation method, sintering temperature, thermal processing time, synthesis atmosphere, precursor compositions and doping or substituting various cations and anions have an important influence on the properties of superconducting materials. In many HTSC families of compounds, the rare-earth elements play an important role in establishing the proper structure. The substitution of  $\text{Ca}^{2+}$  (divalent) by trivalent rare-earth elements in HTSC causes a drastic change in the carrier concentration and results in transition from superconductor to an insulator [11–13]. Many reasons have been suggested for the decrease in the carrier concentration such as structural modulations or change in oxygen stoichiometry change in the Cu valence or both [14]. The substitution by different elements of the rare earths in HTSC concerned usually at high doping levels.

For a better understanding the mechanism of the superconductivity and their unusual physical properties, the influence of small concentrations of doping atoms should be considered. In particular, we have studied the effect of doping by low content of yttrium at Ca and Sr sites on the structural

and microstructural properties of Bi(Pb)-2212 superconducting ceramics [15], where the superconductivity properties are better when the doping is realized between the  $\text{CuO}_2$  planes at Ca site. In comparison with the Bi-2201, Bi-2212 and Bi-2223 compounds that have been extensively studied, only two papers were published on the Bi-2202 phase [4,16]. The influence of the annealing atmosphere on the superconductivity has been mostly studied [17–20], which revealed that the oxygen content and its distribution are the key points to the improvement of superconducting properties in HTSC. The Bi-2202 phase could show a superconducting behaviour only if it is doped by small amounts of Ca at the Sr [16]. Indeed, a sample of stoichiometry  $\text{Bi}_2\text{Sr}_{1.9}\text{Ca}_{0.1}\text{Cu}_2\text{O}_{7+\delta}$  elaborated in air shows a highest onset transition temperature ( $T_{c,\text{on}}$ ) of about 90 K. The oxygenation at high temperatures seems to have no effect on the value of  $T_{c,\text{on}}$ .

In this paper, we focused on the effect of doping with low content of yttrium ( $\text{Y}^{3+}$ ) on structural, microstructural, mechanical and electrical transport properties of the  $\text{Bi}_2\text{Sr}_{1.9}\text{Ca}_{0.1-x}\text{Y}_x\text{Cu}_2\text{O}_{7+\delta}$  cuprate.

## Experimental procedure

### Chemical synthesis

The polycrystalline samples of  $\text{Bi}_2\text{Sr}_{1.9}\text{Ca}_{0.1-x}\text{Y}_x\text{Cu}_2\text{O}_{7+\delta}$  cuprate ceramics with  $x=0, 0.025, 0.05, 0.075$  and  $0.10$ , each of about 2 g of total weight, were prepared by solid state synthesis method, using high purity chemicals of  $\text{Bi}_2\text{O}_3$ ,  $\text{SrCO}_3$ ,  $\text{CaCO}_3$ ,  $\text{CuO}$  and  $\text{Y}_2\text{O}_3$  (Aldrich >99.9%). Stoichiometric amounts of the ingredients were accurately weighed using an electronic balance, thoroughly mixed and ground using an agate mortar. The mixture was then subjected to a stage calcination process in air at  $800^\circ\text{C}/24\text{ h}$ . After calcination, the samples were formed into cylindrical pellets under the pressure 30 kN at room temperature. The process of sintering comprised three steps. The first step of sintering was conducted at  $820^\circ\text{C}$  for 24 h. Next, the samples were crumbled, wet milled and again formed into pallets. The second step of sintering was carried out at the temperature of  $835^\circ\text{C}$  for 24 h, consecutively. The procedure of crushing and milling was repeated before the final sintering at  $850^\circ\text{C}$  for 24 h.

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