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# Synthesis of BiSrCa(Y)CuO Superconductor from the Sol-Gel Method and the Effect of Y Substitution

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**Abstract.** Superconducting Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>1-x</sub>Y<sub>x</sub>Cu<sub>2</sub>O<sub>8</sub> ceramics samples have been prepared by Sol-Gel methods using citrate process. The influences of the conditions of preparation of oxide powder compound on structural and superconducting properties have been investigated by X ray diffraction (XRD), Scanning Electron Microscopy (SEM) equipped with EDS. The critical transition temperatures T<sub>c</sub> have been determined by resistivity versus temperature measurements. Cell parameters samples were calculated from XRD patterns. The polyacryl amide gel makes the citrate process easier, more rapid and affords the possibility of synthesis of high quality oxide powders.

**Keywords:** Bi2212 phase, Superconductivity, Sol-Gel - Polyacrylamide Gel.

**PACS:** 61.10-i, 68.37-Hk, 74.62-c, 74.72-h.

## INTRODUCTION

The general chemical formula for the Bi-based superconducting phases can be written as Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>n-1</sub>Cu<sub>n</sub>O<sub>4+2n+d</sub>, with n=1, 2, 3 corresponding to the number of CuO<sub>2</sub> planes in their structure and having a T<sub>c</sub> of 22K (Bi2201 phase), 80K (Bi2212 phase) and 110K (Bi2223 phase) respectively [1].

In order to study their behavior, many methods have been used for the synthesis of Bi2212 cuprates such as sol-gel process [2-6], co-precipitation method [7], citrate gel process [8, 9], solid state reaction, oxidation of liquid quenched precursor alloys, and matrix reaction. Each of these techniques has limitations and problems associated with the quality of the material produced. Bismuth containing powders are mostly prepared by solid-state reaction at high calcinations temperatures over 860°, with many processes to reduce particle size of generally non homogeneous mixtures to a microscopic scale. Contrary to the solid state reaction, the sol-gel colloid solution combustion synthesis is a process which synthesizes on high-purity multi compound and involves a low temperature combustion process which is found to allow the preparation of very fine and homogeneous powders. Pure and homogeneous products may be obtained by this method in a shorter reaction time [10].

It is well known that, in Bi2212 phase [11], Ca<sup>2+</sup> cations, staying between CuO<sub>2</sub> planes, compensate the structural disorder induced by Sr<sup>2+</sup> cations which are located above the apical oxygen of the same planes. In this paper, we prepared the

Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>1-x</sub>Y<sub>x</sub>Cu<sub>2</sub>O<sub>8+d</sub> superconducting phase by sol-gel method to study the effect of Y substitution for Ca on its structural and superconductive properties.

## EXPERIMENTS

Bismuth, strontium, calcium and copper nitrates were dissolved in distilled water in a stoichiometric ratio. Y powder dissolved in nitric acid was added to the Bi solution. A solution of tri ammonium citrate with a concentration of 2mol/Kg, was made by reacting citric acid with ammonia solution. The organic gels were made with acryl amide CH<sub>2</sub>=CHCONH<sub>2</sub> and N,N methylene di acryl amide CH<sub>2</sub>=CHCONHCH<sub>2</sub>NHCOCH=CH<sub>2</sub> was added in order to complex the Bi, Ca, Sr, Cu cations. The obtained solution was mixed, stirred and heated at 80°-90°C with a magnetic stirrer on a hot plate. Few drops of a solution of AIBN in acetone were added to help the formation of the gel. The gel is transformed in Bi2212 powder by heating at 400°C for 2h and successively at 700°C for 6h. The obtained powder is ground in an agate mortar and calcined during 12h at 860°C reached with a rate of 5°C/min. After calcination, the samples were ground again, and then pelletized under a pressure of 3 tons. The pellets were then sintered during 12h at 860°C reached with a rate of 5°C/min.

## RESULTS AND DISCUSSION

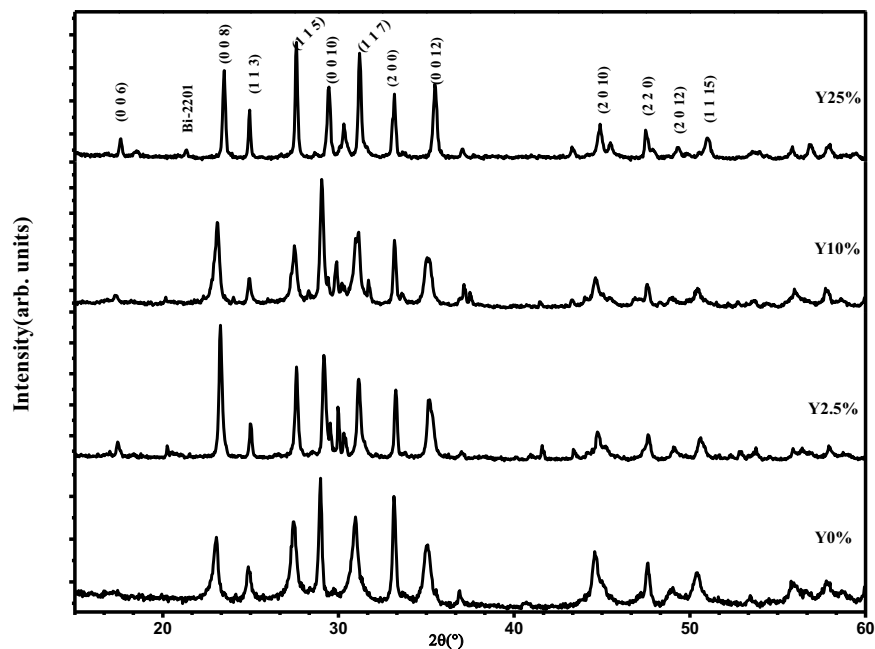
The Bi2212 phase is dominant in the XRD patterns of the samples shown in figure 1. The patterns show weak peaks corresponding to Bi2201 impurity phase. The variations of lattice parameters against Y content, calculated from the XRD patterns using (hkl) parameters and assuming an orthorhombic symmetry, are shown in table 1. Substituting of Y for Ca preserves the basic crystalline structure. XANES spectra measurements [12] have shown that at the Cu K-edge the substitution of Y for Ca in Bi2212 decreases the formal Cu valence, resulting in an increase of the Cu–O distance and a decrease of Cu–O bond covalency. The invariant Bi L-edge XANES spectra suggests also that the charge transfer along the Cu–O–Bi bond will influence the metal–insulator transition of Bi2212 system upon increasing the amount of Y.

**TABLE 1.** Lattice parameters *a*, *b*, *c*, T<sub>c on</sub> and ΔT of the samples.

Y content (%)	<i>a</i> (Å)	<i>b</i> (Å)	<i>c</i> (Å)	T <sub>c on</sub> (K)	ΔT(K)
0	5.21316	5.61848	30.79548	90	35
2.5	5.35879	5.64283	30.65850	83	53
10	5.38880	5.78880	30.57478	110	70
25	5.40792	5.90487	30.31062	85	71

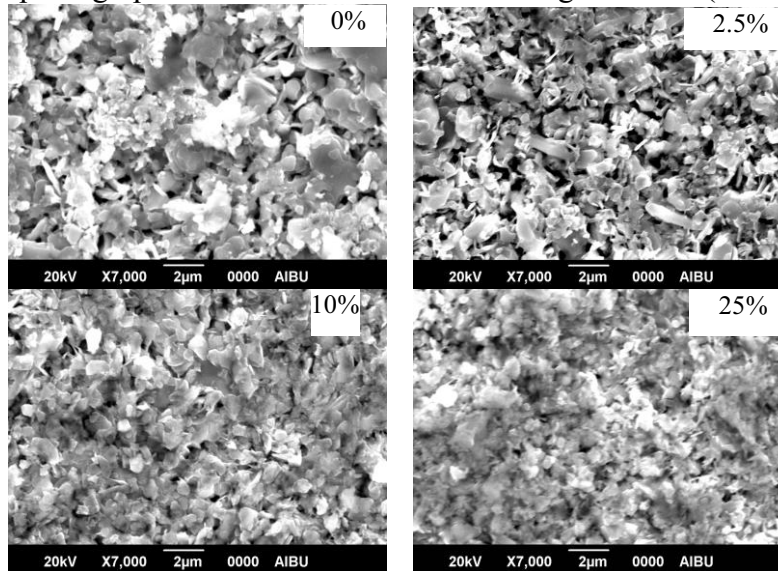
*c* axis parameter decreases with the content of Y meanwhile both *a* and *b* in plane parameters increase slightly. This evolution of *c* has been already observed in the literature [13, 14]. The Y<sup>3+</sup> cation has a smaller ionic radius than Ca<sup>2+</sup>. This explain the decrease of *c* which can be also due to the increase of oxygen content of the system as it has been suggested for Ce substitution on Sr site

[15]. Previous experimental results exhibit the decrease of the average valence of Cu with Y content, explaining the trend of *a* and *b* parameters [16].



**FIGURE 1.** The XRD patterns of the samples with indexation of the peaks belonging to the Bi2212 phase.

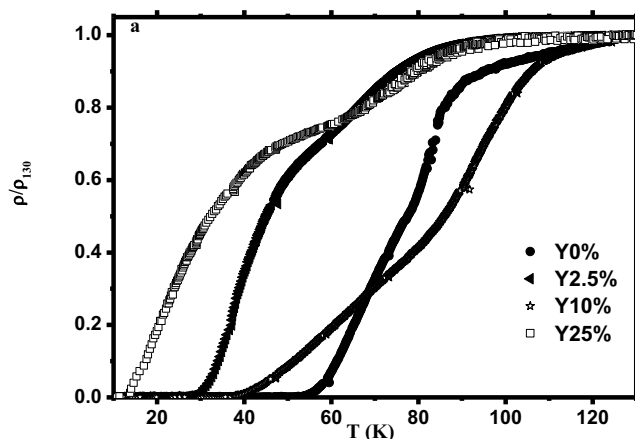
The SEM micrographs of figure 2 show the morphology of the surface of the samples. The photographs were taken with the same magnification (x 7000).



**FIGURE 2.** SEM micrographs of the samples with the content of yttrium indicated on each one.

The samples present grains with a size of about 2 μm. The undoped sample has flat shape grains much larger than those of the doped one with a uniform distribution of their size. There are voids in the doped samples while the surface of the undoped sample is smoother and denser. Doping with yttrium decreases the size of the grain and worsens the surface morphology.

The resistivity  $\rho(T)$  of the samples, measured using the standard four-probe dc technique between 10 and 130 K and normalized to its value at 130 K  $\rho_{130}$ , is displayed in figure 3. Doping with Y degrades moderately the onset of transition  $T_{c_{on}}$ , which decreases to 83K and 85K for  $x=0.025$  and  $x=0.25$  respectively, but the width of the transition  $\Delta T$  is much more degraded confirming the morphological quality observed by SEM analysis. The sample with  $x=0.1$  present the same  $\Delta T$  but a  $T_{c_{on}}$  much higher of about 110K suggesting a higher presence of Bi2223 phase.



**FIGURE 3.** Resistivity  $\rho(T)$  of the samples normalized to its value at 130K  $\rho_{130}$ .

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